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Technical Memorandum 33-357

Review of Advanced Composite Materials for Spacecraft Applications

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Contents

Abstract

A survey of the present status of composite materials technology was made with emphasis on materials and applications useful in spacecraft structures. Information was obtained from the literature, by attendance at meetings and symposia, discussions with workers in the field, and visits to some of the organizations active in composite development.

Brief discussions of the theory of composite strengthening and reinforcement mechanics are included. Advantages, disadvantages, problem areas, and actual and potential applications are described. The status of testing, processing, forming, fabrication, and joining are noted. Many of the more important programs and organizations active in composite development are identified, as are anticipated advances in composite technology. A separate appendix contains abstracts and comments on a number of papers and reports on fibers and composites.

Composites possess significant advantages over presently used spacecraft structural materials; these include superior strength-to-weight and stiffness-to-weight ratios, more design freedom and flexibility, improved resistance to crack propagation, and greater potential for improvement of properties. Major problems include lack of uniform test methods, difficulty of joining, high cost, difficulty of fabrication and processing, and lack of information on reliability and reproducibility, particularly for the more advanced composites.

Review of Advanced Composite Materials for Spacecraft Applications

I. Introduction

The purpose of this report is to describe the present status and anticipated future developments in advanced, high-efficiency structural composite materials. This report represents the results of a literature survey that is the first phase of a program on High-Efficiency Spacecraft Structures (NASA Code 124-09-01-02) being done by the Materials Section of the Jet Propulsion Laboratory. This work was undertaken to determine where and how composite materials could best be applied to future spacecraft designs.

Information reported herein was obtained by surveying pertinent published reports, attendance at meetings and symposia, personal discussions with other workers in this field, and visits to some of the organizations particularly active in composite materials development.

As a result of this extensive survey, it has been possible to draw several conclusions as to the present status and most likely future progress in composites. Problem areas, potential successful applications, and approaches have been identified. The most advanced composites currently available are also noted.

The advantages and disadvantages of composites are discussed, and a brief summary of the presently obtained material properties and product availability is given. Pitfalls, problems, and peculiarities in the testing, forming, joining, and usage of composites are presented. The status of primary and secondary fabrication techniques also is included. Some of the more important current programs are described, and a projection is made of probable advances in the state of the art. An appendix is included to provide abstracts of a number of reports and papers on advanced fibers and composites.

The advantages and potential utility of composites have been described and promoted extensively (Refs. 1–7). The major problems are the bewildering number and variety of composite systems that have been prepared and tested, the problems and variability encountered in processing composites, and the lack of information and experience in structural applications. Development of reinforcement materials and composite fabrication techniques has been rapid and extensive, with nonmetallic matrix systems more advanced than metal matrix composites. However, few large structures have been fabricated except for glass-resin rocket motor cases. No single advanced material-process combination is completely developed; although several are well advanced, particularly resin matrix systems. Since composite technology presently is in a state of rapid change and development, it appears likely that no major use of composite materials for spacecraft structural applications will take place in the immediate future.

A vital point in the successful application of composites is the need for a cooperative effort among the materials, design, and structures engineers (Refs. 2, 3, 8, and 9). Since composites are made to order, they can be produced with a wide range of fiber content and fiber orientation or fiber location, thus tailoring properties to a given use. For best results the material, fabrication technique, and application should be considered as a system to perform a specific function. This is the only approach which will permit maximum material utilization and minimum design problems without an extensive iterative process. Therefore, interdisciplinary cooperation is a must if an efficient, minimum cost design is to result.

II. Background

Composite materials discussed herein contain two phases or elements: (1) the reinforcement which carries the major portion of the load and resists deformation; and (2) the matrix, which provides stress transfer between the reinforcement and the stress field; it also protects the reinforcement from the environment and absorbs some of the strain energy in the composite. The reinforcement may be discontinuous, i.e., whiskers (single crystals), or short fibers (discontinuous polycrystalline filaments), or continuous filaments. Each composite system can be designed from the initial material components and fabrication processes to satisfy a specific application requirement (Refs. 2, 6, and 8).

This report excludes those materials which are strengthened by a second phase, by a dispersion of one material in a matrix of another, or by a change in microstructure caused by heat treatment or primary metal working (e.g., precipitation hardening, maraging, strain aging, etc.), except for unidirectionally solidified eutectics.

There are certain attributes common to all composite systems; e.g., the reinforcing member should be higher in modulus of elasticity and ultimate tensile strength than the matrix. These properties usually are associated with high hardness and low shear strength. The matrix should resist deformation in shear, have a relatively high modulus, and be capable of absorbing appreciable plastic strain to protect the more brittle load-bearing phase. Load transfer through the matrix between the load and the reinforcement should be uniform and without stress concentration (Refs. 7, 10, and 11). Gross differences in fiber-matrix thermal expansion coefficients can cause delamination or other failure of the composite if either the fabrication or service temperatures are too high. Chemical interactions between fiber and matrix also may be deleterious, and must be considered.

Composites may be grouped into two major categories according to the matrix material: metallic or nonmetallic. Each of these categories can be further subdivided by type of reinforcement: nonmetallic or metallic. A further subdivision is by character of the reinforcement: whisker, fiber, filament, or wire; cloths would be considered as special cases of filamentary reinforcement.

The systems using shorter reinforcements, i.e., whiskers or fibers, must have enough fiber overlap to transfer the stresses from one row of fibers to another or else failure will occur along planes where reinforcement is absent and the composite is weak.

Both continuous and discontinuous reinforcements must exceed a critical minimum aspect ratio L/d of fiber length L to diameter d, known as L_c, for best results. This critical length L_c is the value required to permit the fiber to carry the full load which it is capable of bearing. It is measured by tensile testing actual composite samples and comparing results to the theoretical strength of the composite with infinitely long fibers. If the L/d is less than L_c, the fiber will break at less than its true maximum strength or, if it is near a free surface, it will pull out of the matrix (Ref. 12). The value of L_c varies with the matrix and reinforcement within relatively narrow ranges. Fortunately most composites have relatively low fiber L_c values. For example, values of L_c for tungsten (W) in copper (Cu) of as little as 4 have been reported. Also, as test temperature increases, L_c increases (Refs. 12 and 13). Practically speaking, when L/d exceeds 100 it is essentially equivalent to $L/d = \infty$ and is well above L_c in most, if not all, systems (Refs. 13 and 14).

Both elastic and plastic stress transfer must be considered to obtain the true value of L_c (Ref. 15). Neglecting plastic stress transfer can cause the estimated L_c to be appreciably higher than the actual L_c (Ref. 15). Also, L_c

is affected by fiber diameter and spacing; L_c increases as fiber diameter decreases. For optimum stress transfer L_c should be about equal to fiber spacing. If the spacing is too great, failure occurs along the weak planes between fibers; if it is too close, the fiber stress fields interact and cause stress concentration effects (Refs. 6, 16, and 17).

The interface between the matrix and reinforcement must be capable of smooth load transfer along the entire length of each whisker or fiber. The interface itself ought to be relatively thin, compatible with both matrix and reinforcement, and should not be brittle or form a sharp notch in the whisker or fiber (Refs. 5 and 18–20). There is some controversy as to whether chemical or mechanical bonding between matrix and reinforcement is necessary. Apparently it is possible to obtain good stress transfer and reinforcement when the bonding is only mechanical (Refs. 21–24). The major problem in mechanically-joined systems is assuring that there is good contact and stress transfer along most or all of the fiber (Refs. 5–7, 11, and 21). A coating often is applied to the fiber to provide better bonding and stress transfer between matrix and fiber; sometimes this coating also is designed as a diffusion barrier for elevated temperature applications (Ref. 6). Selection and application of fiber coatings is a very broad field which is not well understood and requires much more effort.

Additions of small amounts of active elements to the matrix have been shown to be an effective method for increasing interfacial bonding and bond strength (Refs. 11 and 22–25). It has been suggested that in metal-ceramic composites there are two mechanisms operating (Refs. 11 and 23). In one case, the active element forms an interfacial compound which forms a strong bond. Excessive reaction may cause the bond region to thicken undesirably, resulting in a brittle, weak area. In addition, the net fiber area may be reduced so greatly that the reinforcement strength is reduced significantly. This effect has been noted in the copper-tungsten (Cu-W) system; notches formed in the fiber by the reaction zone also act as stress concentrators, causing premature failure (Refs. 18 and 26). In the second case, a roughening of the surface of the ceramic reinforcement promotes mechanical adherence. This surface roughening also acts to form notches and failure sites, and usually greatly weakens the ceramic (Refs. 11 and 23). A summary of the status of interface studies is given in Ref. 27.

Another approach to improved fiber-matrix bonding is the use of surface treatments to increase bond strength. Most of this work has been done for glass filament/resin, carbon (C) filament/resin systems (Refs. 28–39) and boron (B) filament/resin systems (Refs. 20 and 31), but there has been some work on B-filament/metal matrix composites (Refs. 20 and 32). The need for and effectiveness of such surface treatments is dependent upon the nature and type of the system being considered.

The mechanics of fiber reinforcement have been studied extensively, considering both the effect of the binder or matrix and the reinforcement material (Refs. 15 and 33–44). A number of authors have developed expressions for the theoretical properties of composites based upon matrix and reinforcement properties (Refs. 12 and 45–48). The effects of fiber shape (Refs. 31, 43, 44, and 49–51), length-to-diameter ratio (Refs. 12, 13, and 16), spacing (Refs. 16 and 17), and orientation (Refs. 12, 52, and 53) have been treated by a number of workers. The optimum reinforcement shape was found to be oval, with tapered ends; a spacing equal to fiber diameter seems to give maximum strengthening. Those interested in a more detailed discussion of the micromechanics aspect of composite technology are referred to the given references which will provide a broad cross-section of the work in composite mechanics.

Processing of both the whiskers or filaments and the composite itself is an extremely critical phase of composite design and fabrication. Since the composite is produced concurrently with the incorporation of the reinforcement into the matrix, the method of fabrication and processing can have as severe an effect on final properties as the type and amount of reinforcement or the nature of the matrix.

Often the selection of a material system is severely limited or altered by the availability or applicability of fabrication processes (Ref. 54). For example, excessive heat and/or pressure during hot pressing may damage the reinforcement element or cause unwanted reaction with the matrix. Conversely, the selection of a process may be influenced greatly by the materials being used and their compatibility with or reaction to certain types of processing. Fibers which are attacked by the liquid matrix material cannot successfully be formed into composites by casting techniques.

The next consideration is alignment of the reinforcement. Misalignment of as little as 3 deg between reinforcement and the applied load can result in appreciable loss of strength as compared with the maximum composite capabilities (Ref. 52). When tested at 45 or 90 deg to the reinforcement direction, the composite may be weaker than the reinforced matrix (Ref. 52). This consideration is particularly important when the reinforcing material is whiskers or short fibers which are difficult to orient.

It is possible to increase the isotropic strength of composites by adding whiskers or fibers in random directions, by wrapping in different directions, by three-dimensional weaving, or by laminating uniaxially aligned layers in various directions (Refs. 13, 14, 52, and 55). In none of these cases is the increase in strength for a given volume percent of reinforcement comparable to that obtained for a composite which is reinforced and loaded uniaxially.

III. Properties of Materials

The preceding comments should have pointed out the impossibility of describing the properties of composites definitively at this time. Almost any combination of physical and mechanical properties can be obtained, depending upon the materials employed, their proportions, and the methods of fabrication and application. It may be more useful to cite some maximum properties obtained for various reinforcements and matrix/reinforcement combinations to indicate what can be achieved.

Highest ultimate strengths and modulus values of reinforcements have been obtained on whiskers. As whisker diameter decreases, strength and often modulus increase (Refs. 56–64). Values as high as 6.2×10^6 psi ultimate tensile strength have been reported for sapphire (Al_2O_3) whiskers (Ref. 62), and moduli of elasticity of as much as 145×10^6 psi have been reported for graphite (Ref. 63), over 100×10^6 psi modulus has been reported for tungsten carbide (WC), boron carbide (B_4C), beryllia (BeO), silicon carbide (SiC), and Al_2O_3. One Al_2O_3 whisker sample had an indicated modulus of 300×10^6 psi (Ref. 62). The moduli of filaments are much lower; typical values for B are $55–60 \times 10^6$ psi; SiC has about 70×10^6 psi modulus, while WC has a reported modulus of 104×10^6 psi (Ref. 65).

Composite strengths vary widely, but some examples are: 0.285×10^6 psi for 50 vol % of 9-mil type 355 stainless steel wire in aluminum (Al), 0.154×10^6 psi for 4 vol % of W in nickel (Ni), 0.308×10^6 psi for 24 vol % of B fibers in Ni, 0.1×10^6 psi for 25 vol % of 10-mil beryllium (Be) wire in Al (Ref. 65), 0.098×10^6 psi for 35 vol % of B fibers in Al (Ref. 66), 0.25×10^6 psi for 20 vol % of Al_2O_3 whiskers in nichrome (Ref. 62), 0.44×10^6 psi for 40 vol % of silica (SiO_2) whiskers in Al, and 0.226×10^6 psi for 10 vol % of SiC fibers in Ni (Ref. 67).

It must be noted that the strength and modulus values reported are not considered to be absolute, particularly for whiskers and fibers. Testing techniques are not standardized, and comparisons between laboratories sometimes are not valid. The ASTM is organizing a committee to establish test techniques and standards for fibers and composites (subcommittee VI of committee D-30). Proposed standards for tensile, fatigue, creep, and impact should be prepared by late October 1967.

Whisker strengths are particularly prone to error: first, because the cross-sections usually are not regular; second, because very small errors in area determination or breaking strength result in appreciable errors in strength and modulus values; and third, because there is a certain amount of unintentional preselection of relatively strong whiskers for test (Refs. 68 and 69). Weaker whiskers tend to break up during handling or placement in the test fixtures. This skews the statistical strength distribution toward the high end of the normal distribution curve. This testing bias is less important as the diameter and length of fiber increase. No one has satisfactorily demonstrated the *true* average strength of whiskers, either within a batch or for a group of batches. It is uncertain what proportion of the highest reported strength and modulus values could be obtained under normal conditions of whisker preparation and would be available for composite strengthening. Statistical rather than absolute whisker strength values should be used in estimating composite properties.

Continuous or very long filaments are much less affected by material variation and property scatter; however, as fiber length increases the probable occurrence of a defect or weak spot increases, and measured strength and modulus usually decrease. This length effect normally is unimportant once the filament is formed into a composite, since the reinforcement merely breaks into shorter but stronger sections, each of which usually is longer than L_c. However, there are indications that increasing L/d ratios can give higher strengths. Composites of Ag and Al_2O_3 whiskers with $L/d = 300$ were about 30% stronger than similar composites with whisker $L/d = 100$ (Ref. 70).

Sometimes whiskers are tested en masse by first preparing the composite and then determining the overall strength. Unfortunately, this is also affected by processing techniques, making it difficult to separate materials and process variables. This inability to separate variables is particularly true when the process is not well defined or well established, or when the properties of individual whiskers are extremely variable. Such data probably is useful more for rough screening to determine relative material merit than as a qualitative test.

Fatigue strength and stress-rupture life of composites are greater than that of the unreinforced matrix (Refs. 17 and 71–73). The nature of failure in fatigue is sensitive to the type of test and whether or not the fiber is brittle. Brittle fibers in a work-hardenable matrix, when tested in tensile fatigue, failed by fiber breaks from internal cracks (Ref. 71). Ductile fibers tested in bending failed by matrix cracking; the fibers tended to stop crack growth (Ref. 72). Final failure was mainly by interfacial cracking.

A great deal of development has been done in improving test techniques and developing test equipment, particularly by the Air Force Materials Lab (Refs. 3 and 74). There has been a fair amount of work in test development by the various educational organizations, research organizations, and some of the commercial companies, such as P. R. Mallory, Douglas, General Precision Aerospace, Avco, and United Aircraft Research (Refs. 20, 69, and 75–77). Unfortunately, no one technique or one type of machine has been agreed upon as standard; therefore, all test results, and particularly test results on whiskers, must be evaluated with considerable caution. In view of this, it is important that test data should include the test method used. If it is not included, the results may be suspect, or at the very least may not correlate with other data. A good example of this is the values often obtained on whiskers for tensile strength and modulus of elasticity by a bend test. This data is rarely comparable to that obtained by simple tensile test.

IV. Fabrication

Various fabrication methods will be touched upon briefly to point out what types of processes have been used, and some of their advantages and disadvantages.

For metal matrix composites, casting can be used when the fiber is not attacked excessively by the molten matrix material, when the casting temperature is reasonable, and when no undesirable side reactions occur with the mold or atmosphere. Examples of compatible systems are B in molten magnesium (Mg) and, for short times, molten Al. It is possible to obtain very high volume fractions of reinforcement by this method; as much as 80% has been achieved (Ref. 78).

Vacuum infiltration is a modification of straight casting which gives better flow of the melt around the fibers and higher density final composites.

Powder metallurgy techniques are generally used when the matrix is a refractory material, or when fiber-matrix reactions are excessive. Both cold pressing followed by sintering and hot pressing have been used successfully. Harmful reactions between matrix and fiber can be reduced, but it is hard to obtain high densities, uniform fiber distribution, and controlled fiber orientation (Refs. 5, 6, 20, 62, and 78).

Some composites are made by coating individual fibers or fiber bundles with the matrix material followed by a consolidation process, such as casting, diffusion bonding, rolling, swaging, or extrusion, to achieve densification. One approach is to electroform Ni, Cu, or Ag into a bundle of filaments and apply a secondary densification process to achieve maximum density and strength. However, this technique is susceptible to shadowing under the filaments and has a very low production rate. Voids caused by this shadowing have been demonstrated to be sources of weakness and failure sites (Refs. 29 and 79). Reactions between matrix and fiber are minimal and bonding between fiber and matrix is excellent, except where shadowing has occurred. Both electroless and electroplating techniques have been used successfully to form composites and to coat fibers and whiskers (Refs. 80–86).

Another interesting technique is the formation of graphite-Ni and graphite-Co composites by electroless deposition of the metal on the fiber followed by a swaging operation. The major problem has been embrittlement of the Ni matrix by phosphorus (P) in the bath (Ref. 87).

Vapor deposition and vacuum metallizing also may be used, but both processes require a relatively long time to form appreciably large objects. There may be porosity where the filaments shadow the area below the layer of reinforcement. Costs are relatively high, but matrix purity can be controlled to a greater degree than with other processes (Refs. 20, 88, and 89). Reinforcement contents of 92% have been obtained by vapor deposition followed by diffusion bonding (Ref. 90).

Plasma spraying has similar shadowing disadvantages, although careful control of spraying parameters can eliminate the shadowing. Naturally, the high temperatures required can cause degradation of the fibers, or alloying between fibers and matrix. Deposition rates are higher than by electrodeposition or vapor deposition, and the process is more amenable to successive spraying and wrapping the filaments until the desired size and shape are obtained (Ref. 91).

Extrusion or co-extrusion may be used when both components are compatible and behave similarly during extrusion, and when the fibers are not excessively damaged or broken up during extrusion. Fracture of the fibers, uneven distribution of the fibers both from end-to-end of the extrusion and across the cross-section, and tooling and processing problems have limited the usefulness of this method (Refs. 20, 69, 73, and 92–95). Whittaker Corporation recently reported that they have obtained reinforcements of as much as 95 vol % by this process. Composites such as steel wires/pyrex have been co-extruded (Ref. 20).

Roll bonding has been used successfully for metal matrices when both components may be rolled under similar conditions and are mutually compatible. The amount of deformation is limited by the allowable fiber deformation since the fiber is the harder and stronger component. The choice of hot rolling or cold rolling is dependent upon the materials system. Sometimes fiber properties so restrict the amount of working possible that maximum matrix properties cannot be obtained (Refs. 20, 73, 81, 92–94, 96, and 97).

Diffusion bonding has been used in systems where interactions between fiber and matrix are undesirable, but good bonding is desired. High temperature properties are improved because fabrication reactions are suppressed. Diffusion-controlled deterioration in service will require appreciable time before significant reductions in properties occur. The wrought properties of the matrix are retained much more readily by this process. There is some limit to the size and shape of parts which can be fabricated*, and only metal-matrix systems can be formed in this way. Addition of diffusion bonding aids at the interface may be necessary when bonding more reactive metals like titanium (Ti), zirconium (Zr) or Al. Preliminary data indicates that fatigue strength and notch-tensile strength of diffusion bonded Al-Be composites is excellent (Ref. 98). Not all useful matrix materials can be formed successfully by this process (Refs. 20, 67, 89, 96, 97, and 99–101), but it is becoming the most widely used fabrication technique.

*However, it is notable that the largest metal-matrix composites prepared to date are 1 × 8-ft plates of Al formed by diffusion bonding plus a slight final rolling reduction of about 4%.

High energy rate processes, such as Dynapak or explosive compaction, also have been used to form metal matrix composites. There is some doubt as to the amount of fiber fracture which would occur during fabrication (Refs. 102 and 103). Alignment of fibers is a problem, as is uniformity of fiber distribution. Advantages include the good densification which can be obtained and reduced fiber-matrix reactions (Refs. 104–110). Relatively complex shapes would be difficult to form and may require costly tooling.

Unidirectional solidification of eutectic and monotectic alloys has been used to prepare composites which have the reinforcement fiber formed as an integral part of the casting. The size and spacing of the fibers can be varied to a certain extent by controlling the growth conditions. Fiber shape appears to be determined mainly by the composition of the eutectic. Rods are favored below about 28% fiber phase, while platelets are favored above 28% fiber phase (Ref. 107). Secondary working of eutectic composites has been demonstrated: $Al\text{-}Al_3Ni$ composites have been forged and extruded successfully and have been cold rolled up to 75% perpendicular to the fiber direction (Ref. 108). The advantages are: excellent fiber-matrix bonding, chemical compatibility of fiber and matrix, ease of uniaxial fiber alignment, and composite formation in a single step. High strengths, good fracture toughness, and fatigue strength are obtained. The fracture and fatigue behavior are influenced by the shape and spacing of the fibers, which can be controlled by altering growth conditions; thus, it may be possible to grow eutectic composites specifically for fatigue or fracture-toughness limited applications (Refs. 109 and 110). Disadvantages include the relatively limited number of materials systems (eutectics or monotectics) which can be used, the need for high purity starting materials, and the relatively slow production rates (Refs. 107, 111, and 112). Simple shapes such as bars and turbine blades have been made by this process (Refs. 20, 107, 111, and 112). Ingots of 3-in. diameter × 1-ft long are produced regularly (Refs. 107 and 110) for further fabrication. Recent work suggests that eutectic-like fiber reinforcement is possible over a wide range of alloy compositions (Refs. 113–115).

An interesting and novel type of composite laminate, which was recently developed and may be useful where isotropic properties are desired, is vacuum metallized B on a 0.5-mil polyamide substrate (in this case Kapton) to form a layer from 0.2 to 0.4-mils thick (Ref. 116). The resultant film is isotropic and possesses the stiffness and strength characteristic of boron composites. Layers of the film may be laminated to form shapes, such as cylinders, corrugated flats, or plates. Since the process and material are in an early stage of development, little property data is available.

The most extensively used composites are the resin matrix, i.e., epoxies and polyamides, reinforced with non-metallic filaments, usually fiberglass. These materials are well developed, and have been used extensively in the fabrication of a wide variety of products ranging from car bodies, boat hulls, tanks, piping, gliders, propellers, and rocket cases. Both domestic and foreign organizations have been active in this field (Refs. 117–123). Since resin matrix systems are well developed and understood, it is relatively simple to substitute more advanced filamentary materials such as B for the fiberglass reinforcement, and fabricate components using standard techniques. This has been done in the developmental fabrication of a B/epoxy wing box, and a B/epoxy stabilizer section for the F-111 (Refs. 124–127). While these composites do not offer the high shear strength and modulus or tensile strength possible in metal matrix composites, they are more developed and have actually been made into full-scale components and tested.

V. Joining

This is one area where there is a need for much more information. Unfortunately, the type of joining technique used is dependent upon the material system and its fabrication history, and neither of these is at all standardized. In resin matrix systems, joints are simply made by adding more resin to the joint area, perhaps with some additional reinforcement filaments. Some metal matrix systems can be welded or brazed, but others may be damaged by the heating and cooling cycle. Absence of reinforcement from the joint area also may prove to be a critical problem. Harvey Aluminum reports that only 50% of composite strength could be obtained when steel-reinforced aluminum was joined by brazing specially prepared joints (Refs. 19 and 128). If the joints were integral designed, efficiencies of 85–90% were obtained (Refs. 19, 20, and 128). North American has done some studies on joining Be-reinforced Al by riveting, TIG welding, and EB welding. Riveted joints must be designed with two to three times the edge-to-rivet distance of normal joints. Welding also shows promise of providing good joint strength (Refs. 98 and 129). So far, most investigators are willing to acknowledge that a joining problem exists and may be a potential limiting factor in the usefulness of some systems, but they are content to cross that bridge later (Refs. 20, 82, and 130).

VI. Present Materials Availability

No composite material can be considered to be *available* in the sense that steel rod or beryllium sheet is available. The materials are tailored for each application and are essentially custom made. The closest to a stock item for any composite is pre-impregnated glass fiber in a resin matrix in tape form. The B fibers in resin matrices are available on order, with reasonable lead times (6–8 wk). Some whisker-metal and fiber-metal composite fibers also are available on special order. Plates of steel and boron-reinforced aluminum are available with a 4–6 wk lead time. All of these would require some further processing into useful shapes.

VII. Anticipated Future Progress

In addition to B, continuous fiber materials such as SiC, graphite, titanium diboride (TiB_2) and B_4C are becoming available in significant quantities. Besides B, only SiC and graphite have reasonable strength, modulus, and reproducibility of properties at this time; their status is that of B about 2 or 3 yr ago, but improvements are being made. Further increases in fiber properties, improved reproducibility and reliability, and reductions in price are likely.

Fabrication presents the greatest problems and the greatest challenge to composite technology. There are a number of potentially satisfactory fabrication techniques and matrix materials. However, it is generally conceded that more effort at this time should be devoted to understanding and improving process techniques for composites. This area is receiving some attention, and there are a number of programs directed toward solving this problem (Refs. 20, 25, 54, 57, 64, 126, and 131). But most of the fabrication techniques under study are designed to provide test samples which will demonstrate that the process is feasible. These programs which provide relatively small samples do not guarantee that usable material in engineering sizes can be obtained, although some successes have been achieved; for example, Harvey Aluminum has made steel reinforced aluminum composites in sheets as long as 8 ft (Refs. 19, 97, and 132), and United Aircraft reports that they have made unidirectionally solidified eutectics as much as 3 in. in diameter and over 1 ft long (Refs. 20, 107, and 110). Accordingly, composite application programs must focus more on particular components, as the work done on helicopter rotor blades, wing boxes, or compressor blades (Refs. 2, 3, 127, 133, and 134). Some of the boron-epoxy components are being flight tested as wing sections on the F-111 and have shown excellent results (Ref. 133).

It is likely that a resin matrix composite, probably boron reinforced, will be developed and applied for aircraft components within 1 to 2 yr. This requires only a slight additional advance in the technology of glass-epoxy composites. Metal matrix composites are not as advanced, but development of flight hardware has begun, and results should be available by early 1968 (Refs. 126 and 133). At this time, metal matrix composites are about 1 or 2 yr behind resin matrix composites; applications to actual flight components probably are from 2 to 4 yr away.

Whisker reinforcement is in a relatively early stage of development, and compares to that of metal-matrix continuous-fiber reinforced materials of about 2 or 3 yr ago. However, the whisker composite systems have benefited greatly by the analytical work and fabrication experience which has been developed on other composite systems; therefore, it should not take long to produce actual flight quality hardware using whiskers. The Air Force is currently funding programs in which whiskers, both SiC and Al_2O_3, will be used for spot reinforcement of resin and metal matrix composite systems (Refs. 20 and 126). The advantages of this type of application are: the reinforcement may be placed only in that area where additional strength is required; the proportionate cost of the whiskers is considerably less; whisker misalignment problems are minimized; and there is the advantage of working with a system already well developed and well understood, as B epoxy. It is anticipated that these programs will provide test data, and actual pieces of developmental hardware within the next 1 to 2 yr. The use of small quantities of whiskers for reinforcement of cast structures is practical at this time, and has been performed by several investigators at the Naval Ordnance Laboratory and the General Technology Corporation (Ref. 20). It should be possible to have flight hardware made of whisker reinforced metal matrix and epoxy matrix materials within 3 to 5 yr. This assumption presupposes that there will be a rather extensive effort with adequate funding, both from industry and government, and that developments will be pursued at approximately the same rate and with the same level of interest as that shown in the past 3–5 yr in boron fiber composites.

VIII. Current Programs

There are many projects in the area of filament and whisker composites, both metallic matrix and nonmetallic matrix systems. It is beyond the scope of this review to go into detail on every study which has potential application for spacecraft structures. However, some of the more important or more well-developed programs that have the greatest potential for the near future will be discussed.

The most advanced filamentary material is B, which is plated on a ½-mil tungsten substrate by chemical vapor deposition. This work is being done by Texaco, Avco, and United Aircraft (Refs. 3, 6, 7, and 20). Less well-developed but more interesting because of lower overall density is a B fiber which has been formed by deposition from a metallorganic boron bearing gas onto a graphite coated silica substrate. This work is being done by General Electric and Texaco (Refs. 20, 135, and 136). This deposition system permits more rapid deposition rates on a cheaper, lower-density substrate and should provide lower cost filaments. A number of other materials also have been prepared in filament form using the same basic techniques. General Technology Corp. has prepared SiC, B_4C, and TiB_2 (Ref. 137). Aerospace Chemical Systems has prepared B_4C by pyrolysis of carborane-10 (Ref. 138). Hough Laboratory has made B_4C from organoborones (Ref. 139). General Electric also has made B_4C filaments by vapor deposition (Refs. 136, 140, and 141). Marquardt has deposited continuous SiC filaments on W with B added to strengthen the SiC. United Aircraft and Texaco also are preparing continuous SiC fibers by vapor deposition (Refs. 142 and 143). Workers at the University of Leeds have prepared SiC by decomposition of organo-silanes (Ref. 144). Graphite fibers and yarns are being developed by Hitco, Rolls-Royce, and Union Carbide, using pyrolysis of organic fibers (Refs. 8, 20, 88, 126, and 145). Layered composites of molybdenum (Mo) and TiB_2 have been prepared by vapor deposition by General Technology Corp. (Ref. 146). If successful, this type of fiber could be designed for specific strength, modulus or physical properties (i.e., resistivity). Of these newer filaments, only silicon carbide is reasonably well-developed and has been investigated extensively, either as a fiber or in a matrix system.

Metallic wires are of interest primarily because the cost of fabrication is much lower than for materials which are vapor deposited, production rates are greater, and the costly vapor deposition process and substrate are eliminated. Compatibility problems between substrate and deposit also are eliminated. A number of metallic wires have been prepared either on a production or laboratory scale. These include such materials as beryllium, stainless steel, molybdenum, titanium, superalloys, nichrome, and a number of other refractory and reactive materials such as zirconium (Refs. 1, 57, and 147–154). Aluminum and magnesium also have been investigated; in general, they are too low-melting to be of very great interest. Crucible Steel, Brush Beryllium, National Standard, General Astrometals, Hoskins, Monsanto Research, and Berylco (Refs. 147–156) have been active in this field. One material which is of considerable interest is a superalloy type of stainless steel containing cobalt, vanadium, and chromium. This has been developed recently by Crucible Steel; 5-mil wires of this material have achieved tensile strengths as high as 0.525×10^6 psi at room temperature, and retained useful strengths of 0.310 to 0.315×10^6 psi at temperatures as high as 1100°F (Refs. 147 and 148).

Many organizations are working in the area of improving both the properties of the metallic wires and methods of incorporating them into a matrix. These include Harvey Aluminum, Avco, TRW, General Dynamics Convair, Cevite, General Electric, International Harvester division of Solar, General Technology Corp., Tyco Labs, North American, Melpar, United Technology Corp., Allison Division of General Motors, Hoskins, Battelle, IIT Research Institute, and others (Refs. 20, 73, 81, 83, 84, 92, 95, 96, 100, 102, 126, 134, 154, 155, 157, and 158).

Whisker formation and whisker properties are becoming the objects of an increasing study and a great deal of effort is being expended in trying to understand both the whiskers and the techniques for fabricating, handling, testing, and forming whiskers into composites. A list of the organizations which are working in the area of whisker formation includes General Electric Space Sciences Lab, Carborundum, P. R. Mallory, Thermokinetics, Avco, United Aircraft, the Air Force Materials Lab, the English Ministry of Aviation, IIT Research Institute, and a number of others (Refs. 5, 20, 25, 57, 85, 118, 126, 131, and 159). The only whiskers available in commercial quantities at this time are Al_2O_3, B_4C, and SiC; although other whiskers have been prepared on a laboratory basis, and are available in research quantities. These programs in fiber, filament, and whisker formation are primarily sponsored by the Air Force Materials Lab at Wright Field with additional work sponsored by NASA, the Army, and, to a lesser degree, the Navy. They are surveyed in Refs. 126 and 130.

Most of the eutectic composite work is being done by United Aircraft; other organizations studying these material systems include MIT, RCA, Lockheed, and Tyco (Refs. 107–115, 160, and 161). Composites prepared include $Al\text{-}Al_3Ni$, $Ta\text{-}Ta_2C$, $Cb\text{-}Cb_2C$, $Al\text{-}CuAl_2$, InSb-NiSb, Al-Be, $Ti\text{-}Ti_5Si_3$, Ni-NiB, Ni-NiBe, Ti-TiB, Pb-Sn, Bi-MnBi, and $NiSi\text{-}Cu_3C$ (Refs. 107–115, 160, and 161). Preliminary data indicates that $Al\text{-}Al_3Ni$ can be cold rolled successfully, and that this gives increased strength (Ref. 162). Turbine buckets of Ni-NiMo have been grown directly from the eutectic by United Aircraft (Ref. 107).

The preceding comments on current studies for reinforcement and composite materials development and preparation necessarily are quite sketchy. It would be impractical to comment extensively on the vast number of programs which are now being undertaken in this area. In an Air Force report prepared by the General Electric Space Sciences Lab and covering the period from April 1964 to April 1965, there were over 500 references and several hundred patents in the bibliography, and over 60 personal contacts with research institutes, corporations or other organizations doing work in the general field of composites (Ref. 29). General Electric recently issued an updated survey covering the work from April 1965 to mid 1966. This paper includes almost 800 references in the bibliography, more than 200 patent references, and almost 100 reports of contacts with various investigators in the field. Approximately 80% of these references are related to work which was done only in the past year (Ref. 54). A more recent paper noted that the authors had examined 1187 references; more than a third of these dealt with mechanics of composites (Ref. 47).

Although the emphasis in composites is being directed more towards applications and away from materials development, the overall level of funding and effort remains substantial. In view of the many organizations involved, using a variety of approaches, it is only a matter of time before successful structural applications are achieved. There seem to be no insurmountable problems inherent in composite materials which will prevent this success; it is more a matter of advancing from the technological base already established. The major uncertainties are the time required to obtain useful structural components, and the identity of the first material system to achieve this state of development.

IX. Summary of Conclusions

As a result of this literature search and survey, a number of conclusions were drawn regarding the applicability and status of composite materials for structural use on spacecraft. They are as follows:

(1) Composites possess significant advantages in strength-to-weight ratio and modulus-to-weight ratio, as compared with presently used spacecraft materials.

(2) There are no standard or generally recognized test methods for establishing the physical and mechanical properties of composites or reinforcement materials. Attempts are being made to develop test methods and standards for both composites and reinforcements.

(3) Composites offer a number of advantages compared to other structural materials, including greater design freedom and flexibility. In some instances, only the use of composites will completely satisfy structural or design requirements.

(4) The field of composites presently is in a state of flux; no single material or fabrication process has been developed which is obviously superior for a wide range of applications.

(5) A large amount of effort is being devoted to composite development by many organizations. This broad attack already has achieved significant advances in materials and process development, and undoubtedly will result in successful development of advanced composite systems if the level of effort is maintained.

(6) In spite of this broad and extensive attack, a number of major problems remain: high cost of whiskers and fibers, difficulty in obtaining alignment and uniform spacing of the reinforcement, harmful reactions between fiber and matrix, thermal expansion mismatch between fiber and matrix, general difficulties in fabrication and processing of composites, development of joining techniques, and correlating fiber and composite test data from various sources.

(7) Development currently is being directed more towards applications rather than towards improvement of existing materials or development of new materials.

(8) The nonmetallic matrix composite utilizing high modulus fibers which is closest to practical application is boron-epoxy.

(9) The metallic matrix composite which is closest to practical application is aluminum reinforced with boron or steel.

(10) Metal matrix composites have demonstrated appreciably greater fatigue life, creep strength, and stress-rupture life than the unreinforced matrix.

(11) Greatest effectiveness in utilization of composites requires an interdisciplinary effort which combines materials, structures, and design technology at the inception of the design.

(12) At this time, no metal-matrix composite system is sufficiently developed for spacecraft structural applications. The boron-epoxy systems are well developed, but of more limited utility in spacecraft structures.

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Appendix

Abstracts of Reports and Papers on Advanced Composites

The following abstracts generated by the author include comments on the report or paper and applicability of that particular composite development to structural uses or to other reports on similar subjects. Therefore, these abstracts will not be identical to the original authors' abstracts. The abstracts are presented in alphabetic order by author and a cross-index is provided for ease of locating abstracts of reports or papers from one corporate, conference, or periodical source.

1. Stress-Wave Propagation in Composite Materials

Abbott, B. W., Broutman, L. J.

***Exp. Mech.*, pp. 383–384, July 1966**

Thomsen-Abbott Construction Co., IITRI

Longitudinal stress waves were used to determine the modulus of elasticity of composites using wave propagation velocity. Results compare well with literature values. Propagation velocity varies with fiber orientation. The technique works for composites and homogeneous materials, both high and low modulus in or across fiber direction.

Fibers	Matrices
Steel, glass	Epoxy, glass

2. Micro- and Macromechanics Analyses of Composite Materials

Adams, D. F.

(Paper G-2, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Aeronutronic

Mathematical analysis of composites must take into account the properties of the material and the effects of simplifying assumptions. Macromechanics considers the gross behavior of the composite, and assumes that the elastic properties are known and the material is quasi-homogeneous. Micromechanics attempts to predict macrobehavior by knowing constituent properties and geometrical factors, such as fiber spacing and shape, volume %, etc.; this involves studying interactions among fibers, matrix, and interfaces. Examples of macromechanical and micromechanical analyses are given and discussed. The effects of fiber shape, volume %, and spacing are derived analytically. Typical mechanical analyses and some of the problem areas encountered in using them are discussed.

3. Longitudinal Shear Loading of a Unidirectional Composite

Adams, D. F., Doner, D. R.

***J. Compos. Mater.*, Vol. 1, No. 1, pp. 4–17, Jan. 1967**

Aeronutronic

An analytical study of unidirectionally reinforced composites in shear, and a comparison of theory with available data are given. Matrix was assumed to be elastic with regular fiber array. The effects of various fiber shapes and spacing on shear stiffness are calculated and plotted for B in epoxy. The micromechanical behavior can be described in detail for this system. Local stress concentrations can be very high, but in real composites local yielding occurs, redistributing the stresses. This paper is a companion to the following paper.

Fiber	Matrix
C, B, glass	Epoxy

4. Transverse Normal Loading of a Unidirectional Composite

Adams, D. F., Doner, D. R.

***J. Compos. Mater.*, Vol. 1, No. 2, pp. 152–164, Apr. 1967**

Aeronutronic

Analytical study of unidirectional reinforced composite is given, using the theory of elasticity to treat a regular array of elastic fibers in an elastic matrix. Transverse normal loading is considered, and theoretical and experimental results are compared. The method of analysis is described, along with amplifying assumptions, i.e., regular spacing and perfect bonding. Temperature changes also are taken into consideration. The effects of varying fiber spacing, vol% fibers, fiber shape, and matrix properties are considered. Theoretical and measured transverse stiffness are given for various epoxy matrix systems; agreement is generally good. The theory does not permit comparison with strength data at this time.

Fiber	Matrix
C, B, glass	Epoxy

5. Metal Matrix Fiber Strengthened Materials

Adsit, N. R.

GDC-ERR-AN-867, Dec. 1966

General Dynamics/Convair

Ni was electroformed over B fibers to form composites. Up to 12% B was contained in the Ni; increased strength was observed at all reinforcement levels, even though the calculated critical reinforcement level was 4%. After 6½ h at 1400°F, extensive Ni-B reaction was seen. It is concluded that the Ni-B system should not be heated above 700°F, or deleterious reactions will occur. Graphite cloth could not be Ni-plated successfully, but separation into fibers permitted formation of some composites with rather poor properties. Only one layer of B could be electroformed; additional layers led to excessive Ni buildup. B fibers also were bonded to Ti and Al by hot rolling at 900°F for the Al and 1800°F for the Ti. Both composites were reduced 3 times, 10% each time. Rolling parallel to the fibers caused them to fracture into shorter lengths, while rolling perpendicular to the fibers seemed to be successful, at least in the case of Ti. The Al sheets did not bond. There was no apparent difference in fiber-matrix adherence whether the B was pretreated or not. Ti with graphite fibers was produced the same way, and intentionally reacted to form TiC. Both B and C in Ti reduced the matrix strength. It was suggested that, since at most 12% fibers were present, the fiber loading may have been too low. (The Ti-fiber reaction may have been a greater contributing factor.) A brief review and literature survey also is given.

Fibers	Matrices
B, graphite	Ni, Ti, Al

6. Metal Matrix Composite Materials

Adsit, N. R.

GDC-ERR-AN-1054, Jan. 1967

General Dynamics/Convair

Composites of B-Ni, Cu-C, Cu-B, and Ni-SiC were prepared by electro-deposition of the matrix material onto the fiber. Composites of B-Al, B-Ti, Mo-Ti, Mo-Ni, and SiC-Ti were formed by diffusion bonding; in the SiC-Ti system diffusion bonding was by hot rolling at 1800°F in 3 passes of 10% @. SiC-Ti also was hot pressed from powders, but with poor results. Ag electroplating on the Al before diffusion bonding was unsuccessful. Spacing of the fibers during diffusion bonding was a problem area. Properties of Ni-B were determined to −320°F. Notched to unnotched strength was very good. The poor strength of B-Ti was attributed to reaction of the B and Ti (previously attributed to low fiber content). Machining by EDM, drilling, and grinding was successful. Tensile specimens showed a gauge-length effect: shorter samples had lower strengths. Ni-SiC composites with 2% fibers were weaker than unreinforced electroplated Ni. Aging Al-B for 141 h at 400°F did not give any reaction zone. Roll forming and break-bending of Al-B also was successful; roll forming was done to an 8-in. radius with the rolls parallel to the fibers. Single riveted and spotwelded shear samples were made and tested successfully. Tubes and curved plates of B-Ni were electroformed.

Fibers	Matrices
B, SiC, C, Mo	Al, Ni, Ti, Cu

7. Reinforcement of Ni with High Strength Filaments

Ahmad, I., Greco, V. P., Barranco, J. M.

***J. Compos. Mater.*, Vol. 1, No. 1, pp. 18–29, Jan. 1967**

Watervliet Arsenal

Up to 35-vol% W in Ni and 11-vol% Al_2O_3 whiskers were formed into composites by electroforming. Other systems, such as B and glass filaments and SiC whiskers, have been formed successfully, but they are in an early stage of development and the data on properties are unavailable. Filament and whisker properties are described, as are experimental fabrication and examination methods. Annealing for ½ h at 650°C was required to relieve residual stresses in the matrix. Void formation in W-Ni was reduced by using finer wires, and was eliminated by annealing. In B-Ni, there was irregular fiber spacing and some bridging, causing voids; layer by layer deposition eliminated the voids. The small glass fibers did not cause voids because of their small diameter. Nature of composite fractures is described and discussed. W-Ni and Al_2O_3-Ni failures are due to weak interfaces in most cases, but B-Ni and glass-Ni failures initiated at voids. More work is needed on B and glass fibers and SiC whiskers in Ni to get a higher vol% into the matrix by electroforming and to avoid voids.

Fibers	Matrix
W, Al_2O_3, B, SiC, glass	Ni

8. A Study of Low Density, High Strength, and High Modulus Whisker Filament and Laminar Composites

Alexander, J. A., Macklin, B. A.

NASW 1347, May 25, 1966

General Technology Corp.

Layered composites of Mo and TiB_2 were formed by vapor deposition of Mo from MoF_6 at 850°C and of TiB_2 from BCl_3 and $TiCl_4$ at 1050–1100°C on graphite. No data on properties given. Ni was electrodeposited on SiC yarn and gave individual fiber strengths of up to 0.327×10^6 psi. Best results were obtained when a ½-mil Ni wire was spun into the fiber yarn, and electroplating done on the composite. Tensile strength varied from 85 to 327×10^3 psi. About 8–10% whiskers were present in two of the samples tested. Composites of 2-, 3-, and 4-mil W in Ni were made by molecular forming. Up to 52-vol% fibers were present. In general, both strength and modulus followed Rule of Mixtures curves; there was appreciable scatter in both values. B-Al composites were formed by winding B on a mandrel in a bath and by electroplating Al on it. B-Mg 69% composites were formed by vacuum infiltrating B with Mg.

Fibers	Matrices
B, Mo, W, SiC	TiB_2, Mg, Ni, Al

9. A Study of Low Density, High-Strength High-Modulus Filaments and Composites

Alexander, J. A., Shaver, R. G., Withers, J. C.

NASA CR-523, July 1966

General Technology Corp.

Ni-matrix composites were made by electrodeposition of Ni on whiskers and filaments. In the case of filaments, the reinforcement was wound onto a mandril and coated simultaneously. Composites of W and B in Ni exceeded Rule of Mixtures strengths by 17–35%. Part of this is due to the wide variation in B UTS (20%), and partly to the failure of test uncomposited B at surface defects which were not critical defects in the composite. Al matrix whisker composites were made by blending Al and Si-powder with whiskers in a blender, and hot pressing. Cold pressing and sintering was unsuccessful. Whisker alignment was random, and improvement in composite strength was obtained up to 11.4-vol% whiskers. Whisker strength was low (0.4×10^6 psi) and accounts for part of the ineffectiveness. Ni matrix composites were made by electrodeposition of Ni in a bath in which the whiskers were kept in suspension by agitation, and by electrodeposition of whiskers dropped onto a cathode. Samples were annealed at up to 1000°C, but in all cases, the composites were much weaker than the parent metal.

Fibers	Matrices
Al_2O_3, SiC, B, B_4C, W	Ni, Al-Si

10. The Elevated Temperature Reactivity in Boron Metal Matrix Composites

Alexander, J. A., Sturke, W. F., Chuang, K. C.

(Paper F-8, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

General Technology Corp.

Composites were formed by electrodeposition of Ni and Al, with and without subsequent hot pressure bonding. B fibers also were precoated with Ni before coating with Al and hot pressing. Mg composites were made by hot pressing Mg powder with B fibers, diffusion bonding B and Mg foils, and liquid infiltration. Post-formation heat treatments of 300–1000°C were given to the composites. After 1 h at 500°C and above, the Ni matrix composites were much weaker than as-deposited composites; 800 and 900°C treatments caused the samples to crumble. Al-B samples showed drops in strength as a function of exposure temperature when Ni-coated fibers were electrodeposited with Al. When the Ni coat was eliminated, degradation was much less at higher temperatures. No reaction between B and Mg was observed, even when the Mg was molten for 10 min. Good bonding was found in the B-Mg samples. Strength decreased as a function of time at 600°C for B-Mg, but even after 4 h, it was more than ½ the original values. B is very reactive with most structural metals. Ni forms a weak boride interface and rapidly degrades with exposure to high temperatures. Al and Mg are the only matrices useful for elevated temperature applications with B fibers; their degradation is not accompanied by an observable interface reaction. Short term protection of B may be possible with Ag or Cr barrier. Mg-B systems can be considered for fabrication into rods and panels for non-corrosive uses at the present time; bonding in this system is excellent. Similar bonding in B-Al is desirable.

Fiber	Matrices
B	Al, Ni, Mg, Ti

11. Destruction of Silicon Nitride Whiskers by Reaction with Metals at High Temperatures

Andrews, E. H.

***J. Mater. Sci.*, Vol. 1, No. 4, pp. 377–382, Apr. 1966**

Queen Mary College, London, England

Fiber mats of Si_3N_4 whiskers were vacuum metallized with 100–200 Å of Al and Ni, and were exposed at 560°C for 15 min and 630°C for 30 min in the case of the Al, and at 1000°C for 15 min in the case of the Ni. Both metals attacked the substrate with a certain periodicity of the attack noted. Examination of the whiskers was by electron microscope. At 560°C, attack by Al was extensive; but at 630°C, the whisker was completely disintegrated. Ni formed *limpet* nodes on the Si_3N_4 whiskers and these nodes were eating into the whisker surface. It is suggested that the periodicity of attack was not related to the crystallographic orientation in the case of Ni, but perhaps is related in the case of the Al. The results obtained when a whisker is embedded in a continuous matrix would not necessarily be the same.

Fiber	Matrices
Si_3N_4	Al, Ni

12. Report on B Filament Properties: Strength, Uniformity, and Fracture Characteristics in B Filaments

Anonymous

Texaco Experiment, Appendix II, 1965

Results of tensile tests on over 2500 samples from 5 lots of B fiber are given. Failures initiate at surface flaws (caused by inclusions on the substrate or in the B), at the inclusions themselves, at crystallites in the B, and at interfacial flaws, such as (Kirkendall) voids, as a result of residual stresses at the interface and as a result of crack propagation from defects in the substrate. Inclusions can come from the improper cleaning of substrate or contamination of the Hg reactor seals. Reduction of inclusions will reduce surface nodules and imperfections, and improve fiber strengths. Crystallites can be eliminated by control of the processing. Improving the surface finish of the W substrate will reduce interfacial weaknesses, but some residual stresses seem inevitable due to the conversion of the W to boride. Wide variations of tensile properties both within lots and between lots were noted. Poor lots had their average strengths increased and their scatter somewhat reduced by surface etching; the percentage of surface-induced failures decreased very greatly for poor lots, and noticeably for good lots. Good lots have strength distributions skewed to the high side, and are less affected by etching. Great variations in strength along the length of fiber are possible; causes are contamination of the Hg seals after short lengths of substrate are drawn through the Hg pools, and W surface imperfections. Process variables are a factor also.

Fiber
B

13. N-S Rocket Wire Technical Data

Anonymous

Technical Data Sheet No. SWT-101, Jan. 1965

National-Standard Co.

Strength levels are described as obtained by special fabrication process in steel-base wires. The 4-mil wires had following strengths: AM355 – 0.5 $\times$ 10^6 psi; 302SS – 0.4 $\times$ 10^6 psi; and steel – 0.575 $\times$ 10^6 psi. Also, fatigue and stress corrosion properties are given.

Fibers
302SS, AM 355, carbon steel

14. Report of the Ad Hoc Committee on Interface Problems in Fiberous Composites

Anonymous

MAB-214M, Nov. 1965

Materials Advisory Board

Report discusses problem area in the interactions of interfaces, matrix, and fiber, and recommends investigations to improve understanding of interfacial reactions and permit composite design on a more realistic basis. Factors discussed included stress transfer, surface activity, surface imperfections, interdiffusion, chemical activity, and the effects of large radii of curvature of fibers on enhancing chemical potential. Absorption of impurities, ductility of interface, and attack at the interface were also discussed. Degradation of fiber, matrix, or interface during processing or service also was covered.

Fibers	Matrices
All	General

15. Review of Recent Developments Whisker-Reinforced Metals

Anonymous

Bulletin No. 1, June 30, 1966

Thermokinetics

SiC and SiN whiskers have been aligned in yarns and impregnated with epoxy. Significant increases in strength and modulus were obtained. SiC whiskers in Al alloys have been observed to knit the grains together. As little as 3–4% whiskers has a significant strengthening effect. Whiskers reduce creep in Ni alloys. At 800°F, Al with 10% whiskers is 2–3 times stronger than 7075 Al. Adding 4% whiskers to Be increased UTS 32% and modulus 10%. SiC-Ni composites have been formed by electrodeposition. SiN whiskers have been formed into aligned yarns by spinning in a liquid. The 4% additions of random whiskers to epoxy gave 38% greater interlaminar shear.

Fibers	Matrices
Al_2O_3, SiC, AlN, SiN	Al, Ni, Be, epoxy, Ti

16. The Freeze-Coating of Filaments

Arridge, R. G. C., Heywood, D.

***Brit. J. Appl. Phys.*, Vol. 18, pp. 447–457, Apr. 1967**

Rolls Royce

Fibers of glass or other materials may be coated by passing them through a bead of molten metal. Coating was by a freezing mechanism rather than boundary layer growth. Predictions of coating thickness as a function of melt superheat and time in the melt are given, and agree well with experiment. Experimental methods for coating and results are described. Formation of blobs at irregular intervals is believed due to oxide layers on the Al melt. Use of reducing gas above and below the melt eliminated the blobs and surface ripples, but reduced fiber strength. This reduction in strength is due to elimination of the oxide layer which forms at the surface of the glass, thus allowing attack of the glass by the Al. When reducing gas is introduced below the orifice, only strength is increased; blobs and ripples are eliminated. Variations in strength of coated fibers is shown to be due to interface reactions, not variations in total fiber diameter. Strength of the fibers was found to depend upon gauge length.

Fiber
Glass

17. Boron-Filament Reinforced Plastic Composites for Aircraft Structures

Atkin, H. P.

***Metals Eng. Quart.*, Vol. 7, No. 1, pp. 17–22, Feb. 1967**

North American Aviation

Article describes program for fabrication and testing of T-39 wing box. This program included analytical studies of composite applications, development of processes and methods of predicting materials properties, and design fabrication and testing of two wing-box beams. Effect of fiber orientation on strength was studied; when continuous fibers were used, the 45° drop was not noted. Actual values are affected by aspect ratio, holes, and edge fixity, which are insufficiently well known. Wide samples gave higher strengths at all orientations than narrow ones. Reversed bending and tension-tension fatigue values were excellent. End attachments during tensile test proved a problem, and results were lower than expected. Box beam failure was at 60% of design load due to stress at the attachment.

Fiber	Matrix
B	Epoxy

18. The Effect of Fiber Volume Fraction and Interfacial Bond on the Fatigue of Al Reinforced With Stainless Steel Wires

Baker, A. A.

***Appl. Mater. Res.*, Vol. 5, pp. 143–153, July 1966**

Rolls Royce

This paper is an example of the importance of specifying test methods and composite specifications. Unlike the R. K. Ham and T. A. Place paper (see entry No. 118), the fiber was ductile, fatigue testing was in reversed bending, and the results were greatly different. Fatigue cracks did not initiate at the fiber and propagate through the matrix, but were stopped by the fibers. The precoated wires were hot pressed at different temperatures to give different amounts of interfacial reaction. Samples with no reaction and excessive reaction had the lowest number of cycles to failure at constant stress; those with some interface reaction had the best lifetime to failure. At stresses below the endurance limit, strain increased very slowly; but at stresses above the endurance limit, strain increased rapidly. Fibers with unreacted interfaces tended to fail at the interface, while those with reacted interfaces tended to turn the cracks from the fiber. As processing temperature increased, the reaction zone thickened and more cracks through the fibers occurred. The theoretical plastic strain gauge of the matrix was used to take into account variations in fiber content and fatigue stress, although the ratio of fatigue stress to theoretical tensile stress also fit the data points. Fatigue failure occurred by interfacial crack propagation, which causes a lowering of flexural modulus. It is suggested that this type of composite would be insensitive to notches in fatigue. Limited interfacial formation was better than too little or too much.

Fiber	Matrix
SS	Al

19. Summary of the 9th Meeting of the Refractory Composites Working Group

Bartlett, E. S., Ogden, H. R.

DMIC Memo 200, Mar. 1965

Battelle Memorial Inst.

General discussion of programs in the refractory materials area is given. W in MgO resisted recrystallization for 24 h at 2750°F. Alfred Univ. is studying ceramic composites, and found that matrix cracked when it had a high thermal expansion coefficient, even though it was reinforced by metal or alumina wires or fibers; these composites had good thermal shock resistance. Wetting between Ni and alumina was aided by additions of Ti, Zr, and Cr.

Fibers	Matrices
W, Al_2O_3, B_4C	Ni, MgO

20. Summary of the 10th Meeting of the Refractory Composites Working Group

Bartlett, E. S., Ogden, H. R.

DMIC Memo 204, May 1965

Battelle Memorial Inst.

General Technology Corp. made Ni-matrix composites reinforced with B and W by molecular forming; 24% B gave 0.308 × 10^6 psi composite. Also, they made Ni reinforced with graphite cloth and Al_2O_3 to demonstrate low temperature, ambient pressure process. Battelle-N.W. reported on strengths of Ni-W wire composites formed by Dynapak; they found 10-vol% aligned W gave about 60% better RT strength and 6× strength at 1925°F. Random wire strengthening was low, but very small percentages were used. Harvey Aluminum made steel-reinforced Al by diffusion bonding Al alloy sheets; also, they made Al sheets with Be wire reinforcement, but modulus UTS was not exciting. Allison made mullite with 20-vol% Mo wires, and found modulus of rupture 50% better than theoretical; oxidation behavior was very poor/bad. Atomics International found that BeO fibers of 7-mil diameter had tensile of 2.6 × 10^6 psi. Whiskers were grown from molten flux. A ½-mil W wire was carburized by 30 s 2590°F passage through 2% methane 98% H_2; tensile was 0.106 × 10^6 psi and modulus was 0.104 × 10^6. IITRI made polycrystalline Al_2O_3 fibers by extruding-drying-firing paste mix; modulus was 70 × 10^6 psi and tensile was 70,000 psi. Cincinnati Testing Labs are reinforcing plastics with SiC.

Fibers	Matrices
W, B, steel, Mo, Be, BeO, SiC, WC, SiC, Al_2O_3	Al, plastic, Ni, mullite

21. Summary of the 11th Meeting of the Refractory Composites Working Group

Bartlett, E. S., Ogden, H. R.

DMIC Memo 212, Apr. 1, 1966

Battelle Memorial Inst.

Summary of programs in the area of refractory composites is given. AVCO is working on growing filaments, pretreating whiskers and filaments, consolidating specimens, and mechanically evaluating filaments and composites. They have made SiC fibers by vapor deposition on W wire in a B deposition apparatus; Ni precoating of Al_2O_3 was successful; B-Ni composites were made by electroforming over aligned B filaments held in a fixture; Ni-plating B filaments improved compatibility of B and Al in B-Al composites; and Mo with 30 vol % of Al_2O_3 had better oxidation resistance than plain Mo. Battelle Northwest has made carbon-Ni composites which did not react up to 1650°F by electrodepositing Ni on the C. Coating adherence was excellent. They are also studying interdiffusion in metal-matrix systems. Pneumatic extrusion of Ti, Mo, Al, Ni and W reinforced with B, W, Al_2O_3, and SiC was studied. Small additions of random, chopped fibers gave little or no strengthening; significant YS increase was noted with relatively small, oriented fiber additions; good mechanical and/or diffusion bonding was obtained; and fiber-matrix interactions can be reduced by this technique. General Electric Space Sciences Lab is working mainly with Al_2O_3-Ni. Precoating the whiskers with electrodeposited Ni gives best wetting; and additions of Ti and Zr weaken the whiskers, but Cr gives better wetting without weakening whiskers much. Ni reinforced with continuous Al_2O_3 was about 50% stronger at 1850°F than when reinforced with discontinuous whiskers. Al_2O_3-Al composites were also studied by G. E.; Ni-coated fibers were Al infiltrated without loss of fiber strength; and 19% whisker composites were made in this way. Harvey Aluminum has scaled-up steel-reinforced Al plates to 1 × 8 ft. Cold work followed by aging and more cold working increases UTS. Lap joint efficiencies of up to 72% were obtained by diffusion bonding. Adding overlapping reinforcing wires before diffusion bonding gave 92% efficiency. Vacuum infiltration of exposed wire ends with Zn alloy gave 54% joint efficiency. Melpar has chemically vapor deposited metals on Al_2O_3 whiskers in Ni, Cu, and Al matrices. Consolidation was by electrodeposition, coprecipitation, liquid phase sintering, and infiltration. Fabrication by swaging, rolling, or extrusion is being studied. Cold rolling Al-12Si with whisker reinforcement damages the whiskers, but at 700°F no whisker fracture occurs. United Aircraft has unidirectionally solidified Cb-Cb_2C and Ta-Ta_2C alloys, and has shown that zone leveling increases strength. Matrix additions may increase shear strengths, and improve high temperature properties. Wright Patterson AFB made oriented composites of B/Ti-6Al-4V by diffusion bonding, and got strengthening, although there were undesirable phases formed by interdiffusion. Investigations with other materials, temperatures, and pressures are underway. Douglas reinforced ZrO_2 with prestressed metal wires, and got about 4× improvement. IITRI reinforced Al_2O_3 with prestressed steel. Carborundum achieved good alignment of SiC whiskers in resin by extrusion with up to 12% whiskers present. BN fibers have been carded and felted; knitted and woven BN fabrics have been made also. General Electric made oriented Al_2O_3-resin composites by air elutriation and liquid gyratory techniques. They are also studying molding techniques, stress-concentration, moisture effects, and fracture mechanics. Brush Be is trying to make high-strength Be wires. Drawing in glass tubes was unsuccessful in general, but a BeO wash decreased reaction with the tubing. Strengths were about equal to cast Be. Vapor deposition on a cold substrate looks promising. General Electric is surveying the state of the art in technology of ceramic fibers, whiskers, tapes, and flakes. Growth parameters of B_4C vapor deposited whiskers were studied, and tensile strength was determined. Study and comparison of B deposited on W and SiO_2 substrates are given. Effects of chemical surface pretreatment are discussed. General Dynamics/Pomona has developed a high-temperature XRD camera to follow the structural changes of vapor deposited B on a Ta substrate. Changes in structure vs thickness were described. Marquardt has followed diffusion of C in Fe by autoradiography.

Fibers	Matrices
SiC, B, W, Al_2O_3 355 SS, graphite	Al, Ti, Ni, Cu, W, Mo, SS, epoxy, Ti-6Al-4V, ZrO_2

22. Fiber-Reinforced Metallic Composite Materials

Baskey, R. H.
IR-8-242(III), Aug. 1965
Clevite

Short wires (1/16 and 1/8 in.) of Mo had little reinforcing effect. The 1/4-in. wires improved tensile strengths, especially at elevated temperatures. Compacts were hot pressed from powder and hot rolled into sheets 50-mil thick. About 70% of the wires were aligned after rolling. The X-rays do not support this figure. Continuous wires were also rolled in Ti alloy. Rolling in the direction of reinforcement seemed to break up the wires into 1/8–1/16-in. fragments. Rolling across the reinforcement direction did not have this affect. Use of Mo mesh had little effect on UTS. At 800°F, there was little effect with wires less than 1/4 in. long; even these wires only gave about 10% strength increase.

Fibers	Matrices
Mo, W, KW, Mo	Ti-6Al-4V, Waspalloy

23. Fiber-Reinforced Metallic Composite Materials

Baskey, R. H.
IR-8-242(V), Mar. 1966 (AD-480186)
Clevite

Billets were formed by extrusion at 10:1, hot pressing, cold pressing, hydrostatic pressing, and rolling. The 14% W in Waspalloy caused shrinkage and rolling cracks, but the 9% one was okay. W in Hastelloy C did not cause cracks either as hot-pressed or as hot-pressed and rolled. Hot-pressing and rolling Mo in Ti-6Al-4V also was successful. TZM Mo in Ti alloy was twice as effective in strengthening at 1200°F than KW Mo. Continuous Mo wire was more effective than 3/8-in. wires. At 12% Mo, there was little difference in the strength of Ti alloy at high temperatures as a function of wire alignment. Extruded Waspalloy with up to 30% W 1/4-in. wires was not improved in UTS, but it was improved when hot-pressed and rolled. The composites did not seem to be notch sensitive. Failures appeared to occur along the interface. Stress-rupture life of Ti alloy with 16.5% Mo wires, hot-pressed and rolled, was better than Ti alloy with 30% Mo wires, extruded.

Fibers	Matrices
W, Mo	Ti-6Al-4V, Waspalloy, Hastelloy C

24. A Contribution to the Question of Compatibility Between Metals and Certain High Modulus Fibers

Bates, H. E., Wald, F., Weinstein, M.

(Paper E-4, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

AVCO Corp.

Both thermodynamic and kinetic compatibility between fibers and matrix must be considered. Phase diagrams give an insight into thermodynamic stability, but other factors are important also. In B systems it was noted that the presence of alloying elements may enhance harmful reactions, since B may react with one component more than another to change the local composition enough to greatly affect matrix behavior. CoSi and FeSi are suggested as diffusion barriers around B. SiC is unstable in molten Fe, Pt, Pd, Mn, Cu, and Au, and is unstable with V, Ti, Zr, Nb, Ta, and W. Kinetics are favorable in Ni and Ti up to 1000°C for SiC. In alloys of Ni-Cr and Fe-Ni-Cr-Si, SiC is unstable. BN is unstable at high temperatures with Fe, Ni, and W (but low temperature stability is not mentioned). At certain temperature ranges, BN may be stable with metals. Phase diagram studies indicate that B_4C and Si_3Ni_4 are unstable with all group IV, V, and VI metals. It is suggested that these fibers be precoated for high temperature use with metals. It is suggested that metal carbides, borides, and oxides be studied for high temperature use with metals.

Fibers	Matrices
B, SiC, Si_3N_4, B_4C, BN	Ag, Au, Cu, Ni, Ti, Ni-30Cu, Mn, Fe, Pd, Pt, V, Nb, Ta, W, Zr

25. Controlled Solidification of Metal Matrix Composites Utilizing the Monovarient Ternary Eutectic Reaction

Bates, H. E., Wald, F., Weinstein, M.

(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)

Tyco Labs

Paper discusses directionally solidified monovarient pseudo-eutectics in Ag-Au-Si and Ni-Cr-C-Si to form fibers in a eutectic-like matrix. States that it is possible to get eutectic structure along sections of ternary phase diagrams connecting two eutectics or a eutectic and peritectic; this allows formation of a range of compositions with eutectic structure and integral fiber reinforcement. It is not necessary for all three sides of the ternary diagram to include eutectics for successful application of directional fiber formation. They had to add 5% Si, which replaces Ni without changing the equilibrium conditions, to get directional growth. It is not known why this Si was needed. Cr_3C_2 fibers about 3–5 mm in diameter with $L/d = 100$ were grown. One problem was the tendency for fibers to grow at about 15° off axis, and then suddenly grow at 90° to the axis. This growth is thought to occur because composition is not balanced. They obtained little increase in UTS, but had some improvement in YS and about 50% higher modulus. Si may be segregating. Failure occurred at 90° to the whiskers. They did not examine phase equilibria in Si-modified ternary, since it would take too long and cost too much.

Fiber	Matrix
Cr_3C_2	Ni-Cr-Si

26. Metallic Fibers

Batha, H. D., Mark, S. D., Jr.

***Ind. Res.*, Vol. 9, pp. 88–94, Feb. 1967**

Carborundum Co.

General survey is given on various fabrication techniques for fiber and whisker preparation, including drawing, Taylor wires, vapor deposition, and pyrolysis of fibers. Composite fabrication and material requirements are discussed, and problem areas, such as whisker alignment, are mentioned. Potential applications are cited, such as electrical conductivity, strengthening, etc.

Fibers	Matrices
C, Al_2O_3, SiC, BN, B, SS	C, Ti, Ag, Ni, plastics, ceramics

27. Effect of Elevated-Temperature Exposure on the Microstructure and Tensile Strength of Al_3Ni-Reinforced Aluminum

Bayles, B. J., Ford, J. A., Salkind, M. J.

***Trans. AIME*, Vol. 239, pp. 844–849, June 1967**

United Aircraft

Heat treating eutectic composites of Al-Al_3Ni for up to 96 h at 610°C caused coarsening of the fibers, but caused no loss of room temperature strength, due to chemical compatibility and low interfacial energy between the phases. Preparation and testing are described.

Fiber	Matrix
Al_3Ni	Al

28. A Failure Law for Filamentary Composites

Becker, H., Gerard, G.

***AIAA J.*, Vol. 4, No. 12, pp. 2218–2219, Dec. 1966**

Allied Research Associates

Fracture mechanism in continuously reinforced composites is suggested. There may be an expotential relationship between crack density and applied stress. Data seem to fit theoretical plot when ratio of stress to ultimate stress = 0.6.

29. Mechanical Properties of Sintered Metal-Glass Composites

Belovian, A. F., et al.

***Poroshkovaya Met.*, No. 5, pp. 41–48, May 1966**

Composites containing 0.5–12% glass by volume were made by pressing and sintering in H_2 at 1000–1200°C. Bend, impact and tensile strengths, and shrinkage were studied. Glass additions did not improve the properties, which were dependent on the matrix strength.

Fiber	Matrix
Glass	Fe

30. Problems in the Design of Joints and Attachments

Berg, K. R.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

Whittaker Corp.

Composites are much harder to join by bolts, pins, or rivets than are isotropic materials, because of the fiber-matrix characteristics. There are many types of joints; analysis of the joint stresses is difficult due to redundancy in most fasteners; and attachments in composites must consider pin bearing, interlaminar shear, and bearing and transverse tension, particularly for high modulus fibers in polymer matrices. For pin bearing loading, failure is by shear tearing when laminates are oriented at a low angle. At 45°, lamination failure is by fiber tensile failure; for ±60°, failure is bearing at the hole; for 90°, orientation failure is by tear out at the hole. By varying orientation of the laminations, a tradeoff is obtained between bearing strength and composite properties. Cutting the filaments for pin or riveting requires stress redistribution around the hole and some fiber breakup, which can reduce fatigue strength. Joint design should consider the bearing load on the cut fiber ends. Adding metal doublers at the joint decreases the fatigue problems, but it is harder to make and more costly. Metal-matrix composites appear easier to join. Adhesive joint approaches may be macro, where the properties of the constitutants are not considered; or micro, where the fibers, matrix, etc., are considered. Lap shear joints have high shear stress at the ends. Use of plastic adhesives lowers the end stresses. Failures in multiple layer composites were found to occur at the outer ends of the laminate where it was joined to tabs. Use of laminated tabs gave good results; failures were in bearing at the pins. Joints may not be as serious a problem as was thought, but they certainly need more design study and analysis. One approach may be to knowingly accept some joint redundancy. Both riveting and adhesive bonding appear to be acceptable joint techniques.

31. Progress and Planning Report for MAMS Internal Research on Metal Matrix Composites

Blackburn, L. D., et al.

MAM-TM-66-3, Jan. 1966

AFML

Summary of inhouse Air Force studies of filaments and composites is given. They studied effects of surface flaws, fissures, and contaminants, as well as coatings and strength and moduli of whiskers and fibers. Solid and liquid state reactions between fibers and matrices were studied, and are discussed. Cu formed a liquid phase with B and dissolved it; Ni and Ti react with B at higher temperatures; and lower temperature fabrication gives poor composite properties. SiC is more compatible with Ti. TiN on B decreases fiber strength but improves compatibility. Cold pressing and sintering and hot extrusion were studied; both are promising. Mechanical behavior of composites also is discussed.

Fibers	Matrices
B, SiC, Al_2O_3	Ni, Al, Cu, Co, Inconel, Ti, Mg

32. Survey of Composites Research in Israel

Bodner, S. R.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

Technion

Survey of composites work in Israel is given. Composites, especially plastic composites, are very attractive, since Israel lacks resources of more common structural materials, such as wood and metals. A number of components have been made, including car bodies, boat hulls, pipes, and some structures. There is interest in sisal fibers in plastics because they are cheap and readily available. They are springy, need surface treatments to get good bonds, and have lower tensile strengths than glass. Adding cement powder helps increase composite strength. The fibers are cast in while the mold is vibrated to reduce voids. There is some analytical work being done on determining composite properties and the effects of voids.

33. Experimental Investigations on Dynamic Strength of Composites

Bodner, S. R., Lifshitz, J.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

Technion

Experimental techniques for determining dynamic properties of composites were reviewed, and some results were given. Dynamic modulus and damping are measured continuously during constant strain rate tests. Samples were 38% glass-epoxy cylinders. Torsional oscillation was used to determine natural frequencies of the samples. Damping was the same for filled and unfilled epoxy composites, with little frequency dependence. The modulus is increased by filling the matrix. Unbonding did not occur prior to overall failure. Void content seems analogous to the number of fiber breaks.

34. The Fabrication and Mechanical Properties of SiC Filament-Metal Matrix Composites

Bonnano, F. R., Withers, J. C.

(Paper D-4, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

AFML, General Technology Corp.

SiC is less reactive with metals than B, and SiC can have controlled modulus by adding B or Si to lower it, or adding C to raise it. Proper balance of fiber and matrix properties is important in obtaining best composite properties. Ni and Al composites were formed by wrapping B on mandrels and electrodeposition. The Al was then laminated in tape form and hot pressed. Void formation is a problem, but it can be reduced by using small diameter fibers, accurate spacing, controlled deposition rates, agitated solutions, or nonconductive fibers which build up layers of matrix deposit. For SiC-Ni, the critical volume fraction is 15–20%.

Fiber	Matrices
SiC	Ni, Al

35. Gas Pressure Bonding and Extrusion Techniques Applied to the Fabrication of Discontinuous W Wire-Ni Alloy Composites

Boone, D. H., Ryan, E. J.

(Presented at the 96th annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)

Pratt and Whitney

Paper discusses the processing used to obtain W wire composites reported earlier in the session, and previously abstracted (entry No. 81). Fabrication of the composites was a major problem. Several approaches were taken in an attempt to improve the standard powder techniques used. An automatic wire chopper was built to give 1/6- or 3/16-in. wires. Powder used was −325 mesh with 0.1–0.2% O_2, which gave some trouble. The wires were randomly mixed in Inconel powder, were vibrated, and were hot isostatically pressed at 1800°F for 4 h without reaction between wires and matrix. Compacts also were extruded at 1900–2000°F, 4:1 and 9:1, with good results. W content ranged from 7 to 27 vol%. Fiber alignment in extruded samples was good; spacing was fair to poor. Hot isostatic pressing was not as fast, well controlled, or cheap as hot pressing. The large plastic deformation in extrusions is not as desirable to designers.

Fiber	Matrix
W	Inconel

36. Fabrication Effects and Environmental Interaction of Filament-Wound Composites

Brelant, S., Petker, I.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

Aerospace Corp., Aerojet-General Corp.

The effect of processing and environment on filament-wound pressure vessels is unique for each application. If loadings, such as uniaxial tension and NOL shear rings are considered, this effect can be seen, since loads and stress patterns are different for each one. At stresses well below the ultimate, crazing occurs in both test bottles and NOL rings, but the loads are still carried satisfactorily. In rings, the resin shear strength is the limiting factor. Voids are very bad for composites loaded in interlaminar shear. Both temperature and relative humidity affect the glass-resin burst pressure when the part is first prestressed to 80% of UTS. If the part is not prestressed and no crazing of the resin occurs, immersion in salt water for long periods of time has little effect. Interlaminar shear is a function of the resin strength up to the point where voids in the resin act to weaken it. If no voids exist, there is good agreement with the experimental and theoretical shear strength. Interlaminar shear strength of laminates with cut fibers that had been exposed to water for long periods of time were not affected, due to the application of improved fiber finishes which protected the glass. The same type of test with 0.5–9.0% voids gave little change in the shear strength as a function of time immersed, but the strength at all times was lower as void content increased. Burst pressure of bottles is dependent only on the filament tensile strength. If the fabrication methods give composite inhomogeneity or defects, the composite will be weaker than it should be. There is no interaction between processing and exposure; each has an independent effect.

37. Structural C Yarn

Brown, H. J.

(Paper D-6, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

HITCO

Processes for making and handling C yarn for structural use are described. These are defined as containing 99.9% C minimum with crystallite that are well ordered and oriented. Experience winding glass is applicable to winding C yarn. Yarn travel on the rollers, round cross-section of the fiber, and poor quality of roving present fabrication problems. Wetting with resins does not seem to be a problem. Laminate properties and single fiber properties are given. UTS is 0.4×10^6 psi maximum and modulus is 3×10^6 to 75×10^6 psi for fiber 0.2–2 mil in diameter. Rapid improvements are being made in these C fibers formed by pyrolysis of organic fibers and their formation into composites.

Fiber
C

38. Fiber-Reinforcement: the Present State of Development

Buchi, G. J. P., Heap, H. R.

***English Elect. J.*, Vol. 21, No. 5, pp. 8–12, May-June 1966**

Nelson Research Labs

General summary is given on the dispersion strengthened and fiber reinforced composites, including a sampling of property data. Methods of dispersion strengthening such as internal oxidation, blending powders, oxidizing powders, and co-precipitation are covered. Fiber reinforcement, using plastic matrices and glass fibers, is well advanced, but limited by low fiber modulus and plastic temperature capability. Higher modulus glass fibers and resins stable up to 400°C have been developed in the U. S. recently, but they are not universally applicable to laminate production. Much development of high modulus fibers is going on in the U. S. and has been successful with plastic matrices; metal-matrix work both with fibers and whiskers is less advanced. In practice the full strength of metal-matrix composites probably will not be used; they will be most useful where conflicting properties are required. Some work is being done on reinforced ceramics, but applications are limited mainly to re-entry bodies or to others where thermal shock resistance is needed.

Fibers	Matrices
B, SiC, graphite, glass, steel	Al, Cu, plastic

39. Metal Matrix Composite Materials

Burte, H. M., Bonanno, F. R., Herzog, J. A.

***Orientation Effects in the Mechanical Behavior of Anisotropic Structural Materials*, ASTM STP-405, pp. 59–92, 1966**

AFML

Summary of advantages, progress, programs, and problems in metal matrix composites is given. Present work is on development and characterization of filaments, analysis of composite micromechanics, study of fiber-matrix interaction and interfaces, preparation and characterization of composites, comparison of theoretical and actual properties, and development of test methods. Theory is ahead of experiment. Report gives advantages of metal matrix, such as good shear strength, higher temperature potential, protection of fibers, etc. Rule of Mixtures is lower bound of composite properties, due to strain hardening and similar effects in matrix (Hashin says it is upper bound). Effects of fiber diameter, spacing, and overlap are noted. As L/d decreases, required matrix shear strength increases, if maximum fiber strength is to be developed; thus, shorter fibers may be used more effectively in metal than in plastic matrices. Fabrication techniques and fiber-matrix reactions are discussed, and problems are noted. Control of the interface is important. Fiber characterization and testing is described.

40. Determination of the Feasibility of Forming Refractory Fibers by a Continuous Process

Campbell, W. B.

AMRA-CR-63-08-8F, June 1966

Lexington Lab

Whiskers were grown by vapor phase reactions with the halides in a tube. High growth rates (up to 4 cm/s) were obtained. Important process variables include gas mixture and ratios, supersaturation, substrate area, gas velocity, temperature, and thermal gradients. Turbulent gas flow gave better gas mixing, and less of a stagnant boundary layer on the substrate. Both solid and powder substrates were used successfully. Size of whisker was related to size or grain size of the substrate. Tensile strengths were comparable to whiskers formed by other processes. Best quality whiskers were obtained at moderate vapor velocity, 1500–1600°C, and low pressure (5–10 torr). B, SiO_2, and Si_3N_4 whiskers also were grown.

Fibers
Al_2O_3, SiO_2, B, Si_3N_4

41. Metal Laminate Composite Materials for Compressor Blading

Carlson, R. G., Tomalin, D. S.

(Paper F-6, presented at 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

General Electric/Cincinnati

Both B fibers and matrix materials were tested chemically and mechanically before compositing. Test techniques for B fibers are described. Composite formation was by vacuum diffusion bonding; Ti hydride was used with the Ti sheets to promote bonding. Compatibility studies showed that B exposed to vacuum or Ti powder at 1500–1600°F loses strength. Coatings for the B were examined; C coatings were deposited in an electron microscope replication operation; Cu, Al, and Si coatings were vacuum metallized; and Cb was sputted on. None of these coatings improved B strength after exposure to Ti powder. Si coated samples were less reactive to Ti hydride. Actually, the Si seems to promote formation of a glassy bond between Ti and B without lowering the fiber strength. Si coating is recommended for promoting bonding to Ti and for service at temperatures below 1500°F. Fabrication of composites is discussed; the matrix sheets were grooved by a photoresist etching method. Best results were obtained for Ti when Ti hydride power was placed between sheets. Tests run included tensile, ballistic impact, thermal cycling and exposure, and density. The Al composites were formed at 1000°F, and showed Rule of Mixtures strengthening; up to 20% B in Ti gave little strengthening, but did increase modulus. Ballistic impact of Al-B composites was poor, but for Ti-B, it was good up to 200 ft/s. Thermal cycling and exposure had little effect on properties.

Fiber	Matrices
B	Al, Ti-6-4

42. Strengthening Mechanisms in Wire Products

Chandhok, V. K., Kasak, A., Wachtmeister, G. C.

AFML-TR-66-63, May 1966

Crucible Steel

A variety of alloys were strengthened in wire form. Best results were obtained with AFC-77, a precipitation hardenable SS; UTS of 0.55×10^6 psi was obtained, and at 1100°F, UTS was 0.307×10^6 psi; 10-h stress rupture was 0.17×10^6 psi. This strength was obtained by successive drawings and annealings at 800–900°F. Drawing was also done at cryogenic temperatures, and showed some promise.

Fibers
AFC-77, WF-11 steel, 302 SS

43. Metallic Filaments and Their Composites

Chase, V. A., Patton, J. B.

(Paper B-4, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Brunswick

Fine wires (12 μm and less) have been formed from many metals, mainly for use in fabrics. Continuous filaments have been woven into fabrics by many techniques, i.e., twills, weaves, satins, braided, and knotted. As little as 1% SS in fabrics eliminates static electricity, and as little as 10% allows the fabric to be self-heated by resistance. Applications include heated clothing, rugs and drapes, wire strengthened metal and glass matrices, static free textiles, filters, fillers for teflon to increase thermal conductivity and strength, high-strength high-temperature cordage and flexible belts, mechanical rubber goods, and flame barriers. Unlike most small diameter fibers, strength drops as diameter drops due to inclusions in the wire.

Fibers	Matrices
Ta, 304SS, N6, Al, Pb, Ti, Zr, Mo, W, Be, 80Ni-20Cr	Resin, glass

44. Stress Fields Around Parallel Edge Cracks

Chen, P. E.

***J. Compos. Mater.*, Vol. 1, No. 1, pp. 82–91, Jan. 1967**

Monsanto

Stress fields around edge cracks are analyzed theoretically, and are compared with photoelastic results, using a computer program. A number of simplifying assumptions are made, including plane stress or strain, infinitesimal strain, negligible body forces, cracks of equal length through the specimen thickness, and stress-free crack surfaces in an isotropic, homogeneous material which obeys Hooke's law. Both collinear and parallel edge cracks are treated, with good agreement between theory and experiment. The mathematical model and computer program seem valid, given the stated boundary conditions. Effect of crack spacing can be determined, as can the rate of strain energy release or crack extension.

45. Effect of Fiber Orientation on the Tensile Strength of Hot-Pressed Aluminum-Inconel and Aluminum-Tungsten Composites

Childs, B. G.

AECL-2605, Sept. 1966

Atomic Energy of Canada, Ltd., Chalk River, Ont.

Composites were made by winding the reinforcing wire and Al wire on a drum, unwrapping, and hot pressing; they could not be rolled successfully, and fiber orientation varied by up to 20° from nominal. Strength was very dependent on fiber direction, dropping rapidly for more than 5–10° misorientation from test direction. Unreinforced Al showed the same type of behavior, since it also was made from wires. Strength drop was greater than that due to matrix flow stress only; difference is attributed to weakness of the interface. Failure mechanisms and the effect of temperature on allowable misalignment are discussed. At elevated temperatures much less misalignment is tolerable.

Fibers	Matrix
Inconel, W	Al

46. Investigation of Whisker-Reinforced Metallic Composites

Chorne, J.

In *Summary of the 8th Meeting of the Refractory Composites Working Group*, ML-TDR-64-233, Vol. III, Jan. 1964 (AD-470695)

General Electric Space Sciences Lab

Pt-coated Al_2O_3 whiskers were incorporated into an Ag matrix by vacuum infiltration. Composite strength was less than anticipated because of the presence of weak whiskers. The size and strength distribution had considerable effect on composite strength. Ag with 37% Al_2O_3 has UTS of 25,000 psi at 1720°F (98% of MP). Also they are using Ni matrix.

Fiber	Matrices
Al_2O_3	Ag, Ni

47. Development of Composite Structural Materials for High Temperature Applications

Chorne, J., et al.

AD-466957, Feb. 1965

General Electric Space Sciences Lab

Appreciable increase in strength was obtained when fibers were coated with Ni by electroless plating or sputtering, and formed into composites by electroforming or vacuum hot pressing. Cold pressing and sintering gave less strengthening. Liquid Ni infiltration destroyed coatings; Pt, fernico, Ta, and W all were affected. Thick coatings of W were not dissolved, but maximum fiber volume is reduced when coatings are thick.

Fiber	Matrix
Al_2O_3	Ni

48. Research on Fiber-Reinforced Composites

Chorne, J., et al.

AD-615662, May 1965

General Electric Space Sciences Lab

Brief survey of GE programs in the field is given; most programs are previously abstracted. Sapphire-reinforced Ni presented problems of fiber pull-out. Additives such as Cr, Ti, and Zr enhanced wetting and bonding, while Cu and In had less effect. Ti and Zr reacted with the Al_2O_3 more, and tended to weaken it. Tensile properties of Al_2O_3 whiskers were determined as a function of growth direction, diameter, length, surface defects, handling, surface damage, surface coatings, and composite fabrication. Growth parameters and physical properties of B_4C were studied. Formed fibers by vapor deposition on W 1-mil wire. Technology of ceramic and graphite fibers was surveyed; report contains abstracts of 200 patents and 550 items.

Fibers	Matrices
Al_2O_3, B_4C, Ceramics	Ag, Ni

49. Research on Fiber-Reinforced Composites

Chorne, J., et al.

GE Reprint No. 332, May 1965

General Electric Space Sciences Lab

Summaries of work reported at 10th Refractory Composite Working Group on six programs which General Electric is working on currently. Reported in detail in other abstracts, but a brief summary is as follows. High temperature composite materials are being developed from Ni, Ni-Pd, and Ni-Pd-W reinforced with Al_2O_3. Major problems are in fabrication of samples with high concentrations of whiskers, well bonded to the matrix. Infiltration techniques seem unreliable for higher temperature matrix metals. Use of alloying additions in Ni to promote wetting to Al_2O_3 indicates that Cr, Ti, and Zr segregate at the interface and form bonds. Only Cr gave improved shear strengths. Al_2O_3 wool in an Al matrix is being studied as a function of fabrication, including handling and surface damage effects, composite properties as effected by fiber diameter, alignment, and growth direction. Preliminary data show wide scatter as a function of fiber cross section. The B_4C whiskers were grown most successfully by condensation of the vapor evaporated from a source. Chemical vapor deposition was less effective, and has been dropped. Vanadium catalyst increased deposition rates, and promises to give high productivity per run. Micro-composites containing 5–10% whiskers in epoxy or Al have been made and tested. A survey of technology in the field of fibers and filaments useful above 1200°F was made, both by contacting other workers in the field and literature searches. Major conclusions were that a lot of work is being done, test methods are not standardized and often not reported, and increased emphasis is being given to development of composites and whiskers. Some of the well-known fabrication problems are discussed. Continuous B_4C fibers are being prepared by chemical vapor deposition from a halide. Condensation from a source was unsuccessful. A 1-mil W wire was used as a substrate; Mo did not work.

Fibers	Matrices
Al_2O_3, B_4C	Ni, Al

50. Development of Composite Structural Materials for High Temperature Applications

Chorne, J., Bruch, C. A., Sutton, W. H.

JPL 152148-1, July 1966

General Electric Space Sciences Lab

Report describes growth conditions and typical growth defects in Al_2O_3 whiskers. Maximum yield was obtained at 1325°C, but quality and uniformity were unaffected from 1310 to 1400°C. Facilities have been increased, and General Electric now has a capacity for 1 lb of whiskers per year. It is impossible to eliminate debris and whisker defects, but separation by air elutriation helps to remove debris and weak whiskers, and gives some whisker alignment. Composites were prepared by electroforming and pressing. Pre-coating whiskers with electroless Ni may have weakened the composite by S embrittlement; appreciable S is present in the plating solution, and was found in the Ni plate. Pretreating the Ni at 1250°C for 24 h removed the embrittlement, and permitted sintering of the compacts at 800–1000°C without reducing strength levels. Pressures of more than 1/4 tons/in.^{2} are required for full densification. W was applied by sputtering and then electroformed with Ni both with and without pressing. Composite strengths are less than expected.

Fiber	Matrix
Al_2O_3	Ni

51. Development of Composite Structural Materials for High Temperature Applications

Chorne, J., et al.

Third Quarterly Report on Contract NOw-66-0443-d, Aug. 1966

General Electric Space Sciences Lab

Wetting studies were made on Al_2O_3 plaques and whiskers coated with W, Ti, and W over Ti in contact with Ni and nichrome. W coatings gave good initial wetting with Ni, but after all the W was dissolved, the Ni dewetted. Nichrome formed a strong bond with the plaques about 1/2 the time, particularly in the presence of Ti. Often the Al_2O_3 shattered during cooling. Additional work with whiskers coated with W and W over Ti showed some degradation of the whiskers in molten nichrome. Electroplating coated whisker bundles gave low density due to the inability to plate into voids. Pressure bonding was used to consolidate the composites, but was found to have deleterious effects on the properties, mainly due to fracture of the whiskers. Exposing the bundles to the pressure bonding environment without the pressure did not give loss of strength. A grain boundary phase is present in the Ni which causes embrittlement of the composite. It can be removed by heating to 1250°C or above, but is present up to 900°C. Analysis showed it was not S or P, but has an atomic number of 11 or less; H is suspected. Once removed by heat treatment, the phase does not reappear; the minimum time at temperature is at least 1 h, and may be dependent on sample size and geometry, since the mechanism of precipitate removal is unknown. Composite processing methods and test results are given.

Fiber	Matrices
Al_2O_3	Ni, nichrome

52. Development of Composite Structural Materials for High Temperature Applications

Chorne, J., et al.

First Quarterly Report on Contract NO0019-67-C-0243, Apr. 1967

General Electric Space Sciences Lab

Growth of Al_2O_3 whiskers is well developed and needs little supervision. The whiskers are coated with Ti on both sides simultaneously by cathodic sputtering prior to consolidation by pressure bonding. Sputtering conditions are described. Pressure, voltage, and time are the important variables. Air elutriation is used to classify, separate, beneficate, and orient the fibers. The process and equipment are described. The Ni matrix was embrittled during electroplating; this was eliminated by a prolonged vacuum anneal. Composites were formed by manually aligning felts made from whiskers sputtered with either W over Ti, or two coats of W, followed by Ni electrodeposition, heat treatment, and pressure bonding; another method was to use the felts as aligned by air elutriation. Test results were lower than expected. Reasons may have been fiber breakage, interface bond strength was low, or sample misalignment during test. Post-test examination of extracted whiskers indicated that low strength of the composites was caused by fiber breakage during processing, poor bonding between fiber and matrix, and that whisker degradation did not occur; semi-automatic whisker alignment gave lower strength composites than manual alignment. Modulus was determined dynamically, and was 80–90% of Rule of Mixtures values. Coatings of Ti/W gave more damping than coatings of W, when equal amounts of whiskers were tested in the composite.

Fiber	Matrix
Al_2O_3	Ni

53. Metal-Matrix Composite Materials for Aircraft Compressor Blades

Compton, W. A., Akins, R. J., Mnew, H.

(Paper C-6, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Solar

Requirements of compressor materials are reviewed from the standpoint of temperatures, altitudes, and mechanical properties. Desired properties include resistance to impact, erosion, corrosion and fatigue, low density, good notch sensitivity, and high modulus. Available materials for evaluation include: Al reinforced with B, stainless and Be, Ti reinforced with B, Be and TZM fibers and SiC whiskers, 304 stainless and Udimet 700 reinforced with SiC whiskers, and Al reinforced with SiC and Al_2O_3 whiskers. Properties of various materials are given, along with theoretical composite property relationships. Actual properties of various composites are given, and relatively good and bad materials identified for various locations in the compressor. Both Al-stainless and Al-B look very promising; Al-SiC and Ti-SiC were poor, and need more development. Ti-TZM is not attractive for early stages, but has promise for later, higher temperature stages. Al-Ti-Be had a good UTS/density ratio, and is of interest for compressors. Additional work is needed to establish UTS, YS, impact strength, and modulus as a function of temperature, thermal stability, erosion resistance, and flaw detection techniques. A number of graphs are given to show the influence of various design limits on desired properties.

Fibers	Matrices
B, SiC, TZM, Be, SS	Al alloy, Ti alloy, Ti + Al

54. Stress-Rupture and Tensile Test Techniques for Single Boron Filaments at Room and Elevated Temperatures

Cook, J. L., Sakurai, T. T.

(Paper H-1, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Douglas MSSD

Equipment, techniques, test procedure and results of tensile and stress rupture tests on B from room temperature to 1200°F are described. Tests were run in air and argon. Problem areas include gripping, which was solved by using pneumatic grips with Al foil inserts, and strain measurement, which was done optically with a 4-in. gauge length. Both sample diameter and test temperature may have been slightly in error, and added to data scatter. Stress rupture in air gave lower times to stress dropoff than in argon. Strength of B in air dropped rapidly above 900°F, and less severely in argon above 900°F.

Fiber
B

55. Tensile Properties of Fiber-Reinforced Metals: Fracture Mechanics

Cooper, G. A., Kelly, A.

(Presented at International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967 and also to be published in *J. Mech. Phys. Solids*)

Cambridge Univ.

Composites were electroformed by laying down thin deposits of Cu on alternate layers of fibers. No secondary fabrication was used to densify the composites. The effects of fiber diameter and composite thickness were studied from the standpoint of fracture mechanics, and resistance of the composites to crack propagation from the root of notches made by EDM. Strong and weak bonding between fiber and Cu matrix was induced, and it was found to effect failure mode. Composites may be designed to be either notch sensitive or insensitive (failure by splitting or delamination) by changing sample thickness. Thinner samples will fail parallel to the fibers, while thick ones will be governed by the matrix and be notch sensitive. Quantitative values for the glass-Cu composites were hard to obtain; there was appreciable Cu porosity which was not removed by annealing after forming. Vacuum casting Cu around W formed a composite with brittle wires well bonded to the matrix. Failure was noted in the fibers ahead of the notch, not in the interfaces, although the ductile Cu was not fractured. The stress field and propagation of cracks at the notch are a function of the composite rather than either phase. In composites with well-bonded brittle fibers, it was possible to grow the cracks at a controlled rate up to the point at which a critical number of fibers broke and the remaining section was overstressed. Fabrication techniques, fracture mechanics, and micromechanics are discussed. Work of fracture in the composites was measured as 10^8 erg/cm^2. As fiber diameter decreased from 1 mm to 40 μm, the fracture energy decreased, confirming theoretical calculations that coarser fibers should be more resistant to crack propagation. Theoretical calculations of the required volume fraction of fibers to get splitting rather than notch sensitivity varied from 1% for no plastic flow to 73% for matrix plastic flow; actual values fall between.

Fibers	Matrix
W, glass	Cu

56. Matrix-Limited Fatigue Properties in Fiber Composite Materials

Courtney, T. H., Wulff, J.

***J. Mater. Sci.*, Vol. 1, No. 4, pp. 383–388, Apr. 1966**

M. I. T.

Fatigue properties of composites loaded in the direction of the fibers is affected by the work hardening of the matrix. Composite deformation is divided into four stages: elastic deformation, plastic matrix deformation, plastic deformation of both, and failure. The plastic matrix deformation region is of most practical interest. Equations are derived that take into account the work hardening of the matrix, both for reversed loading and all positive loading. If loadings above the endurance limit are used, the predicted lives are short (10^4 cycles or less). As volume fraction of fiber increases, fatigue life drops. This assumes that the fiber and matrix strain equally. The approach neglects micromechanics and differences in Poisson ratios. (Actual data by others does not support this pessimistic attitude; although W-Cu in reverse tensile loading does seem to be adversely affected by Cu strain hardening.) It is a theoretical study with no supporting test data.

Fibers	Matrices
W, steel	Ag, Cu

57. Exploratory Investigation of Glass-Metal Composite Fibers

Cox, J. E., Veltri, R. D., Shulze, C. E.

D910242-6, May 7, 1965

United Aircraft

They attempted to pull Be wires by a modified Taylor wire method. Hand drawing gave fibers up to 15 in. long, but reaction between glass and Be is so rapid that drawing time must be very rapid. Attempts to reduce reactions by use of a protective atmosphere were unsuccessful. BeO coatings on the ID of the glass gave poor results — the glass could not be drawn. BeO and ZrO coatings on Be wire were unsuccessful, but alumina coatings, while uneven, allowed wire drawing. Thin metallic coatings on both the Be and the glass were unsuccessful. Pressure ejection of molten Be at 2000°C did not give fibers, but results were encouraging. A later report, AD-633527, gives the results for 1965–66 on the same program. There still was no success using the Taylor wire process, because of fast reactions between Be and glass, regardless of the protection system used. Pressure ejection of Be from a BeO nozzle into a hollow glass tube seems to be the most promising technique.

Fiber
Be

58. Ceramic Fiber Reinforced Metal Composites Provide High Strength With Light Weight

Cratchley, D., Baker, A. A., Jackson, P. W.

***Mater. Des. Eng.*, Vol. 64, No. 7, pp. 83–85, Dec. 1966**

Rolls Royce

A model system of 2-mil silica in Al was prepared by precoating the silica with Al and hot pressing, winding on a mandrel and hot pressing, or winding fibers with a resin and then burning off the resin. Plastic deformation of the matrix occurs during loading of the composite. Strength was increased over SAP alloy and regular aluminum, both at room temperature and up to 750°F. It also has better damping capacity than cast Fe. Fatigue properties seem dependent on plastic strain in the matrix. In a limited range of plastic strain, fatigue strength is much better than Al; but at high strains, properties approach those of the matrix. More fibers or fibers with higher modulus will increase fatigue strength. Notch toughness also is greater than unreinforced Al. Joining still presents problems.

Fiber	Matrix
Silica	Al

59. Silica Fiber Reinforced Al

Cratchley, D., Baker, A. A.

***J. Am. Ceram. Soc. Bull.*, Vol. 46, No. 2, pp. 191–195, Feb. 1967**

Rolls Royce

Composites of Al-coated silica were formed by hot pressing. The system is used to develop basic information on the behavior of composites at temperatures which are high in relation to the melting point of the matrix. The low modulus of silica is not considered detrimental as long as the matrix deforms plastically. Coating conditions had to be carefully controlled to prevent excessive reaction between Al and SiO_2. Pressing temperature and pressure must be balanced between good bonding and excessive fiber damage. Pressing was done at 450°C with 12,000 psi for 1 h. Strength was determined from room temperature to 500°C. Soaking 500 h at up to 400°C had no effect on room temperature strength; between 400 and 500°C, strength dropped as a function of temperature, this was due to chemical reaction between fiber and matrix. Soaking for 3 min at 565 and 640°C caused rapid degradation and reaction. Reversed bending fatigue tests showed that fiber ends can be sources of cracks, while the fibers act to stop crack growth in the matrix. Notched tensile samples failed by crack propagation at the fiber-matrix interface. Impact strength decreased as pressing pressure increased.

Fiber	Matrix
Silica	Al

60. Missile and Aircraft Systems Constraints and Operational Requirements

Craven, J. P.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

Special Project Office, U. S. Department of the Navy

Requirements for materials in Naval vessels and missiles are discussed. Deep ocean technology requires high strength-to-weight ratios; resistance to biological fouling, to corrosion, and to implosion; and qualities of buoyancy, optically transparency, etc. Submersibles, such as the Aluminaut, have increased depth capability since they have no welds. Syntactic foams are good fillers for honeycomb and are very buoyant. They can be used with fiberglass skins. Segmented fiberglass spheres look very promising. Preloading of fiberglass composites at depths improves the fracture resistance; another technique is to coat solid glass with a more ductile material such as rubber. G.E. is building a sphere of pyroceram with Ti matrix and end fittings which looks somewhat like a stained glass window; it looks very good for survival at depths, particularly when it also has a fiberglass overwrap which is plasma sprayed with Ti. Missile and aircraft requirements emphasize high strength-to-weight and modulus-to-weight ratios, resistance to radiation and nuclear effects, heat resistance, ability to be pressurized internally, etc. Rocket cases are a good example of this.

61. Advancements in Monofilament Structural Composite Technology

Crichlow, W. J., Sorenson, V. S.

***J. Aircraft*, Vol. 3, No. 5, pp. 431–435, Sept.–Oct. 1966**

Lockheed/Burbank

UTS:density and modulus:density ratios were compared for various materials. Use of 3–5 mil glass fibers in high viscosity epoxy as a laminate for a test wing box section is described. Various laminate layup configurations were used, and were tested in fatigue and under simulated service conditions. Notched fatigue tests also were run, and were compared with other materials. Box beam samples failed at less than design stress at about 2/3 of design allowable; failure was by interlaminar shear in an area that analysis indicated was not the most critical. Crack propagation in notched tensile samples was slow. Practical processing and fabrication techniques were demonstrated, but more development must be done to produce a consistent, man-rated product.

Fiber	Matrix
S-glass	Epoxy

62. Ni-Base Composites—Super Superalloys

Davies, C. F., Baskey, R.

(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)

Clevite

The compatibility of W and various superalloys was studied first. W-Hastalloy X was best, although the Udimet alloys looked good too. A 10-mil W wire was used. Bars about 8 × 3 × 1 in. were made by powder metallurgy. They can make 2 to 3 bars per day, using hand layup. With 37-vol% W, at 2200°F, UTS was about 55,000 psi vs about 8000 psi for unreinforced Hastalloy X. The 30-vol% TZM in Hastalloy X had a much higher UTS/density ratio. Above about 1450°F, the 37% W composite was much better than the base matrix. The 33% W had a 100-h stress rupture strength of about 31,000 psi at 1800°F vs about 2000 psi for unreinforced Hastalloy X. The 30% W composite had a fatigue strength of about 50,000 psi at 10^7 cycles and 1800°F; the fatigue strength exceeded 30,000 psi at 4×10^6 cycles. At 4400 psi and 1950°F, the 30% W had a creep of 3% after 60 h, at which time the test was terminated. Initial creep was about 2% at 3 h. The 25% W in Udimet 713C had UTS of 150,000 psi at 1000°F, and 100,000 psi at 1600°F. After 3000 h at 1950°F, the sample had 95% of a Hastalloy X sample run in Ar for the same time. The W wires oxidized on the surface, but still retained their strength. Above 39–40% W, strengthening decreases due to wire interference, overlap, and fabrication difficulties.

Fibers	Matrices
W, TZM	Hastalloy X, Udimet 700, Udimet 713C

63. Measuring the Elastic Moduli of Inorganic Filaments

Davies, L. G., Watson, R. C.

***Mater. Res. Stand.*, Vol. 7, No. 5, pp. 207–210, May 1967**

General Technology Corp.

Method for measuring the elastic moduli of fibers is described, as is the test equipment. A vibrating-reed technique was used for Youngs modulus; resonant frequency is determined; and using sample density, radius and length, the modulus is determined. Shear modulus was determined using a torsional pendulum and sample length, radius, resonant frequency, and moment of inertia; since radius is taken to the fourth power, small errors in measurement have large effects on the values. The method is simpler and more rapid, can be used at a range of temperatures for one sample, and can use small samples. Modulus values tended to be higher than those from flexure or tensile tests.

Fibers
B, W

64. Metal-Metal Composites—A New Structural Material

Davis, L. W., Morgan, W. R.

(Presented at the AIAA/ASME 7th Structure and Materials Conference, Cocoa Beach, Fla., Apr. 18–20, 1966)

Harvey Aluminum, Marshall Space Flight Center

This paper describes program to fabricate steel wire-reinforced Al sheets 8 × 1 ft. Co-extrusion was initially tried, but tool pressures were too high, and extrusion ratios were too great. Diffusion bonding was successful without use of diffusion aids. Best results were obtained when bonding was done at 900°F and 14,000 psi, followed by hot rolling to flatten, solution heat treating and aging, cold rolling 1 or 2%, aging, and cold rolling again. If an intermetallic bond between the Al and steel is formed, strength of the composite is reduced appreciably. Major object was a material with good cryogenic properties, so 2024 was chosen. It also is heat treatable, and available. Al sheet was prepared for bonding by wire brushing just before placing the wires and bonding. Machining by EDM was satisfactory, but sawing and milling was unsuccessful; abrasive cutting under coolant gave good results. Be and B reinforced Al plates were prepared and tested, with results near those predicted by the Rule of Mixtures. Only spot welding seems to give good fusion joints. Overlapping joints also have been tested; a 1-in. overlap with plates designed for joining gave 92% joint efficiency. Diffusion bonding offset strips of composite gave only 57% efficiency. Liquid infiltration and explosive compaction also are being investigated.

Fibers	Matrices
Steel, NS355, Be, B	Al-2024, 1100, 7178, 2219, 3003, 5052, 5456, 7075

65. New Metal-Metal Composite Materials

Davis, L. W., Morgan, W. R.

***J. Spacecraft Rockets*, Vol. 4, No. 3, pp. 386–391, Mar. 1967**

Harvey Aluminum, Marshall Space Flight Center

This is basically the same paper as that given at the AIAA/ASME Structures and Materials Conference, Cocoa Beach, Apr. 1966, and previously abstracted (entry No. 64). Main difference is a brief discussion of the use of extrusion to form the composites. Ratios as high as 625:1 were tried, later reduced to 156:1, without success because of high tool pressures. Subsequent development of hydrostatic extrusion may make it possible to co-extrude the composite.

Fibers	Matrices
Steel, B, Be	Al alloys 1100, 5456, 5052, 7075, 2024, 2219, 3033, 7178

66. Stainless Wire + Al Matrix = Strong, Light Composite Plate

Davis, L. W.

***Metal Progr.*, Vol. 91, pp. 105–114, Apr. 1967**

Harvey Aluminum

Composites were formed by diffusion bonding prewrapped sheets of NS-355 rocket wire and Al. Use of bonding aids such as Li, In, Sn, Cu, Zn, and Ag were only partly successful. Wire brushing gave a better bonding surface than Ag or zincate coatings. Too long at temperature gave brittle intermetallics and reduced wire diameter, lowering composite strength. It is necessary to press at under 950°F to prevent loss of wire properties. Variations in cooling rate after bonding and working and aging treatment affect properties. With 7178 Al, the best schedule was bonding, hot roll flat, solution heat treat, 2% cold roll, age, and 2% cold roll. Sawing or milling was no good, but samples could be cut by EDM. Small amounts of Al reinforced with B, Be, and SiC have been made. They prefer to work with large diameter wires, although smaller wires are stronger.

Fibers	Matrices
B, SS, Be, SiC	Al, Al alloys

67. Properties of Composite Materials Reinforced by Whiskers

de l'Estoile, H.

NASA TT F-9372, May 1965

Doc-Air-Espace

Brief review of composite properties is given. Ultimate strengths at various stresses and strains were calculated for 50-vol% fibers. Whiskers must be oriented for maximum effect. Adding 12-vol% Al to light alloy increases strength 10×.

Fibers	Matrices
C, Fe, Ag, Cu, Zn, Cd, Al_2O_3, SiO_2, Al	Ti-Mo, Steel, Co alloys

68. Be Fine Wire

Denny, J. P., Stitzlein, C. F.

(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)

Berylco, Astrometals

Paper describes Be wire drawing technique, as discussed in AFML-TR-66-312. Wire can be drawn at up to 300 ft/min, about 12½% per pass, and with 10 dies in line. A 2-mil wire can be made on a production basis. For success, the stock must have low inclusion size and frequency. Various starting materials, including low-oxide hot-pressed block, ingot, and Pechiney SR were tested. The cast ingot looked best; it had 0.49% BeO and 0.071% Fe. Selected hot-pressed block was not as good; it had 1.50% BeO. The starting material was extruded at 1950°F. The cast material recrystallized during extrusion. It was then sheathed in Ni and drawn at 750–800°F without intermediate annealing. There was considerable grain refinement. Average properties of 10 lb of 5-mil wire were 0.151×10^6 psi UTS and 3.7% elongation. If fabrication of composites from the wire requires too much heat, the wire will recrystallize and the properties will drop; thus, some of the advantages may be lost. The Ni cladding can be etched rapidly and continuously. A total of 100,000 ft of 2-mil wire has been made so far.

Wire is now available at 16¢/ft. A cost of 1¢/ft for 5-mil wire is projected, with great improvement in properties as experience is obtained. The industry total of Be wire is less than 30 lb. Also 5-mil Ti wire has been made. The dies used for drawing are WC; a DAG type lubricant is used on the Ni The Be wire surface is rough, but it can be bent cold to 4T.

Fiber
Be

69. Off-Axis Strength and Testing of Filamentary Materials for Aircraft Applications

Dickerson, E. O., DiMartino, B.

(Paper H-3, presented at the 10th SAMPE Symposium, Advanced Fibrous Composites, San Diego, Calif., Nov. 9–11, 1966)

North American Aviation

Composite properties can be predicted from component properties, geometry of each layer, relative ply orientation, and shape of the actual part and its edge configuration. Critical design areas and restraints are noted, and a detailed structural discussion is given. Complicating factors include nonlinear material response, crossover effects, interaction between plys, uneven fiber spacing, and indeterminate edge conditions. Structural aspects such as unidirectional and cross ply layup are considered. Stress variation under uniform load is noted. Joints in organic matrices are best made with adhesives. Adhesive shear stress must be kept low, as by tapering the plates. Test methods are important in determining stress allowables. Brief discussions are included on tensile, compressive, shear, and cross-ply properties. Composite laminates should not be considered a material, but a design, where configuration and edge restraints are as important as material properties. Polar plots of axial and shear properties of boron-epoxy are given.

Fiber	Matrix
B	Epoxy

70. Structural Uses of Composites

Dietz, A. G. H.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

M. I. T.

Problems involved in use of composites in engineering structures were discussed. Earthbound structures are characterized by long lifetimes and cost limitations. Advantages of composites are formability, number and variety of shapes possible, strength, weight, and ability to transmit light; disadvantages include cost, size limits, low modulus, must be prefabricated, joining problems, and unknown durability. A number of examples of structures were shown: radomes up to 150-ft diameter; U. S. pavilions at the Brussels Fair and an exhibit in Moscow; columns, roofs, walls (such as part of the vertical assembly building for Apollo); the Monsanto house at Disneyland; etc. On the macro scale, the composite is considered to be homogeneous. It is assumed that elastic properties are linear and that strain is equal in all directions and in all parts of the composite. Stress is then plotted as a function of angle. The analytical results are only approximate for real structures.

71. Evaluation of the Potential of Advanced Composite Materials for Aircraft Structures

Dow, N. F., Rosen, B. W., Kingsbury, H. B.

AFML-TR-66-144, May 1966

General Electric Space Sciences Lab

Analytical comparisons are made for the efficiency of metals such as Ti, Al, and Be vs composites such as boron-epoxy, boron-Mg, and glass-epoxy. Boundary conditions and assumptions are given. In the case of some of the composites, properties data are assumed rather than experimental. For stiffened-panel metal wing box beams, Be is the lightest construction material, both in strength-limited and stiffness-limited applications. Glass-epoxy systems are inefficient both in strength and stiffness. Boron-epoxy and boron-Mg systems with isotropic fiber orientation are essentially equal to Be. Be-epoxy compares favorably with 7075-T6 Al, and seems preferable to Be sheet. For anisotropic composites, ±45° skin, 0° stringer boron-epoxy is the most efficient. Efficiencies are extrapolated to low and high temperatures (using assumed data) and a boron-Ni composite was most efficient; it weighed 15% as much as Ti at 1000h and 1500°F. Shear webs also were studied. All the composites were more efficient than the metals, with little difference between composites. Flat plates were studied both as plates and as sandwich face plates. In plate form the Be is most attractive; in sandwich form Ti, Be, and maraging steel all are attractive. In the case of composites, elastic efficiency is greatest for ±45° laminates, while sandwich structures in inelastic stress are most efficient in uniaxial form. B-epoxy and B-Mg are better than Al, but Be is better than both of them. Multiweb beam structures were analyzed, with similar results. At low stresses Be is best, but at high stresses B-Mg was best. Glass-epoxy was worse than Al. Fuselage loads are low enough so that boron composite sandwich or waffle would be most efficient. The composites are all better than metals in shear loading. Attainment of the potential advantages of B-metal matrix composites for high temperature applications depends on development of fabrication techniques and protection of B from attack at high temperatures. B-epoxy systems for room temperature applications requires proof of structural response to insure reliability. Advanced composites are not of major interest for use in shear webs. Glass-epoxy is about as good. B composites are very attractive for use in flat plates, particularly if experimental data bear out theoretical data.

Fibers	Matrices
B, glass, Be	Epoxy, Mg, Ni

72. Studies of Mechanics of Filamentary Composites

Dow, N. F., Rosen, B. W., Hashin, Z.

NASA CR-492, June 1966

General Electric Space Sciences Lab

An analytical study is given on the efficiencies of different fiber and reinforcement systems for monocoque and stiffened launch vehicles. Composites with uniaxial properties formed by lamination of tapes gave better efficiencies than metal shells; elastic compression governs design except for most efficient stiffening concepts. Low concentrations of high modulus isotropic filaments have better buckling resistance than metals. Hollow glass fibers are inefficient; hollow fibers only look useful where density is important, as in minimum gauge monocoque shells. Shell buckling efficiency is a weak function of filament modulus, a stringent function of filament density, and a much weaker function of binder density/modulus ratio. The matrix should have good fracture toughness to inhibit propagation of fiber failures. Fiber strengths must vary statistically and, thus, overall composite strength must be treated statistically. As fiber length increases, the chances of a weak spot or failure in the fiber increase. An analysis of the viscoelastic behavior of composites is given (using only 119 equations). Analysis of 3-dimensional reinforcement, to reduce interlaminar shear failures, is given also; only 43 equations were needed, but Eq. (33) was 3 pages long and Eq. (42) was 4 pages long. Eliptical fibers improve the transverse composite strength.

Fibers	Matrices
Glass, B, Be, Steel, Al_2O_3, Asbestos	Mg, Epoxy, Ti, Steel, B, 3 light alloys

73. Materials and Engineering Problems

Dow, N. F.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

General Electric Space Sciences Lab

There is more theoretical understanding of tensile testing than actual ability to correlate experiment and theory. Use of NOL rings for filament wound composites is useful, but compression tests are more difficult to interpret. Usually the values are only related to the 0.2% offset yield strength. Additions of third phases to the matrix can improve composite properties. Various test methods were discussed; NOL rings have a number of deficiencies. Measurements designed for homogeneous materials cannot be applied directly to composites without understanding the differences and interactions in composites. Studies of composite mechanics contribute to tailoring or designing composites for special applications, or using particular properties.

74. A New Look at Testing and Applications of Reinforced Plastics

Driver, W. E.

(Paper H-2, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Northrop/Carolina

Composite properties at angles other than 0° have been penalized due to reported low off-axis strength. Standard test methods give results which are too low except at 0 and 90°. A specimen was designed and tested with 45° plys continuous, and with unidirectional plies discontinuous in the test direction. Biaxial dome-shaped samples gave the same strength as uniaxial samples. Derived relationships for various loading conditions are tabulated. Under biaxial loading of biaxial cloth laminates, strength is the same for all directions. Shear mode failure is critical for orientations other than 0 and 90°. Beefing up edges will reduce shearing and gives better strengths at 0 to 90°. Bias testing of laminates in tension and compression is only useful for actual part data. Each design must be analyzed for the failure mode that is applicable.

Fiber	Matrix
Glass	Epoxy

75. Metal Coated Whiskers

Duff, R. H., Mansur, F., Wolff, E. G.

(Paper F-4, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

AVCO Corp.

Coatings on whiskers are useful for aiding wetting between whiskers and matrix and to obtain high interface bond strength, provide diffusion barriers, assure whisker separation in the composite, assist in alignment, and protect the whiskers during fabrication. Solutions and methods for applying electroless Ni, Cu, and Cr are given. Vacuum metallizing Cr, Ni, and 80Cr-20Ni coatings also is described. Electroless Cu on SiC gave good but uneven coverage, only the larger SiC whiskers showed coatings of Cr, but it was uniform; electroless Ni on SiC and Al_2O_3 gave complete and uniform coverage. Electroless Cu on Al_2O_3 was generally okay, with some uncoated areas. Metallizing gave spotty results, incomplete coverage in most areas. Hot pressing SiC in Co and Ni seemed to dissolve the SiC. Electroless and Cr metallizing coatings need more work; care is needed to avoid shadowing effects and to get uniform coverage. Multiple coatings of Ni and Cu on SiC are possible by electroless plating, but electroplating over the electroless coating gives better results.

Fibers	Matrices
SiC, Al_2O_3	Co, Ni

76. Survey of Composites Research in France

Duflos, J.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

Pierre Genein and Cie

Use of composites in France was surveyed. There is appreciable usage of glass-reinforced plastics in aircraft and missiles. In missiles, the main problem is temperature, which has been solved by using phenolics, improved sizings, silica fill, etc. Carbonized rayon yarn with round fiber cross sections has given good physical properties. C and graphite fiber fabrics have been laminated and used successfully in rocket nozzles. Precharring the resin stabilized the composite. Both dip and vacuum impregnation were used for glass fibers with advanced resins. Most effort seems aimed at the resins rather than the fibers. They also are doing some work on whiskers and mechanics of structures. Improved fibers are being made from glass; as many as 91 four-component and 20 two-component glasses were studied. A glass was developed with 0.6×10^6 psi UTS. Special equipment and handling techniques have been developed for making composites. A film was shown describing some of the filament winding techniques used; components pictured included tanks, pressure bottles, rocket nozzles, and integral nozzles and tanks.

77. Development of Design Criteria for Composite Materials

Ebert, L. J., Hamilton, C. H., Hecker, S. S.

AFML-TR-67-95, Apr. 1967

Case Institute of Technology

Theoretical and experimental study is given on the variables involved in rheological interface interactions for cylindrical composites. Large diameter rods of a soft core were encased in a hard case by machining out the core and shrink fitting it into the case, followed by swaging; the Al was cast into the Be core and then heat treated. Rule of Mixtures estimates of composite strength or predictions limited to elastic behavior are inadequate. Mathematical derivations for elastic and plastic regions are given, and boundary conditions are defined. Both maximum shear stress and distortion energy theory were considered in developing the analysis. Some of the simplifying assumptions are known to be wrong, but were used to aid computation. Experimental data gave results closer to those predicted by this analysis than Rule of Mixtures, but some deviations were noted, generally below predicted results. Interface interactions could be identified in most tests, but to varying degrees. Residual stresses also were found to be important in predicting composite behavior. The analytical predictions are erroneously low because of errors in the boundary conditions; the major error is the assumption that the case is a free surface. W-Cu composites made by vacuum infiltration showed a synergistic effect at 70-vol% W which is attributed to interface reactions (others found the same effect, and put the strain hardening of the matrix ahead in importance). It was possible to predict the synergistic effects, but not quantitatively. More work needs to be done to evaluate the end and residual stress effects, and the geometry of actual composites. (These composites are inside-out compared to the usual ones, where the ductile phase surrounds the harder one; some of the results may not be applicable to real cases.)

Fibers	Matrices
6061 Al, Cu, 4140 W	Be, 4340 maraging steel, Cu

78. Use of Boron Nitride Fibers in Composites

Economy, J., Anderson, R. V.

(Paper B-5, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Carborundum Co.

BN fibers have strength and moduli similar to glass, but are more resistant to abrasion, and are excellent electric insulators. Good thermal conductivity and dielectric properties make them ideal for ablative antenna windows. They are not suggested as metal reinforcements, but have been woven into cloth, felted, and combined with C to provide ablative protection. Both C-core and plain BN fibers have been woven into fabrics. They also improve thermal shock resistance of ceramics. Other applications suggested are space suits, thermal and electrical insulation, and corrosion protection.

Fiber	Matrix
BN	Resin

79. Modified Silicon Carbide Continuous Filaments

Elkins, P. E., Mallan, G. M., Shimizu, H.

(Paper D-5, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Marquardt

Additions of B to SiC give improved physical properties. A deposition system for continuous SiC filament formation is described. Essential process variables are temperature, partial and total pressures, gas flow rate, and fiber travel rate; also, reaction zone length is important. Best temperature was about 2000°F. The usual 1/2-mil W substrate resistance heated was used. Process variables affect both structure and composition of the deposit. The conditions at a ratio of Si:B = 6:1 give highest strength, a smooth surface, and metallic luster. Techniques for room and elevated temperature testing are described. Average strength is about 0.45×10^6 psi at room temperature, 0.105×10^6 psi at 3000°F. Vibration modulus ranged from 49×10^6 to 64×10^6 psi. Properties varied widely. Large grains, surface, and internal defects lower strengths.

Fiber
SiC

80. The Elevated Temperature Properties of a Ni Alloy Reinforced With W Wires

Ellison, E. G., Harris, B.

***Appl. Mater. Res.*, Vol. 5, No. 1, pp. 33–40, Jan. 1966**

Pratt and Whitney

Flat plates were prepared by winding W wire on Inconel 600 sheets, and rolling with Inconel plates above and below in picture frame. Composites were creep tested at 1200, 1400, and 1800°F, and compared with RT results. As vol% reinforcement increased, yield strength increased and elongation decreased. Samples containing very short fibers had little reinforcement above RT, and none at 1800°F. At elevated temperature, creep strain was reduced and rupture time was increased as vol% of W increased. Short wires improved creep, but critical length was dependent on relative strengths of wire and matrix. W mesh was found to strengthen the matrix significantly at as little as 0.42 vol%.

Fiber	Matrix
W	Ni

81. The Elevated Temperature Properties of a Ni Alloy Reinforced With Discontinuous W Wires

Ellison, E. G., Boone, D. H.

(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)

Pratt and Whitney

Discontinuous W wires, 3 mil in diameter × 1/16 in. and 3/16 in., were put into Ni by gas pressure bonding and by extrusion. Testing at 1200°F obtained good correlation between % W and UTS from 7 to 27%, for 1/16-in. fibers; for 3/16-in. fibers, UTS at 17% was greater than at 27%. No explanation was given, but it was suggested that kinking and agglomeration of the longer wires may have occurred. At 17%, 3/16-in. wires had YS of 39,500 vs 38,000 psi for continuous W. Wires aligned by extrusion gave about the same UTS for 7 to 27% W, but elongation of the aligned composites was better than random composites formed by gas pressure bonding. Creep data for 3/16-in. wires were scattered and hard to interpret. Processing obviously affected the properties. Aligned 1/16-in. wires gave better stress-rupture times at up to 17% W. Above 1800°F, density increased; long time stability decreased for samples with relatively high amounts of W.

Fiber	Matrices
W	Ni, Inconel, Hastalloy

82. Engineering Applications of Nonmetallic Composites

Epstein, G., Hribar, V. F., Smallen, H.

ASM Report C6-7.4, Oct. 1966

Aerospace Corp.

Review of applications of various non-metal and metal reinforced composites, primarily non-metal matrices, is given. Successful uses are described, such as nose cones and re-entry bodies, helicopter exhaust deflectors, window frames, seats, pipes, rafters, sheathing and roofing, power fuses, truck roofs and hoods, rocket exhaust extensions, etc.

Fibers	Matrices
B, glass, Al_2O_3	Epoxy, metals

83. Fiber-Reinforced Metals and Alloys

Farrell, K., Parikh, N. M.

AD-408734, July 4, 1963

IITRI

Hot extruded or cast and extruded composites are discussed; strength is more consistent with Be powder than Be fibers. High Be content gives agglomeration; low Be content gives better results.

Fiber	Matrix
Be	Al

84. Reinforced Pyrolyzed Plastic Cmposites for Aerospace Applications

Forcht, B. A., Harder, I. E., Seeger, J. W.

(Paper C-3, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

LTV Astronautics

Paper describes applications of pyrolyzed plastic composites in X-20 nose cones, fasteners (both threaded and unthreaded), heat shields, leading edges, tension shells for Mars landers (entry probes), re-entry thermal protection shields, and rocket nozzles. Additional suggested uses are hypersonic cruise lift and control surfaces and combined structural-thermal heat shields. Details of composition and fabrication of the composites were not given, but the data are referenced in the bibliography. In general curing was at 300–500°F and pyrolysis was at 1200–1800° for 24 h.

Fiber	Matrix
C	Phenolic

85. Wire Reinforced Metal Composites

Forsyth, P. J. E., Royal, J.

***Aeronaut. Soc.*, Vol. 70, pp. 794–796, Aug. 1966**

Royal Aircraft Establishment

Brief survey of wire reinforced metal composites is given. Strength of composites will depend on fiber. Advantages and problems are discussed in using composites, such as fabrication problems, reactions, getting high volume fraction of reinforcement, and reinforcement in two directions. Low transverse strength is a problem. Wire reinforced sheet materials are the most attractive materials to aircraft designers.

86. Fiber-Reinforcement: the Mechanics of Fiber-Reinforced Materials

Foster, B. K., Beer, F. J.

***English Elect. J.*, Vol. 21, pp. 18–25, July–Aug. 1966**

Mechanical Engineering Lab/Whetstone

Summary is given on the mechanics of reinforcement of composites. Packing densities are given for cylindrical fibers in 1 direction (0.907), in 2 directions (0.785), and in 3 directions (0.588). These are upper limits that are unlikely to be attained in practice. Elastic properties may be estimated by using the assumption of uniform strain (upper bound) or uniform stress (lower bound), in the case of moduli. Values derived from different fiber shapes, packing schemes, and spacings are true for specific directions in the composite. There is little experimental verification of theoretical values. Local stresses in the composite are important and should be taken into account, although this is very hard to do. Failure theory is discussed from the viewpoint of a representative volume or a local stress. Strength of the fibers is assumed to be less in effect that the average fiber strength of a bundle of fibers, unless the fibers are about the critical length (L_c) long, in which case the average is a good approximation. Fracture toughness may be increased if a weak plane (brittle interface) is present in a brittle matrix, and acts to turn the crack. The effect of fibers on fatigue strength is uncertain, but probably will improve it. Fiber ends may improve damping capacity in the composite. Both creep and stress rupture are markedly improved in composites. The effects of thermal stresses are little understood, and will have to be studied further for particular situations. Design of structures using composites is in its infancy and needs much more development.

87. An Evaluation of Various Properties of Unidirectional Composites

Foye, R. L.

(Paper G-4, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

North American Aviation

Methods for estimating the transverse modulus and longitudinal-transverse shear modulus are given. Method of analysis and application to plastic matrices are described mathematically. A computer program was developed to integrate the elastic equations. The effects of array shape, % reinforcement, and fiber shape are considered. Both solid and hollow glass fibers were studied. Diamond-shaped fibers gave less matrix stress concentration than round or elliptical fibers and increased transverse modulus appreciably.

88. Mechanical Behavior of a Continuous Filament C Composite

France, L. L., Kachur, V., Hengstenberg, T. F.

(Paper B-8, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Westinghouse Astronuclear

Continuous filaments of C in an unidentified C matrix were formed by graphitization at 2700°C. Tensile properties at room and high temperatures were excellent, with UTS of 36,000 psi at 2500°C. Fatigue strength also was very good; no failures after 3×10^7 cycles at 32,000 psi. Accurate UTS data were not obtained due to sample failures in the grips; several grip redesigns did not solve the problem. All failures were by shear between fiber and matrix at strengths near that of graphite. About 77% fibers were contained in the composite. Fiber surfaces were rough, but generally round. Major limitation is poor fiber-matrix bond, and low shear strength and strength perpendicular to the fibers.

Fiber	Matrix
C	C

89. Degradation of Composite Materials

Fried, N.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

U. S. Naval Applied Science Lab

There is too much technical literature on applications of composites to allow it to be classified comprehensively. Thus, this paper is limited to the environmental effects of water on glass-epoxy composites. Seawater exposure has less effect on the modulus than on UTS and fatigue properties, but both are decreased. The amount of degradation is pressure insensitive. It also is possible to get an increase in flexural strength during exposure to seawater due to a secondary curing effect. BF_3 amine epoxy laminated glass-epoxy has been shown to be pressure sensitive, and degradation is more severe as pressure increases. Hollow glass fibers are both pressure and time sensitive in sea water. High void content material is more sensitive than low void content material. It is suggested that there is some water penetration and attack near or at the interface. Finishing agents were used, but very thin layers were used. Drying after exposure can give appreciable recovery of strength compared to testing immediately after exposure. Coupling agents do seem to aid resistance to attack by water. There is considerable difference of opinion as to what happens at the interface.

Fiber	Matrix
Glass	Resin

90. Friction and Wear of Carbon Fiber-Reinforced Polyester Resin

Fullerton-Batten, R. K., Lancaster, J. K.

TR-66247, Aug. 1966

Royal Aircraft Establishment

C fibers in resins act to support loads and reduce the coefficient of friction, both for resin-resin contact and resin-steel contact. Fiber vol% and modulus had little effect, but orientation was important; fibers normal to the load were most effective. This system was more effective than flake graphite, MoS_2, WS_2, or BN fillers. The effects of water lubricants also were studied. Test methods are described.

Fiber	Matrix
C	Resin

91. Composite Materials

Fulrath, R. M.

Chapter 6 in *Ceramics for Advanced Technologies*, pp. 184–196, edited by J. E. Hove and W. C. Riley, John Wiley and Sons, New York, 1965

The importance of microstructure and matrix-reinforcement bond is emphasized in determining the properties of composites. Particular emphasis is on ceramic systems. Increasing size of dispersed particles reduces strength due to larger brittle matrix path for given amount of reinforcement, and higher proportion of voids. Voids also increased when proportion of reinforcement became too great.

Fibers	Matrix
Ni, Al_2O_3	Ceramics

92. Preparation, Structure, and Properties of Continuous SiC Filaments

Galasso, F. S., Basche, M., Kuehl, D.

***Appl. Phys. Ltr.*, Vol. 9, No. 1, pp. 37–39, July 1, 1966**

United Aircraft

SiC fibers, 2–4 mil in diameter are vapor deposited on 1/2-mil W wire at 1200°C from dichloromethylsilane by H_2 reduction. UTS was 0.3×10^6 to 0.4×10^6 psi; modulus was 55×10^6 psi. No reaction was noted between W substrate and SiC. No deterioration was noted after heating in air 1 h at 800°C. Process is very similar to B filament deposition process.

Fiber
SiC

93. Properties of Pyrolytically Produced Boron Fibers

Galasso, F. S., et al.

***Trans. AIME*, Vol. 236, pp. 1748–1751, Dec. 1966**

United Aircraft

The properties of vapor deposited B fibers are discussed. At deposition temperatures below 1400°C, the deposit is amorphous; between 1400 and 1500°C, the deposit is beta tetragonal; and above 1500°C, the deposit is beta rhombohedral with columnar grains. Mechanical and modulus measurements were made; strength increased as fiber diameter increased from 2 to 5 mil. Heating fibers for 1h in argon increased the scatter of tensile strengths; there was a minimum in strength for samples heated at 800°C, but at 1000°C, there was little change from room temperature. Rotating beam fatigue tests gave fatigue lives of 10^3–10^7 cycles, with 10^7 strengths of about 0.4×10^6 psi. Heating B fibers in air caused a gradual loss of strength up to 500°C.

Fiber
B

94. Amorphous B Fibers

Galasso, F. S., Kuehl, D., Tice, W.

***J. Appl. Phys.*, Vol. 38, pp. 414–415, Jan. 1967**

United Aircraft

Electron microscopy at 100,000× did not reveal any crystallographic structure in B vapor deposited on W wire. Bragg diffraction showed only amorphous rings. It is concluded that the B is amorphous.

Fiber
B

95. Unidirectional Solidification of the $BaFe_{12}O_{19}$-$BaFe_2O_4$ Eutectic

Galasso, F. S., et al.

***J. Am. Ceram. Soc.*, Vol. 60, No. 6, pp. 333–334, June 1967**

United Aircraft

Composites with 32.5% $BaFe_{12}O_{19}$ plates were grown by unidirectional solidification. The method is described briefly. It is hoped that material with directional magnetic properties can be produced in this way, but so far it has not been possible to orient all the platelet c-axes in the same direction.

Fiber	Matrix
$BaFe_{12}O_{19}$	$BaFe_2O_4$

96. Unidirectionally Solidified Eutectics for Optical, Electronic, and Magnetic Applications

Galasso, F. S.

***J. Metals*, Vol. 19, pp. 17–21, June 1967**

United Aircraft

A number of eutectics have been prepared for other than structural purposes. This paper is a means of bringing them to general attention. Rods will form when one phase of the eutectic is much lower in volume fraction than the other; lamellae will form if the volume fractions are about equal. Directionality of thermoelectric, magnetostrictive, and resistive properties have been obtained in various eutectics, which are described. Some applications are described also. Superconductive eutectics have been studied, but it has been difficult to control them during fabrication. Optical eutectics have been prepared, but many were not produced in clear form. Some work is continuing. Ferromagnetic eutectics have been studied extensively, and the effects of decreasing fiber diameter and increasing the volume fraction of fiber have been examined. These diameter and volume changes resulted in increasing the coercive force and saturation magnetization, respectively. Over 100 eutectic systems have been studied to some extent, and they can be evaluated for various uses. These include electrical and thermal insulation or conductors, optical imaging and conducting, and piezoelectric effects.

Fibers	Matrices
Sb, Bi_2Te_3, NiSb, CrSb, MnSb, FeSb, VAs, MoAs, CrAs, FeAs, LiF, CaF_2, Fe, Co, Ni, $BaFe_{12}O_{19}$	InSb, Te, GaSb, InAs, GaAs, Ag, Sn, Cd, Zn, Bi, Sb, NaF, NaCl, FeSb, FeS, Y_2Co_{17}, CoSb, Ni_3-Sn, $BaFe_2O_4$

97. Refractory Glass Fibers

Gates, L. E., Jr., Lent, W. E.

***J. Am. Ceram. Soc. Bull.*, Vol. 46, No. 2, pp. 202–205, Feb. 1967**

Hughes Aircraft

An electric arc furnace was used to test the fiberizing capabilities of 151 glass formulations; the compositions of the 25 most successful glasses are given. Tensile strengths ranged from about 0.25×10^6 psi to 0.4×10^6 psi (maximum of 2 sigma deviation) with individual values as high as 1.065×10^6 psi. Diameters ranged from 14 to 26 μm for various batches. Best results were with silica-magnesia-alumina, silica-alumina-zinc oxide, silica-magnesia-alumina-zirconia, silica-zirconia, and alumina-phosphate. Worst results were with high zirconia, calcia-alumina, and baria-alumina.

Fiber
Refractory glasses

98. The Synthesis of Boron Carbide Filament

Gatti, A., et al.

NASA CR-59907, July 1964

General Electric Space Sciences Lab

B_4C whiskers were grown best from B_4C vapor, but they were also grown by deposition on hot substrate. Maximum tensile stress of whiskers is 965,000 psi; modulus is about 65×10^6 psi. Wetting studies made in H_2 and vacuum. Fernico 5 is to be used in future. The 10-vol% whiskers were used in epoxy, and 4× strengthening was obtained.

Fiber	Matrices
B_4C	Fernico 5, epoxy

99. Study of the Growth Parameters Involved in Synthesizing Boron Carbide Filaments

Gatti, A., et al.

GE Reprint No. 344, June 1965

General Electric Space Sciences Lab

The B_4C fibers were grown by condensing vapors on a graphite mandrel. V, Nb, and Mo catalyzed the reaction, V is most effective; Ti, Cr, and Zr did not catalyze the reaction. The catalytic reaction was noted when B_4C powder became *exhausted* after several runs and could not produce whiskers. Analysis of as-received and depleted powder identified the elements that had been removed. Design and construction of the deposition apparatus are given. Chemical vapor deposition also grew whiskers, and additions of V to the vapor catalyzed the reaction. Since the process was inefficient and did not give as high quality whiskers, it was dropped rather than redesign the furnace. Adding B did not improve the deposition kinetics. Whisker growth is a function of temperature and concentration gradients. Metal matrix composites were made by vacuum infiltration; epoxy matrix composites were made by forcing the resin around the whiskers under pressure. Use of Fernico 5 as a matrix was unsuccessful because of problems in wetting and filling around the whiskers under the conditions of test. Similar problems were encountered with Al matrix. Powder mixing and pressing are being studied as an alternate fabrication method.

Fiber	Matrices
B_4C	Fernico 5, Al, epoxy

100. Study of Growth Parameters Involved in Synthesizing Boron Carbide Filaments

Gatti, A., et al.

GE Reprint No. 365, Sept. 1965

General Electric Space Sciences Lab

When B_4C whiskers were grown for 20 h rather than the usual 4 or 5 h, they exhibited a variety of sizes and shapes, depending upon location in the reaction chamber. Those near the inlet end were coarse and curved, and had polycrystalline dendritic growths; those near the top were short and fine, and had no growths. The coarse whiskers were very weak. Differences in growth are due to concentration and thermal gradients in the reactor, both are inherent in the system. Composites with Al were formed by hot pressing mixtures of whiskers and Al powder; best results were obtained with excess Al, melted under pressure. Strength was low.

Fiber	Matrix
B_4C	Al

101. Study of Growth Parameters Involved in Synthesizing Boron Carbide Filaments

Gatti, A., et al.

NASA CR-82447, 214-1B6(Final), Nov. 1966

General Electric Space Sciences Lab

B_4C whiskers were produced by condensation of the vapor on graphite plates and by chemical vapor deposition. Important factors in the condensation method are temperature, temperature gradient, reactor geometry, and use of V as a catalyst. Use of flat plates rather than round tubes gave 20-fold increases in production, but rates are still too low for efficient production. Some success was obtained by chemical vapor deposition. Major variables are gas flow rates, composition, velocity and pressure, thermodynamics and kinetics of reaction and growth, geometry, temperature, and nature of the substrate. This process is more efficient than condensation, but needs much more development. Many of the whiskers formed by condensation were curved, and had high residual strains due to growth conditions. Annealing above 1500°C straightened the whiskers, but lowered their strengths. Residual strains in annealed and unannealed whiskers were made and are discussed. While annealing reduced residual strains, it induces surface roughness which acts as a notch, thus, net strength is reduced. Composites were made by vacuum casting Al over whiskers which had been sputtered with Ti, then Ni; no more than 5–10% whiskers could be incorporated into the Al. Whiskers grown to date have been too imperfect. There have been too few to get good data on interface properties and mechanical properties, hence, continuous B_4C filaments were made by vapor deposition on W wire and composited with Al, using the same fabrication method. Continuous fibers gave full reinforcement efficiency, but chopped fibers gave only 70% efficiency. This reduction in efficiency may have been due to having less than the critical fiber length or volume, or misalignment. Other samples were hot pressed and tested, failing in the grips 3 times in 4. L_c for this system is unknown at this time.

Fiber	Matrix
B_4C	Al

102. Investigation of the Reinforcement of Ductile Metals With Strong, High Modulus Discontinuous Brittle Fibers

Gatti, A., et al.

NASA CR-82998, 1st Quarterly Report, Feb. 1967

General Electric Space Sciences Lab

Attempts to scale up whisker reinforced composites often result in only 30% or less of the predicted strengthening being obtained. This may be due to variations of whisker strength, average strengths being less than actual strength of a few measured whiskers, degradation during handling and fabrication, dependence of whisker strength on size or temperature, matrix embrittlement, homogeneity of whisker distribution, matrix flow properties, or others. Thus, continuous and chopped B_4C was substituted for B_4C whiskers to reduce the variables in composite fabrication and testing. Both the matrix and fibers are characterized before compositing. Fiber strength as a function of gauge length has been tested, and found to decrease as gauge length increases. Samples were made by vacuum infiltration of 1100 Al, or by hot pressing (unclear which; both given as sole fabrication technique). Single fiber samples were tested at room temperature and up to 660°C. No reaction was found between B_4C and molten Al. Calculated strength values of the fibers are higher than anticipated, and may be due to poor interfacial bonding and fiber slippage during test. Attempts to vapor deposit B_4C whiskers were unsuccessful, mainly due to contamination and process control problems. Changes in L_c as a function of temperature in single-fiber samples seem to be about as predicted.

Fiber	Matrix
B_4C	Al

103. Application of Advanced Fibrous Reinforcement Composite Materials

General Electric, Cincinnati

X66-83543, Apr. 1966

The task of this joint program of General Electric, Whittaker, and General Technology was to develop composite blades for jet engine use. Design optimization studies were made with and without redesign of components to utilize composite properties. A saving of 13% without redesign and 16% with very limited redesign was predicted for a cruise engine. Applying composites to the engines of a V/STOL aircraft could give a 20% weight reduction. Preliminary B-epoxy laminates had high resin content and low strength. Placing 1-mil tissue-glass and SiC whiskers between the laminates improved the modulus by about 25%. The density of B fibers was determined to be 2.75 g/cm^3, weighing in both alcohol and water, using various sizes of sample. B-resin 1/8-in. wide tape was characterized, and was found to be relatively uniform in resin content and flow. Resin formulation was uniform, but there was some fiber crossover. Rain erosion samples are being prepared, using B-metal samples. B/Ti-6Al-4V composites were prepared by diffusion bonding Si-coated B with Ti hydride interlayers at 1400°F for 10 min at 7000 psi. B-Al composites were thermally cycled and, also, were impacted with steel balls. B/Ti-6Al-4V composites were thermally exposed and were impacted with steel balls; little visual damage was noted at 100 and 200 ft/s. Also, 6-layer composites of Ni flash-coated B-Al with up to 40-vol% fiber were prepared. Fiberglass-epoxy compressor blades were prepared to check out the dies. B-epoxy blades were then prepared and examined. Integral blades/discs were prepared from fiberglass-epoxy to check the tooling, and B-epoxy composites are being prepared. Dynamic tests of blade shape changes were run. The effects of fiber orientation on flexure strength are given.

Fibers	Matrices
B, glass, SiC	Ti, Al, epoxy

104. Research on Improved High Modulus, High Strength Filaments and Composites Thereof

General Electric Space Sciences Lab

AFML-TR-66-98, Apr. 1966

Increased deposition rate was made for B on W from BCl_3 at low pressure; also, deposited B on C-coated SiO_2 both from diborane and BCl_3 and deposited B directly on SiO_2 using microwave heating. C-coated substrate was made by pyrolytic deposition of C on C-coated 0.68-mil SiO_2. Etching both W and C-SiO_2 substrate B fibers increased tensile strength about 2×. Heating 4 s in A or N_2 also increased strength. Failures during test attributed to failures at nodules. Most nodules occur where substrate is rough; shown for both types of substrate. Core-coating interface was site of most failures. Residual stresses in SiO_2 core fibers are tensile at surface; B-W composites where reaction occurs between core and coating have compressive residual stresses. B deposited on W at low temperature from diboranes show tensile residual surface stresses. No microcrystallites found in *amorphous* B. No crystalline phases found in B-SiO_2 composites; but found complex combination of crystalline and non-crystalline phases in B-W fibers. Coating by electrophoresis and in salt baths gave lumpy, low density coatings.

Fiber	Matrix
B	Resins

105. Summary of the 6th Meeting of the Refractory Composites Working Group

Gibeant, W. A., Mayhuth, D. J.

DMIC Report 175, Sept. 24, 1962

AFML

Solar plasma had sprayed W on W wire (similar to United Technology Corp. paper of Mar. 1966, Westec show). Marquardt used steel, SS, and Mo to reinforce Al_2O_3; and SS, Ta, Mo, and W to reinforce ZrO_2. General Electric worked with Ag and Al_2O_3 whiskers in Ag. Ni coating by vapor deposition improves bonding and composite strength 300–400%. H. I. Thompson is making various oxide fibers.

Fibers	Matrices
W, Mo, Ag, SS, Ta, steel, Al_2O_3	Al_2O_3, W, Ag, ZrO_2

106. Influence of Some Processing Variables Upon the Elevated and Room Temperature Strength of Ultrafine Be Wires

Golding, W. H.

NAEC-AML-2441, Apr. 1966 (AD-633992)

U. S. Naval Air Engineering Center

Ingot and powder Be wires made by Berylco were treated by several processes to improve surface finish and mechanical properties. Strengths at temperatures up to 1100°F were determined on 5-mil fibers with and without: surface etching using two reagents; removing 1/2 mil and 1-1/2 mil; coating with TiO_2; and Ni-clad. Any treatment which improved surface finish and removed the severe die marks increased tensile strength at all temperatures. Ni-clad also improved tensile properties; but at 1100°F, the Ni-clad separated from the Be. Ni also added about 20% weight penalty.

Fiber
Be

107. Summary of Composites Research in England

Gordon, J. E.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

Explosives Research and Development Establishment, Ministry of Aviation

Summary of composites work in England is given. There is about an order of magnitude less effort in England than in the U. S.; their fewer programs are more concentrated and oriented mainly towards laboratory work and less towards fabrication. There is no work on B, but glass, asbestos, graphite, and whiskers of SiC and Si_3N_4 are being studied. SiC whiskers are compatible with a number of matrix materials, have good specific modulus, and have the best specific strength. Equipment and techniques were shown for making both SiC and Si_3N_4; Si_3N_4 is no longer being made, since growth rates are very slow and the costs are very high. Large furnace chambers are used in which the SiC is deposited on a graphite heater element by vapor deposition. Cost of SiC whiskers produced in this way is about $15–30/lb. The mass is treated by mixing it with an organic to form a slurry after centrifuging, and by forcing the slurry through an orifice to get an aligned fiber mat. The binder is burned out, and the resultant mat formed into a composite. Uses are being sought for the shorter whiskers removed in centrifuging. They are beginning to extrude SiC whiskers (short ones) in sintered Al powder.

108. Crystallization of Massive Amorphous B

Gillespie, J. S., Jr.

***J. Am. Chem. Soc.*, Vol. 88:11, pp. 2423–2425, June 5, 1966**

Virginia Inst. for Scientific Research

Chemically vapor deposited B is generally considered to be *amorphous*. Found that heating B fibers to 1050°C for as little as 15 s gave beta-rhombohedral surface structure. This occurred whether the substrate was W or C-coated silica. Recrystallization probably is by surface diffusion. Only the beta-rhombohedral form was found, regardless of the temperature to which the fibers were heated. The *amorphous* form appears to be a microcrystalline beta-rhombohedral form.

Fiber
B

109. Advanced Metal Composites—A Status Report

Greenstine, R. B.

***Metals Eng. Quart.*, Vol. 18, pp. 8–16, Feb. 1967**

AFML

Metal matrix composites have certain advantages over non-metals: high shear strength, higher operating temperatures, better ability to take triaxial stress, more resistance to impact, easier to process, drill, etc. Describes AF program to fabricate reproducible test samples by various means. Solid state processes include: powder metallurgy, which is limited by random packing to about 20-vol% fibers; diffusion bonding, which is limited to 30–40-vol% fibers (but some workers report 45–50-vol% fibers by diffusion bonding); hot extrusion; hot rolling; and isostatic compaction or pneumatic compaction. Liquid state processing includes casting, vacuum infiltration, and plasma spraying. Maximum theoretical fiber content by these processes is 80 vol%, with a practical limit of about 75 vol%. Molecular processes include vapor deposition and electrodeposition, which are limited to about 50-vol% fiber. Combination processes also are possible. AFML has made samples, mostly for tensile test, by casting, powder metallurgy, diffusion bonding, plasma spraying, and electrodeposition. The process must produce a good matrix without degrading the fiber. The actual strength was compared with Rule of Mixtures strengths for each material and fabrication method. Results are given for B-Al and SiC-Al by powder metallurgy, B-Al by electrodeposition, plasma spraying, and diffusion bonding, B-Ni by powder metallurgy and electrodeposition, SiC-Ni by electrodeposition, B-Cu by powder metallurgy, and B/Ti-6Al-4V by diffusion bonding. Generally, there was good translation of the modulus into composite properties, but poor translation of the tensile strength, probably because of damage to the fibers during processing. Diffusion bonded B-Al, B/Ti-6Al-4V, and SiC-Ni by electrodeposition had strengths that most nearly matched theoretical values. Vacuum infiltration of Al around Ni-coated B generally has not been successful because Al reacts with Ni to form an eutectic which attacks B after a few seconds at 1180°F. Plasma spraying Al on B gives up to 15% porosity, and layers of deposit do not bond unless a follow-up hot-pressing operation is included. The composite also warped due to thermal stresses in cooling. There was severe fiber degradation, either by thermal shock or chemical reaction. The best way to form Al-B composites is diffusion bonding or electrodeposition, which gave the best results at 12.5-vol% fiber or less. Before using a composite for any application, there must be better understanding of its behavior during use. Specific fabrication techniques are suggested for particular applications and materials combinations. Much more work on fabrication methods and fracture behavior is needed. Non-destructive and standard destructive tests must be developed. Different processes are best used for specific components and applications, and this requires development of many different subscale fabrication techniques.

Fibers	Matrices
B, Sic, C, B_4C, Al_2O_3	Al, Mg, Cu, Ni, Ti-6Al-4V

110. Filament-Reinforced Refractory Metals

Greszczuk, L. B.

(Paper F-3, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Nov. 9–11, 1966)

Douglas MSSD

Strength, elastic properties, elevated temperature behavior and fabrication of composites are discussed. Typical high temperature environments are cited, and properties of fibers and matrix materials are given. Fabrication is by chemical vapor deposition of the refractory metal; precompression of the matrix is accomplished by prestressing the fibers before depositing the metals. Deposition conditions are described. Test results are given. Tubular samples of pretensioned B fibers and W wires were coated with vapor deposited W. The W coated W samples were tested as cylinders in bending; the B reinforced samples were tensile tested. Many of the B-W samples failed in the grips, or by progressive fiber fracture. Both thermal and mechanical prestressing of the filaments was used, and seemed to be successful in strengthening the composites. Vapor deposition parameters were not optimum. Subsequent working of the vapor deposited material may improve its properties.

Fibers	Matrices
W, Mo, B, C	W, Mo, Al, Cb, epoxy

111. Trends and Applications of Structural Composite Materials

Grimes, D. L.

AGARD Report 523, Nov. 11, 1965

Whittaker Corp.

General summary is given on the history and progress in composite materials, with emphasis on non-metal matrix applications. Present research and development areas are fiber development and supply, fiber handling and processing, micromechanics, and applications which demonstrate composite advantages. Future applications are suggested in jet engines, as compressors, aircraft structures, and helicopter blades.

Fibers	Matrices
Glass, B, C, Be	Resins, metals

112. Attachment Concepts and Problems in Fibrous Composite Aerospace Structures

Grimes, G. C.

(Paper G-5, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Southwest Research Inst.

General information on structural joint design, which combines experimental and theoretical studies, is needed to permit full utilization of composite properties. Present joint studies are specifically for particular applications or joint conditions. Properties of the composite and adhesive must be known, as well as failure criteria, and load transfer configurations. The system considered is a double lap joint for airframe structures that are adhesively bonded. Mechanical fastening is not considered. Basic equations and design curves are described for general cases. The need for additional test data to resolve some conflicts in the analyses is noted. Important design parameters include: joint function, type of load, magnitude of load, strain compatibility of joint elements, joint environment, and cost and space requirements. The effects of tapering the layers of composite are discussed, as are stress concentrations. Appendices give discussions of double lap theory and test methods.

113. Analytical and Experimental Micromechanics Studies

Grinius, V. G.

(Paper G-1, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Whittaker Corp.

Micromechanical behavior of composites under tension, bending, shear, and fatigue was studied analytically and experimentally, and the results were compared. Failure by statistical accumulation of fiber breaks was used as an analytical system. Unidirectional and bidirectional samples were tested, and was found to agree generally with this mechanism. Crossplys at 45° failed in shear at lower than predicted strengths for tensile failure. Torsional samples with unidirectional and 45° crossplys were tested to get shear strength. Unidirectional samples failed by interlaminar shear. Crossplys failed in tension. Glass reinforced composites failed in shell buckling due to the low effective modulus. Torsional fatigue failures were similar to static failures. Modulus decreased as number of cycles increased. Matrix crazing occurred well before composite failure. Problems in theoretical treatment of composites are very complex, requiring simplifying assumptions. Process variables in making and testing composites makes comparison of data from different investigators hard to correlate.

Fibers	Matrix
E-glass, S-glass, B	Epoxy

114. Boron Filaments and Composites—Their Evaluation and Potential

Gunn, K. M., Langley, T. M., Link, D. S.

Texaco Experiment, Sept. 1966

Test and evaluation methods for B filaments and composites are described, including some test results. They state that the short-beam shear test is the most reliable, and is very sensitive to matrix properties and fiber-matrix interface conditions. Standard reduced-section tensile specimens do not give reliable data, since all fibers are not stressed equally. They like the General Dynamics/Ft. Worth type of test, which is a 2-point loading of a composite beam with a B-resin skin. Compression testing is done on laminates or tape bonded to flattened ball bearings. Potential applications of B composites depend upon testing refinement and more confidence in test results and material uniformity, design ingenuity, manufacturing technology development, lower costs, and better matrix systems. Use of small amounts of B fibers in conjunction with glass is one attractive area which is actively being developed, and may be applied very soon.

Fiber	Matrix
B	Resin

115. Interfacial Investigations of B Fiber-Reinforced Plastics

Gutfreund, K., Broutman, L. J., Jaffe, E. H.

(Paper E-3, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

IITRI, AFML

Morphology, topographology, and chemical relationships at surfaces are reviewed. An understanding of these relations is needed to develop appropriate surface treatments for fibers. Surface characteristics of B fibers and the effects of various surface treatments are discussed. The B surface is rough and contains contaminants. Removal of these defects can increase fiber strength, or can improve bonding, or both; some treatments increase fiber strength, but decrease bonding or wet strength of the composite. Techniques used for treating the B surface are described. B has a different affinity for different organic compounds. Interfacial bond strength in shear and tension was determined for single B fibers in several resins, using different surface treatments. Pretreatment with Cl_2 or BCl_3 aids wetting and coverage of the B by iminofunctional compounds. Tensile and shear test specimens and techniques are described, and debonding strengths for untreated shear and tensile samples are given.

Fiber	Matrix
B	Resins

116. Laminate Optimization for Filamentary Composites

Hackman, L. E., Stotler, C. L.

(Paper C-1, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

North American, Columbus Div.

Paper presents a method for designers to use in optimization of laminates in filamentary composites, based on minimum cost and weight and structural reliability. Design of face sheet, spars, and ribs by use of a computer program is described. Use of polar diagrams of loads and composite properties is illustrated for externally loaded cylinders.

117. Research on Whisker-Reinforced Metal Composites

Hahn, H., et al.

DMIC No. 63663, Feb. 1966 (Letter Report No. 5)

Melpar

Vapor deposited Ni from carbonyl on SiC, but not on Al_2O_3 because of side growths that result in tangling. Al-10%Si alloy with 15-vol% Ni-coated Al_2O_3 whiskers gave improved high temperature strength, but below 400°C, controls were stronger. Samples were made by liquid phase, hot pressing, and hot rolling.

Fibers	Matrices
SiC, Al_2O_3	Ni, Al

118. The Failure of Cu-W Fiber Composites in Repeated Tension

Ham, R. K., Place, T. A.

***J. Mech. Phys. Solids*, Vol. 14, pp. 271–280, 1966**

McMaster Univ.

Composite fabrication was by casting the Cu around bundles of W wires under N_2. Both simple tensile tests and tension-tension fatigue tests were run. The critical volume % of W was about 6% in tension, and about the same in fatigue. Fatigue fractures for 1.5% W samples were indistinguishable from pure Cu failures, but at 7.4%, failures were brittle. Wire fractures could be heard after only a few cycles in fatigue, even at low stress levels, particularly for low vol% W. Samples with less than the critical vol% of W were fatigue limited by the matrix; those with more than the critical vol% W were fatigue limited by the fibers, and appreciable (but less than expected) reinforcement occurred. Failure is believed to occur by fracture of fibers which forms an internal fatigue crack. This spreads to adjacent fibers, and either fractures them, or moves parallel to the fiber along the interface, or builds up stress concentrations at the crack tip as the matrix fatigue hardens, at which time it can break adjacent fibers which may have stopped it.

Fiber	Matrix
W	Cu

119. Structure of Vapor-Deposited B Filaments

Hammond, M. L., Lindquist, P. F., Bragg, R. H.
AFML-TR-66-358, Nov. 1966
Lockheed Missile and Space/Sunnyvale

The morphology and crystallography of B filaments from two suppliers were studied as a function of deposition conditions, location along the fiber, strength, deposition temperature, etc. Several types of nodules on the surface were described and their sources identified. The radial cracks common to vapor deposited B fibers are attributed to fractures which occur at the core during deposition due to volume changes when the W core wire is boridized. The borides present are tentatively identified. The B is composed of fine crystallites about 30 Å in diameter with occasional 100 Å crystals. Nomenclature for describing B fibers morphologically is suggested. The borides had crystallite sizes of about 0.1 μm. B has about the same structure whether deposited at 870 or 1260°C, and changes in deposition conditions have only slight effects on crystallography, but they do change morphology. The rings, formed where the fiber passes from one deposition chamber to another, etched faster than the rest of the fibers produced by one vendor; the other fibers etched about as fast as the rings of the fibers made by the first vendor. Metallographic techniques for fiber mounting and polishing are described. Differences in the appearance of B fibers are attributed to nucleation and growth on the fiber surface in the first case, and vapor phase nucleation in the second. Previous claims that vapor deposited B is amorphous are denied. The structure is that of very fine, unoriented tetragonal or α-rhombohedral B with crystals less than 1 μm. Heating B fibers in argon at 990–1050°C for 15 to 48 h caused porosity in the fibers; electron beam heating caused formation of 1500 Å particles of α-B. Cyrstallographic identification by electron and X-ray diffraction gave the same results. Small crystals of β-rhombohedral B also were detected in most fibers.

Fiber
B

120. Proposed Method of Density Determination for Some Inorganic Fibers

Hanchak, S. J., Spain, R. G., Mahieu, W.
AFML-TR-67-65, May 1967
AFML

Measurement of density of small, fine fibers should be practical without use of very sophisticated equipment and skilled personnel. A method is described which is very accurate, needs simple equipment, and requires only normal skill. The average density at which the fiber just rises and just falls in a column of liquid of known and adjusted density is taken as the fiber density. Liquid density is controlled by using two or more liquids of known density which are misible, and mixing them in the test column. Details of experimental procedure and lists of liquids for various densitiy ranges are given.

Fiber
Glass

121. Transmission Electron Microscopy of Interfacial Areas in Metal-Matrix Composites

Hancock, J. R.
***J. Compos. Mater.*, Vol. 1, No. 2, pp. 136–142, Apr. 1967**
Midwest Research Inst.

Al alloy 2024 with 25-vol% AM355 stainless steel wires was heat treated 1/2 h at 495°C and water quenched. Tensile-tensile fatigue tests were run at 16,000 psi for 8000 cycles, after which the Al matrix was examined by electron microscopy. Specimen preparation by electrothinning and the solutions and techniques used are described. Dislocations seem to increase as the interface is approached. Photomicrographs of the composites are shown. Within 25 μm of the interface, it is possible to identify dislocations. For this system, the effect of the interface on mechanically induced plastic behavior is limited to 25 μm or less from the interface. Each composite system will require separate development of examination techniques.

Fiber	Matrix
AM355	2024 Al

122. Some General Aspects of Interfaces in Composites

Harrod, D. L., Begley, R. T.

(Paper E-1, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Westinghouse Astronuclear

The importance and nature of interfaces in composites are discussed, taking into account the importance of prior composite history and characteristics of the bulk materials. Classification of interfaces according to atomic registry is described. Various interfacial interactions and physical characteristics, as well as the effects of thermal and mechanical history on the interface, are noted. Kinetic and thermodynamic data are needed to predict the equilibrium interface state, but these data are rarely reliable. Compatibility studies made to establish such data should be more standardized. Topographical features also are important, both as aids to bonding and as stress raisers. The different types of bonding are mechanical, physical, and chemical. Theoretical strengths of chemical bonds are treated from the atomic and thermodynamic approach. The need for good wetting, as defined by low contact angle, and for good bonding, as defined by high bond strength, is emphasized. An understanding of composite behavior requires appreciation of the coupling effects between fiber and matrix and the nature of the interface. Both elastic and nonelastic approaches are considered, and the effects of the interface for each case are examined. In the elastic-plastic case, it is suggested that mechanical bonding may be as important as chemical bonding. It is difficult to identify the location of fracture initiation in composites, but this information is important in understanding composites and interfacial phenomena. Composites are inherently unstable, and interfacial chemical reactions are to be expected. Strong interfaces can be formed if the phases wet each other; what looks like an interface failure may really be a matrix failure next to the interface. Strength values in testing are not measures of bond strength, since the practical strength is determined by the overall nature of the interface. Micromechanics of composites is the only rational method of analysis of the effect of interfacial properties on the composite. Interfaces are generally characterized by a change in composition and crystal structure, and atomic disregistry. It appears that mechanical bonding always will be operative.

123. The Elastic Moduli of Fiber-Reinforced Materials

Hashin, Z., Rosen, B. W.

***Trans. ASME, Ser. E: J. Appl. Mech.*, pp. 223–230, June 1964 (Also GE Reprint No. 298)**

General Electric Space Sciences Lab

An analytical study of the elastic moduli of composite materials is presented. Expressions are derived for hexagonal and random arrays, considering only the fiber ends. Rigorous results were calculated for the hexagonal array, but results are not applicable to the real systems; while the random array gives rigorous results only for limiting cases, these are probably not of any practical significance. Irregular fiber diameters are approximated as cylinders. Effects of hollow glass fibers are also calculated.

124. Survey of Composite Materials Micromechanics

Hashin, Z.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

Univ. of Pennsylvania

The flood of papers with many viewpoints sometimes obscures fundamental principles and theories needed for progress. There is a large scatter in reported data; values reported are statistical. Physical properties should be computed on the basis of phase properties and geometry. The vol% fibers alone is insufficient to describe the composite behavior. Dielectric properties can be compared to elastic properties. Tensor averaging theorems can be used for plastic, elastic, viscoelastic, and viscous flow properties. A statistical homogeneous composite with homogeneous boundary conditions has statistically homogeneous stress and modulus. The requirement for interface continuity makes computation difficult. Using the principle of minimum potential and complementary energy requires that a statistical approach be used to find stress, strain, and displacement. This approach needs much more work. The Rule of Mixtures is good only to give an upper bound to composite properties, although, not a very good one. Scatter bands are reduced using improved boundary conditions, but as component stiffnesses increase, the theoretical property boundaries increase. The real case of a semirandom composite is the hardest to compute; both random and ordered composites are much easier. If the only thing controlled in a composite is the % fiber, there is wide scatter in the data on modulus. Poisson's ratio is not given by the Rule of Mixtures. Use of a random array boundary condition for B-epoxy gives better bounds than use of an arbitrary cylinder. If a composite is inelastic or viscoelastic, properties, such as effective relaxation modulus and effective creep compliance, must be defined. There are only a few cases where the effective elastic modulus of a composite is known and the calculations can be made. It is difficult to predict the plastic strength of composites. If a regular array of rigid fibers is cut at 45°, the strength is that of the matrix. For porous materials, the yield strength is bounded by the % porosity. Elastic-plastic predictions cannot be made, but for an assembly of composite spheres, the plastic behavior can be treated. The % pores has been studied and has a significant effect.

125. BiCompositional Carbon-Silica Fibers

Hawkins, H. T., Schmidt, D. L.

(Paper D-7, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

AFML

C-silica fibers were prepared by a number of techniques, which are described briefly. Processes included dispersions of particulate silica, molecular dispersions of silica, dipping in aqueous solutions with and without subsequent heat treatment, vapor deposition, and electrophoresis. Other oxides such as zirconia, vanadia, Cr, Ti, Mo, and Co also were introduced into the coatings. Pyrolytic C has been deposited on silica fibers. Only limited data on properties are available. Epoxy composites have been prepared successfully, using various types of C-silica fiber. Phenolics with these reinforcements have been tested as ablative materials in rocket nozzles, and have shown less erosion than C or C composites in oxidizing atmospheres. Potential uses include heat shields, high temperature structures, elastomerics thermal insulation, and for specialized electrical uses.

Fiber	Matrix
C-silica	Epoxy, phenolics

126. Survey of Composite Structural Mechanics

Hedgepath, J. M., Haskell, D. F.

(Presented at the International Conference on Mechanics of Composite Materals, Philadelphia, Pa., May 8–10, 1967)

Martin Marietta

They examined 1187 papers on composites, and found that 36% of the papers were on theory of composites, with the least amount of work on components. About 68% of the theory was on mechanics. Fracture mechanics theory was reviewed in relation to the data, e.g., stiffer matrices give shorter critical fiber length, and better overall composite properties. Both tensile and compressive strengths are considered. Use of hollow glass fibers increases the compressive strength of glass. Plots of the yield surfaces for laminated composites were shown to be very different from those of isotropic materials. The statistical distribution of materials properties must be considered when studying composites. For a compressive cylindrical structure, use of isotropic materials gives better results than any winding pattern. If enough layers are used, behavior approaches a homogeneous structure. Much more work is needed on joints which are a problem.

127. Applications of Composite Materials in Space Vehicles

Heldenfels, R. R.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

NASA Langley

The applications and problem areas for composites in space vehicles were summarized. Development of materials is a critical item. There is limited data on environmental effects, and no experience in composite reliability or reproducibility. The main advantage now is increased design flexibility. Applications for various vehicles were listed. Honeycomb material in launch vehicles would improve mass efficiency, but solid Be is as good as honeycomb. Relative weights of tubes and joints were shown for truss structures; joints are a problem area in composites. Be is almost as good as B-epoxy. Generally there are few present applications in spacecraft. A few possible uses are Mars landers where they may be useful as aeroshells; but Be is better. Another possibility is lunar labs or shelters.

128. Use of Composite Materials in Naval Ships

Heller, S. R., Jr.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

U. S. Naval Ship Engineering Center

The Navy definition of a composite is anything that is not metal, wood, or paint. Composites represent a great advance in materials technology. Typical applications include radomes, boat hulls, antenna masts, pipes, sonar housings, submarine rudders, deckhouses, antenna housings, superstructures, minesweeper plugs, tanks, and tubing. One composite that was unsuccessful was a hull made with paper core honeycomb; it became waterlogged, but still floats. Advantages of composites include lower maintenance, weight and cost, and improved performance. For some applications, composites are the only good solution for adequate performance (i.e., radomes). Biggest problem areas are lack of reproducibility and inadequate inspection techniques. Non-destructive testing methods are very poor and must be improved before composites can be used more extensively and in more critical applications.

129. Surface Treatments for Fibrous Carbon Reinforcements

Herrick, J. W., Gruber, P. E., Mansur, F. T.

AFML-TR-66-178, Part I, July 1966

AVCO Corp.

A major strength limitation in composites is bonding between fiber and matrix. Lack of proper bonding leads to shear failures along the fiber and premature failure. Various surface treatments are considered to improve bonding between C and graphite yarn and organic matrices. Orientation of plies at 5–7° off parallel to fiber direction gives little loss in strength. Crimped or non-straight fibers tend to elongate when the composite is stressed, giving local stresses and delamination of the composite. Better bonding can reduce or eliminate this effect. Coating fibers with nylon gave poorer lap shear strength in phenolics and better strength in epoxies, probably because wetting is better in epoxies. Use of oxidizing agents to activate the surface of the C and graphite gave better bonding and strength, but if not properly controlled the fiber strength is reduced. Hot HNO_3 or air oxidation seemed most effective. Boiling in H_2O, solvent extraction, and various surface finishes (such as chrome complexes, silanes, or other glass finishes) did not increase interlaminar shear strength, and often reduced it. Oxidation resistant coatings for ablative composites also were studied, but they are beyond the scope of these abstracts.

Fibers	Matrices
C, graphite	Phenolic, epoxy

130. Research on B Filaments and B Reinforced Composites

Herring, H. W., Baucom, R. M., Pride, R. A.

(Paper B-3, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

NASA Langley

B fibers prepared from halides and organometallics were tested and compared. The average tensile strength of organometallic fibers was low; these represented very early production, and probably are not indicative of present fibers. Strengths of halide fibers were about 50–70,000 psi below present fibers. The strength of fibers decreases as test length increases (as expected, since the chance of flaws increases). High temperature exposure before test gave very low strengths after 1 min at 1500°F or above; exposure in air gave lower UTS than exposure in A. Both B-epoxy and glass-epoxy composites were made, tested, and compared. B-reinforced cylinders are more efficient than glass for axial loading, but are less efficient for internal pressurization. B is also more efficient for external pressurization on a weight basis.

Fibers	Matrix
B, glass	Epoxy

131. Progress Report on Chromium Composite Materials

Herron, R. H.

In *Summary of the 8th Meeting of the Refractory Composites Working Group*, ML-TDR-64-233, Vol. II, Jan. 1964 (AD-470694)

Bendix Corp.

An alloy of 6% MgO in Cr was formed by extrusion of hydrostatically pressed and sintered billets at 2100°F, at 9:1. Sheet was rolled to 50 mil by breaking down at 2200°F and warm rolling at 900°F. A 3% MgO in Cr was drawn to 5-mil wire. It was drawn from 1/2 in. to 30 mil at 2200°F, and then drawn at room temperature. The UTS of the 6% MgO-Cr material was 45,000 psi minimum, 51,000 psi average after annealing at 1800°F. At 2300°F in air, the average UTS was 4100 psi. No fibering of the MgO was noted.

Fiber	Matrix
MgO	Cr

132. Fracture Mechanisms in Controlled Cu-Cr Eutectic Alloy

Hertzberg, R. W., Kraft, R. W.

***Trans. AIME*, Vol. 227, pp. 580–585, June 1963**

United Aircraft

Unidirectional solidification of the Cr-Cu eutectic gave about 2% discontinuous whiskers with L/d of about 100. Fewer voids were found in the controlled eutectic than in normal Cr-Cu eutectics. Failure of tensile specimens was in shear, and occurred mainly at voids formed when the Cr whiskers broke. The interface bond was good, with no failures initiating there. (Both % fiber and L/d were too low to reinforce the Cu; the whiskers probably weakened the matrix.)

Fiber	Matrix
Cr	Cu

133. Metal Composites, Their Reinforcing Components, and Their Problem Areas

Herzog, J. A.

AFML-TR-67-50, Mar. 1967

AFML

Description of the development, advantages, disadvantages, and problem areas in composites is given. Fiber production, characterization, compatibility with matrix materials, sample fabrication, and understanding micromechanics of composites are major problem areas discussed. Early data on whisker strength was obtained by bending tests, which were hard to correlate; tensile testing has been developed and is now reliable. Fabrication techniques are surveyed. All but plating required heating the composite and may be detrimental. Protective coatings to prevent these reactions are possible; they also may aid wetting between composite and fiber. Orientation of whiskers is important and may present problems. More than 8° misalignment reduces reinforcement efficiency appreciably. Problems of sample design, fabrication, and testing are noted. The ratio of fiber length to diameter is very important, as is the ratio of composite shear strength to tensile strength. Application of composites in structures and their experimental verification is needed, as are studies of newer reinforcements, protective coatings, effects of geometry, interface behavior, wetting of surfaces, etc.

134. Refractory Composite Materials Development

Hessinger, P. S.

In *Summary of the 8th Meeting of the Refractory Composites Working Group*, ML-TDR-64-233, Vol. III, Jan. 1964 (AD-470695)

National Beryllia

BeO whiskers were grown by oxidization of Be and by decomposition of BeOH. Whiskers up to 1-1/2 in. long were formed and Be-epoxy composites with up to 60% whiskers have been made. Metal-plated BeO particles were pressed into ceramic matrix composites.

Fiber	Matrix
BeO	Epoxy

135. Boron Carbide Continuous Filaments

Higgins, J. B., et al.

AFML-TR-65-354, Part II, Aug. 1966

General Electric Space Sciences Lab

B_4C fibers were deposited onto a 1-mil W substrate in a multi-stage glass reactor by depositing B first, followed by B_4C from BCl_3, H_2, and CH_4. Surface appearance was affected by nucleation mode; surface nucleation gave a rougher surface, while gas phase nucleation was much smoother. Fiber strength is sensitive to deposition temperature, with 1150°C as the optimum. Deposition rates are sensitive to C content of the gas, with lower deposition rates in the presence of C; C seems to increase uniformity of fiber strength. Highest modulus and strength values were obtained at 27.5% C. The filaments were not affected by mineral acids or H_2O_2. Composites with epoxies were made and tested in bending, and improved mechanical properties were obtained. B_4C was deposited on silica substrates from decaborane, but strength properties could not be determined. Evaporation and condensation from solid B_4C was unsuccessful.

Fiber	Matrix
B_4C	Epoxy

136. A Survey of Developments in Whisker Composites in the United States

Hoffman, G. A.

AD-614991, Apr. 1965

Rand Corp.

Report gives a survey of whisker formation and characterization, composite fabrication and testing, and analyses of composite physical and mechanical properties. Thirty-two references are included (mostly by contract number) with brief abstracts of work and results of each.

Fibers	Matrices
All	All

137. Be Wire Reinforcement of Al and Ti

Hoffmanner, A. L.

(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)

TRW

Al-Be composites were formed by making a slurry of lacquer-coated Al and Be wires with diffusion bonding at 900°F. Times ranged from 5 min to 4 h; pressures ranged from 24,000 to 8,000 psi. Up to 92-vol% Be was obtained. The wires showed appreciable deformation. At 41% Be, there was reasonably good alignment. For a given fabrication technique there was good reproducibility. Maximum Be content was obtained by vacuum metallizing Al on the Be wires before diffusion bonding. Be-Ti composites were drawn at 750–800°F, and exposed about 200 h at temperature. From 32 to 68 vol% Be, UTS was independent of amount of reinforcement, at 0.1×10^6 psi for 15 min. There was some YS dependence on %Be at 8000 psi for 4 h. The Be wires in Ti also were deformed, and there was incomplete bonding. The modulus was measured acoustically, and agreed well with theoretical values.

Fiber	Matrices
Be	Al. Ti

138. Formation of Continuous Filaments by Drawing a Substrate Through Molten Boron

Holinbeck, D. G.

AFML-TR-66-121, June 1966

Bjorksten Research Lab

Coating various fibers with B was attempted by passing the substrates through a molten pool of B. Pulling the substrates vertically through a melt and, then, a hole in a BN crucible was unsuccessful; hence, they attempted to draw them through a molten bead on a pedestal. Screening of candidate substrates for reaction with the B was done by firing the fibers through a B melt with a spring-operated piston gun. The 2-mil W wire could not be coated successfully; nodules of B rather than a smooth coating were obtained. Silica was coated by shooting it through the B pool, but Cu and Fe melted in passing through the B.

Fiber
B

139. Boron Carbide Filaments from Organoboranes

Hough, R. L.

(Paper D-3, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Hough Lab

B_4C fibers have been produced by vapor deposition from organoboranes onto 10-mil heated W wires to check out the process. Later batch processing was done with 1/2-mil W wire. A brief discussion of the use of carborane-10 is given. Fibers formed below 1000°C were too brittle to test. Modulus was 40×10^6 psi, UTS ranged from 43,300–113,400 psi; and density was 2.48 gm/cm^3.

Fiber
B_4C

140. A Study of the Use of Vapor and Vacuum Deposition Technique for the Development of High Strength Filamentary Materials

Hughes, E. J.

N66-26257, June 1965

Pyrogenics

Pyrolytic graphite was deposited on liquid metal substrates. Metals were chosen for melting point, vapor pressure, and reactivity with C. Pt was used first, but it reacted with C boat. Au substrate gave deposit from CH_4. Au was chosen rather than Cu for initial work (experimental equipment design was dubious, as was substrate selection).

Fiber
C

141. Precision Winding of Cylindrical Composites With Shaped Glass Filaments

Humphrey, R. A.

NASA CR-517, Aug. 1966

DeBell and Richardson

Various shapes of glass fibers have been made and wound directly onto a mandrel by bonding window glass into the desired shapes and drawing it down to fibers. Hexagons, rectangles, hollow hexagons, hollow tubes, and thin flats were made. NOL rings were wound and tested from these fibers. There are some problems getting the maximum packing density during winding.

Shaped filaments were also formed from organic polymers at lower temperatures, E-glass, lead, fused silica, and borosilicate.

Fiber	Matrix
Glass	Resin

142. Optimization of Shell Structures Under Bending and Torsional Loads

Hutter, U.

(Presented at he International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

Technichen Hochschule Stuttgart

Cylindrical thin shells built with composites and subjected to bending, torsion, and internal and external pressure were studied. A movie demonstrating fiber and cylinder response to these forces, without and with hoop stiffeners, was shown. Each failure mode had a specific strength value which can be characterized by an algebraic matrix and described by a 3-dimensional space vector. A number of 2-dimensional sections of such response surfaces were shown, and were similar to those shown by Hedgepath earlier (see entry No. 126). Failures occurred in compression before tension in the system studied. Fibers in the shell axis carry bending with optimum stiffness and strength-to-weight ratios. Crosswise laminated layers support circumferential shear stress, and have some longitudinal stiffness also. Thus, optimal cross layers are less than 90° to the shell axis. General diagrams and equations can be used to find optimum crossing angle and layer cross sections.

Fiber	Matrix
Glass	Resins

143. Survey of Composites Research in Germany

Hutter, U.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

Technichen Hochschule Stuttgart

Summary of composites work and research in Germany is given. A film was shown illustrating various structural uses of composites, particularly glass-reinforced plastic. Windmill rotor blades, 34 m long, were made and are in use. The fibers are attached at the hub by an endless wrapping technique. Other applications are glider bodies and wings, propellers, and rotor blades. Research indicated no effect of glass fiber diameter on UTS of glass-resin composites. The composites are pigmented to control the temperature and, also, to give radar echo where desired. Notch sensitivity is less than that of homogeneous material. Special equipment was shown and described for testing tubes in combined bending and torsion. Refrasil fibers have been wound in resin nozzles with radial fiber orientation. Laminate strengths were tested as a function of wrapping angle, and some results were noted. This year's work was begun on B fiber preparation, together with whisker development work. Another development program was internal strain gauges which can be placed within the body of the composite in various planes and locations to determine internal stress patterns and compare them with surface stresses.

144. Summary of the 10th Meeting of the Refractory Composites Working Group

Hyelin, L. N., James, D. R., Beardslee, E. H.

AFML-TR-65-207, Aug. 1965 (AD-472867)

Battelle Memorial Inst.

General Tech. Corp. made Ni-matrix composites by powder metallurgy, liquid infiltration, and electroforming. The W in Ni and B in Ni gave good strengths. Harvey Eng. Labs improved Al strength with 50-vol% steel wires; Be wires in Al also gave good results. Norton has made short fibers of Al_2O_3, SiO_2, ZrO_2, and SiC, and films and tapes of Be, Ta, W, Re, and TiB_2. Atom. Inter. has grown BeO whiskers. IITRI made Al_2O_3 fibers by extruding a mixture of coloidal alumina and suspension agent. Boeing is making composites from flakes of metals and ceramics, usually mixed oxides and Mo or Ni. Battelle used W in Ni by puenmatic compaction. Good bonding, density, and strength increases were obtained; considerable interdiffusion at 1050°C. CTL div of Studebaker uses SiC flakes and whiskers in epoxy. G.E. uses Al_2O_3 in Ni-Pd alloys and Al by infiltration. Adding Cr, Ti, or Zr increases Ni-Al_2O_2 wetting. They make B_4C by evaporation and condensation and did literature survey on filaments useful over 1200°F.

Fibers	Matrices
Al_2O_3, B, W, glass, steel, BeO, Be, SiC, B_4C	Ni, Ti, Al, Cu, epoxy, Ni-Pd

145. Ship Systems Constraints and Operational Requirements

Jackson, L. L.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

U. S. Department of Navy

The ship and operational requirements for materials are described, both now and in the future. Differences between naval and commercial ships were enumerated. Materials used must be fabricable by acceptable techniques with predictable and reliable results, and must be durable, repairable, inspectable, and cost effective. Some of the goals are: increased surface speed, at least a factor of 2; deep diving submersibles; and improved materials for pressure hulls.

146. The Effect of Fiber Orientation on the Tensile Strength of Fiber-Reinforced Metals

Jackson, P. W., Cratchley, D.

***J. Mech. Phys. Solids*, Vol. 14, pp. 49–64, 1966**

Rolls Royce

Composite strength is a function of three things: fiber strength, matrix shear strength, and matrix failure in plane strain. Overall composite strength as a function of fiber orientation falls below the sum of the curves for these three limiting cases. Experimental data confirmed this, and showed that misalignment of 10% for steel in Al did not lower UTS with 35% fiber (2-mil, hot pressed with Al foil at 500°C for 1 h). The 50% SiO_2 in Al had reduced UTS at 5% misalignment; 40% SiO_2 retained almost all of its strength up to 20% misalignment. The difference between the two was caused by extensive fiber damage in the 50% composite during pressing. Laminated steel-Al composites also retained their strength at up to 20% misorientation. The 50% SiO_2 in Al had a sharp loss in strength at only 1–2% misorientation in laminated samples, again because of fiber breakage at crossovers. The 40% SiO_2-Al had a smooth drop in UTS with misorientation for laminated samples. Appreciable misalignment of fibers is possible without decreasing fiber strength or composite strength greatly.

Fibers	Matrix
SS, SiO_2	Al

147. Behavior of Wire Reinforced Plastic Using the Wire Sheet as Reinforcing Material

Jaray, F. F.

(Preprint of paper presented at 22nd Annual Meeting of the Society of Plastics Industry, Washington, D. C., Jan. 31–Feb. 3, 1967)

This paper describes *wire sheet* which is made by laying 60 strands of 10-mil steel wire on a bonded cloth of acrylic or polyester fibers, using a special epoxy. Laminates with up to eight layers of sheet were made; strength was a function of number of wires, not number of laminates. Wrapped pipes were made from the laminates and tested by hydro-burst and crushing with good results. The laminates had no voids or inclusions. They also resisted cyclic loading without weakening. Boiling in distilled water naturally did not reduce UTS. *Wire sheet* has been formed into simple shapes by molding. Suggested uses are car bodies, pipe, and dished ends of pressure vessels.

Fiber	Matrix
Steel	Epoxy

148. Measurement of Contact Angles in Filaments to Indicate the Effects of Surface Treatments

Jones, W. C., Porter, M. C.

(Paper E-2, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

AVCO Corp.

Paper presents apparatus and techniques for measuring contact angle of liquids on fibers. A microscope is focused on the reflection of a light beam from the surface of the drop, and the contact angle is read directly on a calibrated platform when the reflection is extinguished at the liquid-fiber surface. Accuracy and precision are very high, rapid readings are possible, and the readings are made directly. The present instrument is limited to angles of less than 75°, and requires considerable patience. Contact angles of B in Epon 828 were measured after a number of surface treatments. The most effective in lowering contact angle were acetone and hot ethanol. Wet strength retention also was very high for B cleaned in hot ethanol.

Fiber	Matrix
B	Epoxy

149. Rolls Royce Materials Work Stresses Filament Composites

Judge, J. F.

***Missiles and Rockets*, Mar. 7, 1966**

Rolls Royce produces glass fibers coated with molten Al, and then hot-pressed to form a composite. The fatigue characteristics are much better than plain Al. Varying the thickness of the Al allows variation of fiber spacing.

Fiber	Matrix
Glass	Al

150. NASA-AF Funding AVCO Multidirectional Reinforced Plastics

Judge, J. F.

***Technol. Week*, Oct. 31, 1966**

Avco produces fabric blocks in which the fibers are woven together in three planes to give reinforcement in several directions. Angles need not be 90°. Different fibers can be woven in each direction if desired.

Fibers	Matrix
Glass, B, C	Epoxy

151. Oriented Noncontinuous Fiber Reinforcement for Structural Composites

Kane, J. L., Lindsay, E. M.

AFML-TR-66-247, Aug. 1966

Owens-Corning Fiberglass

Short fibers of glass and B were formed into tape by orienting the fibers using various techniques. Failure was by resin shear, so strengths were low. Air blowing the fibers onto mechanical orienting devices was only partly successful. Fibers tended to hang up in passages and along the walls. Electrostatic alignment gave fibers which clumped up and clung to the walls. As fiber diameter decreased and length increased, clumping tendencies increased under all types of alignment systems (air and liquid). Best liquid dispersions were obtained in acid solutions with PH 3 and a wetting agent. They could not be dispersed in resins. Use of nylon brushes for combing the fibers gave best results. B fibers were chopped into 1/2-in. lengths by a rotating cutter, and were fed through screens onto a tape via a V-groove onto a silicone-coated tape. Vacuum curing of the tape was required to remove all of the MEK. Laminates with up to five layers were made and tested. Adding B powder to the resin had little effect on tape strength. Maximum strength was obtained at about 50% B; tapes with 35–65% B were made and tested. Post-curing the resin at 450°F for 16 h in N_2 improved composite strengths. Variations in resins and fiber treatment did not improve composite strengths (because it was limited by matrix shear). Non-continuous B fiber composites are influenced greatly by fiber content.

Fibers	Matrix
Glass, B	Epoxy

152. Oriented Noncontinuous B Composites

Kane, J. L.

***J. Compos. Mater.*, Vol. 1, No. 1, pp. 42–53, Jan. 1967**

Owens-Corning Fiberglass

A method is described for preparation of continuous tape from oriented, discontinuous B fibers. Both unidirectional and laminated bidirectional tapes were made and tested. Effects of process parameters, such as B-fiber cleaning, post-curing schedule, and additions of B powder to the matrix and hardener to the resin, were studied. None of these improved composite properties; B powder additions and B cleaning seemed to have reduced properties. Up to 62-vol% noncontinuous B fibers were incorporated in resin-matrix composites successfully with 80–100% of the longitudinal stiffness and about 40% of the strength of continuously reinforced resin composites.

Fiber	Matrix
B	Epoxy

153. Tensile Properties of Fiber-Reinforced Metals: Cu/W and Cu/Mo

Kelly, A., Tyson, W. R.

***J. Mech. Phys. Solids*, Vol. 13, pp. 329–350, 1965**

Cambridge Univ.

Composites were made by vacuum casting Cu around the wires, in a graphite mold. At a given aspect ratio, strength was a linear function of % reinforcement. The slope of the strength-volume fracture lines varies with aspect ratio. Composite strength is strongly dependent on the shear strength of the interface. Fiber failure was both by pullout and fracture. As vol% fiber increases, the effect of aspect ratio increases. As test temperature increases, the effect of aspect ratio decreases. Cu/Mo composites had several times Cu strength.

Fibers	Matrix
W, Mo	Cu

154. The Principles of Fiber Reinforcement of Metals

Kelly, A., Davies, G. J.

***Met. Rev.*, Vol. 10, No. 37, pp. 1–77, 1965**

Cambridge Univ.

A general review is given on metal matrix composites and principles of fiber reinforcement. Differences between dispersion hardening and fiber reinforcement are given. Roles of matrix and fiber are noted. Various types of reinforcements are briefly discussed. Much of the material in this section is now outdated. Excellent section on reinforcement principles, including stress/strain behavior, composite strength (including calculation of strengthening to be expected), minimum and critical fiber volume fractions, effects of strain hardening matrix, differences between continuous and discontinuous fiber reinforcement, end effects, critical fiber length, interface strength, nonmetal matrices, orientation effects, mean fiber strength, fracture toughness, creep, and fatigue. A few of the points made include: discontinuous fiber composites will always be weaker than continuously reinforced ones; and when L/d exceeds L_c by 10 times or more, there is little practical difference. Shape of the fiber ends can have a great effect on stresses at the ends, which may exceed stresses along the fiber in some cases. High volume fractions of fiber, or close fiber spacings, may act like dispersion strengthening particles in the matrix, raising composite strength above simple Rule of Mixtures predictions. The effects of differing fiber orientations on composite strength are treated theoretically; since, at the time the paper was written, little published data on off-axis strength had been published. Maximum theoretical fiber volume is given for round and square fibers in 1, 2, and 3 dimensions. Composite fracture seems to occur from a random accumulation of fiber breaks at weak points along the fibers, either at imperfections or at local thin spots. Fracture toughness may be obtained when the matrix or matrix and fiber are ductile, or when the composite fails by delamination. Another method is when the interface or matrix are weak in shear, making transfer lengths long. Fabrication processes and principles are discussed in some detail. Brief comments are included on the preparation of fibers and the fabrication of fibers, both by separate incorporation of the reinforcement and by growing the fibers when the composite is formed. Fabrication methods for separately incorporated fibers include powder metallurgy, liquid infiltration, slip casting, and coating the fibers followed by consolidation. Problem areas include reaction between fiber and matrix which weakens the fibers or alloying with and perhaps embrittling the matrix, maintaining fiber alignment, avoiding fiber contact, possible fiber fracture during secondary fabrication (such as rolling or swaging), and need to control interface reactions and bonding. Grown composites with lamellar rod and dendritic reinforcement are discussed, as are some of the advantages and disadvantages encountered. It may be necessary to design for high strain rather than high strength or stiffness. Joining will be a problem, thus, many composites will be fabricated as a structure in one piece. High cost will limit applications. Enough is understood about composites to permit their use and application in specific cases.

Fibers	Matrices
All	Metals

155. Fiber-Reinforced Metals

Kelly, A.

***Sci. Am.*, Vol. 212, pp. 28–37, Feb. 1965**

Cambridge Univ.

Article gives a general survey of composites, beginning with dislocations and plastic deformation in metals and ceramics, differences between theoretical and actual strengths, and why these values are not obtained. The greater perfection of ceramics and lower mobility of dislocations makes them stronger than metals, but their lower plastic deformation makes them more sensitive to notches or cracks. Combining brittle but strong ceramic fibers in a metal matrix able to bind, protect, and contain them gives best overall strength. Metals are more useful than resins if high temperatures or corrosive environments are encountered, which resins cannot tolerate. Examples of some successful composites are given. Improved toughness and lower notch sensitivity can be obtained by weakly binding the fibers and matrix so that failure will occur parallel to the fibers rather than across them; this is done by applying loads parallel to the fibers with a notch perpendicular to the fibers. This notch insensitivity also may be obtained if the fibers can be made to pull out rather than break. The principles of fiber reinforcement are clear, but problems, such as preventing reaction between fiber and matrix, aligning the fibers, and reducing costs still have to be solved. Controlled eutectics is one way to make and align the whiskers at the same time. Successful use of composites requires changes in conventional engineering designs, and may need allowable strains of 2% or more, rather than the present less than 1/4%.

Fibers	Matrices
Glass, Al_2O_3, steel, SiC, W, Cr, graphite	Resin, Ag, Cu, Co, Al

156. Tensile Properties of Fiber Reinforced Metals — II. Creep of Ag-W

Kelly, A., Tyson, W. R.

***J. Mech. Phys. Solids*, Vol. 14, pp. 177–186, 1966**

Cambridge Univ.

High purity Ag was cast around chopped W wires with aspect ratios of 30 and 60, and creep tested at 400, 500, and 600°C. Creep rates were greatly reduced by additions of discontinuous fibers, as was strain to failure. Creep rate appeared to be governed by creep in shear of the matrix. At elevated temperatures, only the fibers carried appreciable fractions of the load. Critical aspect ratio increased with increasing temperature, and was more than 30 at 600°C. All tests were made with about 40-vol% fibers. Continuous fiber composites failed by shear at the grips, while discontinuous fibers failed in the gauge.

Fiber	Matrix
W	Ag

157. Microfiber Stress-Strain Apparatus

Kelsey, R. H., Krock, R. H.

***Rev. Sci. Instr.*, Vol. 36, No. 7, pp. 1031–1034, July 1965**

P. R. Mallory Co.

Paper describes P. R. Mallory microfiber stress-strain apparatus which uses balanced capacitance to sense displacement without friction error. Direct drawing of stress-strain curves is possible. It can also be used for creep testing with an addition of a furnace.

Fiber
Al_2O_3

158. Some Observations and Results on Tensile Testing Alumina Whiskers

Kelsey, R. H., Krock, R. H.

(Presented at the ASM Metals Congress, Chicago, Ill., Oct. 31–Nov. 3, 1966)

P. R. Mallory Co.

Paper presents test procedures and data for alumina whiskers. Extreme difficulty of obtaining accurate cross-sectional area measurements are noted. They conclude that data obtained from light microscope measurements are unreliable. Tensile data were obtained by assuming modulus of 60×10^6 psi.

Fiber
Al_2O_3

159. Tensile Testing Alumina Whiskers

Kelsey, R. H., Krock, R. H.

***J. Mater.*, Vol. 2, No. 1, pp. 146–159, Mar. 1967**

P. R. Mallory Co.

Article gives brief history of whisker studies, followed by discussion of tensile testing of whiskers. Whiskers have been shown to be very effective reinforcing agents; but very few have been individually tested, less than 150 to date. Results may be biased, since weak whiskers are broken during handling. This may give greater than actual strength indication. Modulus values also are uncertain. Size and orientation effects have been reported, but are disputed. Tensile test apparatus and test procedure are described. The critical measurement is cross sectional area, which is taken before test by measuring maximum and minimum dimensions of the whisker under the microscope. Area determinations using the light microscope are subject to extensive error, as illustrated by variations in UTS for a particular whisker of from 0.825 to 3.92×10^6 psi taking maximum and minimum areas found. Plots of whisker strength as a function of area are given, using an assumed modulus of 60×10^6 psi. Very few whiskers were broken in handling, so the data is not biased by preselection of the whiskers.

Fiber
Al_2O_3

160. Solidification of Eutectic Alloys

Kerr, H. W., Winegard, W. C.

***J. Metals*, pp. 563–569, May 1966**

Univ. of Toronto

Discussion of solidification of eutectic alloys is given, including history, theories of eutectic solidification, and some of the eutectics which have been prepared by controlled solidification.

161. Research on Continuous High-Quality Filaments by the Melt Process

Kimpel, R. F., Moss, R. G.

AFML-TR-66-185, July 1966

Aerojet-General Corp.

The melting point of technical grade B (98.9%) was found to be 3736 ±30°F. High purity B (99.5%) was found to melt at 3755 ±30°F. A thermal arrestment was noted in the 3625–3650°F range. Melt supercooling of 100–200°F occurred often, even with as much as 2.5% C, 20.7% Zr, and 13.7% Ti. The melting point of 99.5% B was unaffected by added Zr and Ti; adding 0.3–0.5% C reduced the melting point 30–50°F; at 3.1–3.3% C the melting point rose 50–75°F. The compatibility of molten B with candidate container materials (BN, TiB_2, ZrB_2, NbB_2, TaB_2, and HfB_2) was studied.

All diborides were attacked by molten B: HfB_2 the least, and TiB_2 and ZrB_2 the most. BN is satisfactory up to at least 4000°F. Metal carbides, oxides, and silicides were considered theoretically unsatisfactory containers. Regression rates of hot pressed and pyrolytic BN were determined in A up to 4200°F. Pyrolytic BN showed less weight and dimensional changes at all temperatures. Pretreatment of hot pressed BN for binder removal from thin-walled parts was studied; neither heating in vacuum at 2800°F nor in 1 atm. A at 3150°F followed by a short dimensional stabilization at 3400°F was satisfactory. Molten B viscosity was determined by mechanical extrusion to be 41–106 cP for 98.9% B at or near the melting point. Pneumatic pressure extrusion measurements gave more consistent and reliable values of 41–50 cP for 98.9% and 99.5% B. Adding 0.5% C doubled the viscosity; melts with 1–2.5% C could not be forced through the orifice (5-mil diameter × 10-mil long) at 105 psi. Adding up to 20.7% Zr and 13.7% Ti had no effect on viscosity. The surface tension of 98.9% B was calculated as 1008 dyn/cm. Short B fibers were formed by gas pressure extrusion from the melt, but it was difficult to form long fibers. Major problems include the instability of the stream, the wide slushy range of B as it freezes, and the orifice erosion which permits the stream to wander and change dimensions.

Fiber
B

162. Fabrication and Properties of Be Wire

Klein, J. G.
(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)
Brush Beryllium

Paper describes the process for making fine Be wire; start with 1/2-in. extruded rod and draw to 1-1/2 mil. They worked with G.E. to improve the process; below 16 mil, drawing is on G.E. equipment by a G.E. proprietary process, at more than 400 ft/min. Unclad Be with 1.3% BeO is dipped in a lubricant before drawing; the lubricant is removed in a salt bath and acid etch. As drawn, UTS is 0.17×10^6 psi, YS is 0.145×10^6 psi, and 1% elongation. Annealing at 1650°F for 1/2 min gives UTS of 0.09×10^6 psi, YS of 0.06×10^6 psi and 2.5% elongation. As drawn, wire can be bent to 50T; annealed wire can be drawn to 10T. Thickness tolerance is ±3% of the diameter.

Fiber
Be

163. Some New Metal and Metal-Ceramic Composites

Korman, S.
NASA SP-5060, 1966
RAI Research Corp.

Summary of NASA-sponsored work on composites, applications of interest to NASA, and properties obtained is given. Composites are defined as a system with two or more distinct, identifiable components which, taken together, extend the useful thermal tolerance of structural components, or which limit thermal energy transfer or otherwise inhibit chemical reactions. (This is a very limited definition, influenced by the idea that aerodynamic heating is the only major materials problem encountered.) Problems involved in aerodynamic heating are discussed in some detail. Dispersion strengthening is described, as are strengthening mechanisms and factors influencing alloy properties. Applications and organizations active in the field are identified. Fiber composites with discontinuous fibers give the same improvement in properties as continuous ones, provided that the fibers are oriented in the direction of loading and do not touch, that there is adequate stress transfer across the interface, and that the fibers are as uniform as possible in strength, size, and shape. Mechanical properties can be predicted, and follow the Rule of Mixtures. Whiskers are described and their preparation and factors affecting their properties are discussed. Typical properties of fibers and whiskers are tabulated. Problems in fabrication are noted, such as fiber fragmentation, misalignment, poor bonding, scatter in fiber properties, joining, thermal expansion mismatch, and fiber-matrix interactions. Other problems include preparation of low cost fibers with reproducible high properties, control of interface properties, and understanding test methods and results. Approximate costs of some fibers and whiskers are given. Use of reinforcements to prevent brittle fracture of ceramics and to improve their resistance to thermal and mechanical shock is touched upon briefly. A bibliography giving sources of fundamental discussion, NASA-sponsored work, and sources of composites is included.

164. Microstructure of Unidirectionally Solidified Al-$CuAl_2$ Eutectic

Kraft, R. W., Albright, D. L.
***Trans. AIME*, Vol. 221, pp. 95–102, Feb. 1961**
United Aircraft

If the balance between solidification rate and thermal gradient at the interface liquid can be adjusted properly, eutectics should be capable of freezing as aligned lamellae rather than eutectic colonies. Experimental procedure used to test this theory is described. Lamellae can be formed under controlled growth conditions, but faults analogous to stacking faults were found and are described in some detail. Another type of solidification defect called banding was noted and discussed. Microstructure is affected by cooling rate, thermal gradient, and contamination. Mechanical effects showed no relation to microstructure, i.e., vibration, variations in water flow in the quench block, etc.

Fiber	Matrix
$CuAl_2$	Al

165. Anomalous Thermal Stability of Al-$CuAl_2$ Eutectic Specimens

Kraft, R. W., Albright, D. L., Ford, J. A.

***Trans. AIME*, Vol. 227, pp. 540–542, Apr. 1963**

United Aircraft

Eutectics were unidirectionally solidified and then heated for up to 600 h at 490°C, which is 60°C below the eutectic temperature. Areas where the lamellae were aligned and regular did not recrystallize, probably because the eutectic is a low-energy structure in this form. Where the lamellae were twisted, or there were eutectic colonies, recrystallization occurred.

Fiber	Matrix
$CuAl_2$	Al

166. Controlled Eutectics

Kraft, R. W.

***Sci. Am.*, Vol. 216, pp. 86–92, Feb. 1967**

Lehigh Univ.

Review of eutectic solidification and controlled eutectics is given. Methods of formation are described briefly, and examples are shown. Applications, such as electronics and structural materials, are discussed; e.g., Fe in FeS or FeSb gives a material with magnetic properties in specific directions; and conductors grown in semiconductors can be made to increase resistance greatly in the presence of a magnetic field. Other applications are as polarization filters, or to make materials with directional thermoelectric or electrical properties. Some work on directional effects of superconductivity has been done also, but with limited results. High strength eutectics have been made also. Much effort is underway both here and overseas on this new field.

Fibers	Matrices
Al, $CuAl_2$, Fe, NiSb, Cr, NbC, Mg	FeS, InSb, FeSb, Cu, Nb

167. Services and Materials Necessary to Develop a Process to Produce Fibrous Reinforced Metal Composite Materials

Kreider, K. G., et al.

ASD-IR-8-370(II), Feb. 1966

United Aircraft

Plasma sprayed Al and Ti on B fibers wrapped over a mandril. Most tensile specimens failed in the grip regions. Obtained about 70% of theoretical strengthening in Al composites. Ti appeared to cause the B to fail, probably due to thermal shock. Hot pressing as-sprayed composites increased densities and decreased porosity. It also appeared to increase strength slightly. At 500°C, strengths of 73,000–78,000 psi were obtained in Al matrix composites. Low shear strength of the matrix was blamed for low composite strengths. Moduli of 33×10^6 were obtained. Preheating substrate gave better bonding to Al and B. Strong bonds were obtained at all temperatures for Ti.

Fiber	Matrices
B	Al, Ti

168. Boron Fiber Metal Matrix Composites by Plasma Spraying

Kreider, K. G., Leverant, G. R.

AFML-TR-66-219, July 1966

United Aircraft

Boron reinforced composites were made by plasma spraying Al and Ti over B wrapped on a mandrel. Spraying was done under inert gas blanket, with added H_2, and in an inert gas chamber. Tensile, creep fatigue, stress-rupture, and notch sensitivity were measured. Post-spraying sintering and hot pressing did not improve density much, but cold rolling did improve density. B-Al composite strengths were less than those predicted by Rule of Mixtures, because of the scatter in B strength. When this scatter plus the ratio of gauge length to critical stress transfer length is considered, results agree well with predictions. Modulus values agree with Rule of Mixtures predictions. The B-Al composite with 45% B had good strength retention at 500°C. No B degradation was noted as a result of plasma spraying. B-Al composites showed no notch sensitivity with K_t of 2.9; they had little transverse strength, due to matrix weakness. Bidirectionally reinforced samples had no reinforcement at 45° to the load, but they had some reinforcement parallel to the fibers. Creep behavior showed rapid first stage, very small strain in second stage, and almost no third stage which had sudden, catastrophic failure. Stress-rupture life is a function of fiber content. Low cycle fatigue curves were relatively flat; endurance limit is mostly affected by matrix properties. No stress corrosion was observed at 60,000 psi and 17 days. Both 2024 and pure Al were used. Ti spraying caused severe B degradation. Coarse Ti powder caused the B to break, and fine powders gave 50% loss of strength. Preheating of the B was necessary, and oxidation of both Ti and B became a problem. Coating with 0.2 mil of Ni decreased the thermal shock of spraying Ti and gave usable composites, but hot pressing at 450°C or sintering at 450–500°C caused degradation of the B (sintering B-Al for 3 h at 500°C had the same effect). Modulus of B-Ti composites was as predicted. Stainless-Ti composites also were prepared. Strength at 500°C was higher than at room temperature. All Ti composites had poor strength and brittle matrix behavior.

Fibers	Matrices
B, SS	Al, Ti

169. Boron Aluminum Composite Fabricated by Plasma Spraying

Kreider, K. G., Leverant, G. R.

(Paper F-1, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

United Aircraft

Plasma spraying of composites is advantageous because relatively complex shapes can be formed, flexibility of fiber loading, distribution, and orientation can be used, and diffusion reactions between fiber and matrix are minimized. Shapes of revolution can be wound on mandrels, or flexible mandrel can be wrapped and slightly distorted, to get more complex shapes. Process variables which must be controlled include gas flow and type, arc enthalpy, powder size distribution and flow rate, substrate temperature, and atmosphere around the deposit. Sample preparation and testing are described briefly. Hot pressing the composite after spraying improved properties; there was 12–15% porosity as-sprayed. Spraying conditions which gave the best matrix properties did not give as good properties when B was present. Spraying conditions for Al in the presence of B are different from those for Al alone. Tensile strength was measured at room temperature and 500°C. Fatigue endurance limits were determined, and were found to be better than Al alloys. Creep and stress rupture strengths were significantly better than unreinforced matrix. Strength of extracted fibers was essentially unaffected by plasma spraying.

Fiber	Matrix
B	Al

170. Some Comparisons Between Fiber-Reinforced and Continuous Skeleton W-Cu Composite Materials

Krock, R. H.

***J. Mater.*, Vol. 1, No. 2, pp. 278–292, June 1966**

P. R. Mallory Co.

Reinforcement of continuous-skeleton W infiltrated with Cu and Cu reinforced with W fibers and particles was compared. At all levels of reinforcement, fiber reinforcement was superior in increasing strength and modulus. Increases using fibers followed a Rule of Mixtures curve, while W-skeleton data fell below the line. Yield strengths of both fiber-reinforced and continuous skeleton samples increased with increasing composition of reinforcement. The superiority of fiber-reinforced samples was attributed to higher strength of the fibers. It was suggested that solid solution or other hardening of the W skeleton would increase strengths to greater levels.

Fiber	Matrix
W	Cu

171. Whisker Strengthened Materials

Krock, R. H.

***Int. Sci. Tech.*, pp. 38–48, Nov. 1966**

P. R. Mallory Co.

Review of progress and problems in whisker and fiber strengthened composites is given. Advantages of reinforcement, and desired fiber and matrix properties are described. Initial additions of reinforcement weakens composite, as there are not enough to help, but they act as notches. As reinforcement strength increases, critical volume fraction decreases. Lack of good bonding to the matrix, and misalignment of the fibers reduces the efficiency of whiskers; random additions of whiskers means that only about 1/3 are carrying the loads in any one direction. Adding more whiskers will make it harder to get good densities, since whiskers do not compact well and liquid infiltration may not fill the spaces either, while attacking and weakening the fibers. If the matrix work-hardens rapidly, more fibers must be added to get a given strengthening, and as fiber length decreases, the volume of fibers needed increases. It is hard to get whisker composites with good alignment and properties without careful hand layup; production processes usually give much poorer properties. Most promising system for early utilization of whiskers is thought to be unidirectionally solidified eutectics.

172. Mechanical Test for Anisotropy; Failure of Alumenoborosilicate Glass Fibers Under Combined Loadings of Tension and Torsion

Kroenke, W. J.

***J. Am. Chem. Soc.*, Vol. 49, pp. 508–513, Sept. 1966**

B. F. Goodrich/Braecksville, Ohio

The strength of E-glass fibers in tension and torsion was determined to be the same. This indicates that E-glass is isotropic, and has no structure or preferred orientation. Failure follows the maximum stress theory, and was in conformance with the Weibull statistical theory of failure. Surface flaw density on the fibers was low enough for the theory to predict tensile and torsional strengths, and that they would be equal. Anisotropy can be found by simple mechanical tests.

Fiber
E-glass

173. Feasibility of Continuous Forming of Boron Carbide Monofilaments

Kuhn, W. E., Woodworth, V. C.

NASA CR-60498, Jan. 1965

Spindletop Research Center

Report explains the attempt to form B_4C fibers by drawing C filament through molten pool on water-cooled Cu. Partial coatings were dependent mainly on dwell time of C in melt.

Fiber
B_4C

174. Exploratory Investigation of Novel Filament Formation Techniques for Continuous Crystalline Alumina

LaBelle, H., Jr., Mlavsky, A. I.

AFML-TR-66-246, Aug. 1966

Tyco Labs

Dendrites of Al_2O_3 were grown from the melt by use of a floating Mo orifice. Crucibles were of Mo with W support rods. Both W and Al_2O_3 seeds were used successfully. It is suggested that the dendrite growth was down into the melt, but no good evidence of this was presented. A theoretical analysis was made which suggested that the process was rate limited by heat rejection from the dendrite; no mention of concentration gradients, surface energies, or convection losses in the melt was made. The analysis seems oversimplified. Dendrites, 5–20 mil in diameter $\times$ 6 in. long, were grown at rates of 30 ft/h. E was found to be 30–50 $\times$ 10^6 psi by the vibrating reed method. UTS was 0.125 $\times$ 10^6 psi and flexure modulus was 25 $\times$ 10^6; both were tested in bending. Four types of dendrites were grown, all parallel to the c-axis. Crystallographic data are given.

Fiber
Al_2O_3

175. B Filament for Structural Composites

Lasday, A. H., Talley, C. P.

(Paper D-1, presented at the 10th SAMPE Sypmosium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Texaco Experiment

Paper presents the history and description of boron formation process by chemical vapor deposition. A 1/2-mil W fiber is passed through Hg seals into a H_2 chamber where the surface is reduced, and then into a plating chamber with H_2 and BCl_3, which is recovered and recycled. The wire is resistance heated through the Hg seal. Important process variables include gas flow rate and concentration, byproduct concentration, and substrate temperature. Average and standard deviation of tensile properties for different process conditions are given. Present average is 460 $\pm$92 psi. Average modulus is 60 $\times$ 10^6 psi. Torsional modulus is 24–26 $\times$ 10^6 psi. Hardness is 4000 kg/mm^2 Vickers. Thermal expansion is 2.8 $\times$ 10^{-6} in/in/°F. At 600°F in air for extended times, strength drops. In argon there is little change at 900 h and 600°F. Also, a thin Si coating protects the B from degradation.Recently it became unnecessary to etch the B, but earlier fiber was improved by a hot HNO_3 etch. Heating to 1500°F in N_2 to form a nitride skin also was used earlier, but treatment is not considered necessary now. Methanol rinses also have been effective at times in improving strength or wetting to the epoxy matrix. No known surface treatment for B is highly reliable and effective. The standard tape is 1/8 in. wide, 30 filaments in a prepregnated resin. Also, tape has been wrapped on a drum, then cut off and laid up. Cloth may be woven by using glass in the warp and B in the fill. Composite processing is described with typical properties given. Projected cost is $500/lb at 100,000 lb/year production. Ultimate costs as low as $100/lb are projected.

Fiber	Matrix
B	Epoxy

176. Selected Activities of the Douglas Aircraft Co. in the Development of Refractory Composites

Leggett, H.

In *Summary of the 8th Meeting of the Refractory Composites Working Group*, ML-TDR-64-233, Vol. II, Jan. 1964 (AD-470694)

Douglas Aircraft

Vapor deposited W on W wires was wound onto a mandril, using WF_6 at reduced pressure. Grain size and orientation depended on deposition rates. No physical test data obtained. Others have done same type of work previously.

Fiber	Matrix
W	W

177. Preliminary Investigation of the Mechanical Behavior of 3-Dimensionally Reinforced Plastics

Lenoe, E. M., et al.

(Paper A-2, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

AVCO Corp.

A technique has been developed for weaving 3-dimensional fabrics in cylindrical form (by braiding), tufted fibers, felts, and multiple warps. Preliminary tests indicated that isotropic properties are approached. The loom design is being improved.

Fibers	Matrices
C, glass	Epoxy, phenolic

178. Mechanical and Structural Properties of a 3-Dimensionally Reinforced Plastic

Lenoe, E. M., et al.

Paper No. 67-171, *AIAA 5th Aerospace Sciences Meeting*, Jan. 23–26, 1967

Avco Corp.

Composites were formed by weaving fibers in 3-dimensions on a specially designed loom, which is not discussed; fabrication also is glossed over. Most of the report covers fiber test methods, analysis of shell structures, and comparison of test and theoretical data. Cylinders and blocks were formed by the process, but tests were made on rings and cylinders. Tensile strength and modulus were less dependent on orientation than glass cloth laminates; shear modulus was more orientation dependent. Glass composites contained 2000 threads/in.^{2} horizontally and 2400 vertically; C composites had 100 threads/in.^{2} horizontally and 1400 vertically.

Fibers	Matrices
B, graphite, S-glass, silica	Phenolic, epoxy

179. Recent Advances in Alumina Whisker Technology

Levitt, A. P.

***Mater. Res. Stand.*, Vol. 6, No. 2, pp. 64–71, Feb. 1966**

Army Materials Research Agency

Whiskers have been grown by neucleation on fine particles and substrates in a stream of mixed gases. Tapered whiskers may be grown with maximum D of 20 μm. Reaction rates are very high. Growth is from a mixture of $AlCl_3$, H_2, and CO_2 at 1200°C and low supersaturations. Costs are much lower than otherwise.

Fibers
Al_2O_3, SiO_2, SiN

180. Whisker-Metal Matrix Bonding

Levitt, A. P., Brown, J.

***Chem. Eng. Progr.*, Vol. 62, No. 3, pp. 74–78, Mar. 1966**

Army Materials Research Agency

The effects of adding 0.10% of various additives to Al on shear strength and contact angle were studied. Cu, Mg, Zr, and 7075 Al alloy decreased the contact angle; Pb, V, Mn, Cr, Bi, and 2024 alloy had no effect; and Se, Te, Ge, and 1100 alloy increased contact angle. In general, Group I and II elements decreased contact angles, and Group IV and VI elements had no effect nor increased it. Only Se, 7075 alloy, and 1100 alloy had appreciable higher shear strengths. Zr, V, Mn, Cu, 2024 alloy, and Cr had lower shear strengths.

Fiber	Matrix
Al_2O_3	Al

181. Whisker-Strengthened Metals

Levitt, A. P.

(ASME Paper 66-MD-81, presented at ASME Design Engineering Conference and Show, Chicago, Ill., May 9–12, 1966)

Army Materials Research Agency

Summary of whisker strengthening in metals is given. Whisker strength increases as diameter decreases. Principles of reinforcement are covered, including the effect of critical whisker length. Al_2O_3 whiskers with $L/d = 300$ gave higher strengths in Ag than those with $L/d = 100$, up to the melting point of Ag. Derivations of L_c, fiber stress, and composite strength are given. Importance of interfacial bonding is noted, and examples are given. Effect of additions which improve bonding are noted. Stresses induced by thermal expansion mismatch between fiber and matrix are plotted for different systems. A critical volume fraction of fiber is needed to strengthen the matrix; if less than V_{cr} is present, the matrix is weakened. V_{cr} can be reduced by using the strongest possible fibers. Criteria for successful whisker reinforcement are given: $V_f > V_{cr}$. Fiber strength and modulus must be more than the matrix; fiber and matrix must bond and give good stress transfer over a significantly long length; thermal coefficient expansions should match; fiber and matrix should be chemically compatible; fibers should have tapered or round ends to reduce end effects; fibers must be stable, and retain strength at high temperatures, and fibers must be properly oriented. (Also, $L/d > L_c$.) Properties of some whisker-reinforced metals are given. For randomly oriented fibers, strengthening is 1/3 that of oriented fibers. Unidirectional solidification of eutectics is covered extensively. Advantages, disadvantages, and progress are covered. Advantages are good fiber distribution, orientation and bonding, tapered whiskers are formed, and chemical compatibility is built in. Disadvantages are limited to eutectic composition, and whiskers can only be formed when % fibers is low. Whisker availability has improved, but they are still expensive. Major problems include whisker growth, handling, grading, incorporating and aligning them in the matrix, and joining and fabricating them. Interdisciplinary approach is needed to best use composites.

Fibers	Matrices
Al_2O_3, B_4C, W, Al_3Ni, Si_3N_4, NiBe, TiB, Ni, C, $CuAl_2$	Al, Ag, Cu, Ni-Pd, Fe, Ni, Ta, Ti, Ni_3B, Mg

182. Whisker-Strengthened Metals

Levitt, A. P.

***Mech. Eng.*, Vol. 89, pp. 36–42, Jan. 1967**

Army Materials Research Agency

This survey covers whisker reinforcement, including mechanics of strengthening. It cites strength of whiskers, desirable characteristics, such as high strength and strength retention at high temperatures, high modulus, low density, high melting point, thermal stability, and chemical stability. Critical fiber length is longer when the matrix deforms both elastically and plastically rather than when it only deforms elastically. The critical volume fraction of fiber is dependent on fiber strength. Whiskers should have tapered ends which have a strong and resiliant bond to the matrix; the matrix should wet the fiber and form a good bond. Strength and elastic modulus of the fiber should be higher than the matrix, and fiber concentration and length should be more than the critical amount and length. Thermal expansion coefficients of fiber and matrix should match closely to reduce bond failure due to heating. Chemical reaction between fiber and matrix should be minimized. Fibers should be oriented and distributed for maximum load transfer. Advantages of unidirectionally solidified composites are noted. Whisker costs are high. Barriers to applications are large scale growth; handling grading; aligning, distributing, and incorporating whiskers in metal matrices; and joining and fabricating the composites.

183. Tensile Properties of High Modulus Fibers

Littleton, H. E., Pears, C. D.

***Res./Dev.*, pp. 24–28, June 1966**

Southern Research Inst.

A tensile tester for determining strength and modulus of filaments and whiskers is described, and some of the test results are given. Temperatures up to 4000°F are possible. Specimens are pulled horizontally, elongations are measured with an optical extensometer, and loads are measured with a load cell. Area measurements on the fibers are the main source of error.

Fibers
W, B, 304 SS, Au plated Mo

184. Thermo-Oxidative Stability of Polyphenylene Resins in Asbestos Reinforced Laminates

Long, F., Milward, B. B., Roberts, R. J.

(Paper A-1, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Monsanto, U. K.

An improved polymer which retains good flexural strength and low weight loss at 300–400°C has been developed using new processing methods to form the linkages. Also, 6-layer laminates of asbestos and polymer were prepared and tested; high temperature strength and stability were better than previously tested materials.

Fiber	Matrix
Asbestos	Polyphenylene

185. The National Composites and Fibers Program

Lovlace, A. M.

(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)

AFML

General discussion is given on composites and fibers. Metals have more potential than organics due to higher temperature stability, perhaps better joining characteristics than resins. The filament-matrix interaction presents real problems, as in B-Ti. Some areas under study are listed, including micromechanics, tests and test techniques, fiber preparation, etc. The critical aspect is perfection of manufacturing technology. Both metal and organic matrices have problems, particularly metals. It is hard to maintain quality control and to get big pieces. Combinations of university and industrial groups are working in same area, i.e., Monsanto-Washington Univ. are investigating discontinuous fiber composites, and Case-Bell-Union Carbide is looking at C filament composites. A big improvement in C filaments is expected by the end of the year, up to 40×10^6 modulus and 0.3×10^6 psi UTS. Composites like B-epoxy are real engineering materials now, but not necessarily the ultimate. Composites offer designers great design flexibility. Designers are not now fully satisfied with the composites and metal matrix programs. B-Al and 50% C-Ni show promise right now, but others may be better.

Fibers	Matrices
B, C	Resins, Al, Ni, Ti

186. Fiber-Reinforced Structures from Powder Compacts

Lund, J. A., Howard, G. C.

***Int. J. Powder Met.*, Vol. 2, No. 2, pp. 29–31, July 1966**

Univ. of British Columbia

Prepared composite of Fe and Cu by infiltrating presintered Fe compacts with Cu at 1110°C, and then cold swaged followed by 7-stage wire drawing to 0.035 in. Fe-rich fibers, 10μm in diameter, in a Cu-rich matrix were formed and tested. It is suggested that blending and sintering powder followed by swaging or drawing may also be practical.

Fiber	Matrix
Fe	Cu

187. Effect of Fiber Geometry on Stress in Fiber-Reinforced Composite Materials—Phase 2

MacLaughlin, T. F.

AD-633984, Apr. 1966

Watervliet Arsenal

Models of various fiber end configurations and fiber spacings were studied by photoelastic techniques to determine the stress patterns at fiber ends. When the gap between fibers was small (at the ends) the stress in the matrix was maximum. Photos and plots of stress as a function of end geometry and distance from fiber ends are given.

188. Effect of Fiber Geometry on Stress in Fiber-Reinforced Composite Materials

MacLaughlin, T. F.

***Exp. Mech.*, Vol. 6, pp. 481–492, Oct. 1966**

Watervliet Arsenal

Photoelastic stress analysis of fiber-reinforced plastics is given. Also, this article has been reported and previously abstracted as AD-633984 (entry No. 187).

Fiber	Matrix
Steel	Epoxy

189. Gas-Plated Fibrous Composites

MacNeil, C. E.

In *Summary of the 8th Meeting of the Refractory Composites Working Group*, ML-TDR-64-233, Vol. II, Jan. 1964 (AD-470694)

Goodyear Aerospace

René 41 cloth was joined by chemical vapor deposition of Ni from $Ni(CO)_4$ and W from WF_6. Spot *welds* were made by passing the gases through hollow electroldes at each joint. Sound bonds with up to 40% more strength than equivalent spot welds were obtained.

Fiber	Matrices
Rene 41	W, Ni

190. Composites Development

Marshall Space Flight Center

Report to NASA Research Advisory Committee, Apr. 1967

AZ31 Mg alloy was diffusion bonded with 4-mil NS355 wire at 700°F for 5 h at 10,000 psi in vacuum. Reinforcements of up to 11.7% gave improved tensile strength. Also, they are making modular filament reinforced sheets which can be stacked to give reinforcement as needed. No test data are available on this material yet.

Fibers	Matrices
Be, steel	Mg, Al

191. Fracture Volume Changes in Laminated Orthotropic Materials

Mast, P. W.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

U. S. Naval Research Lab

A test setup is described for determining crack behavior and fracture in plywood and similar laminated materials by use of a digital and analog computer to control, record, and display the data, as well as store the data for later use. The effects of angle of layup, notch depth, load angle, and load eccentricity were noted for plywood. Volume change at the first stage is crack arresting, and at others is crack promoting. Crack propagation velocity is a critical parameter.

192. Composite Alloy With High Temperature Oxidation and Erosion Resistance

Masterson, J. F.

ASD-TDR-63-297, Apr. 1963 (AD-408957)

Bendix Corp.

They blended 6% MgO in Cr powder, and formed test samples by rolling and extruding in Ni cladding. Rolling was in steel picture frame. It was possible to roll, forge, and extrude the composite with reasonable physical properties and adequate corrosion and erosion behavior obtained. Brittle behavior was noted in rolled and stress-relieved sheets.

Fiber	Matrix
MgO powder	Cr

193. Silicon Carbide Filament Reinforced Epoxy Resin Materials

Materne, H. P., Jr., Kuhbander, R. J.

(Paper A-4, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

AFML, Univ. of Dayton

Composites prepared from SiC-epoxy were tested as unidirectional monolayer tape and bidirectional panels. Continuous fibers of SiC on W were characterized and used as the reinforcement. In spite of data scatter, excellent results were obtained with better than 100% reinforcement efficiency in many cases. The effects of surface treatment of fibers were studied; thermal oxidation plus silane gave most strength increase, and solvent cleaning reduced the strengths. The thermal and oxidation resistance of SiC is greater than B, and makes SiC composites attractive, in spite of the increased density.

Fiber	Matrix
SiC	Epoxy

194. Silicon Carbide Filament Reinforced Resin Composites

Materne, H. P., Jr., Kuhbander, R. J.

AFML-TR-66-383, May 1967

AFML, Univ. of Dayton

Unidirectional and bidirectional laminated samples were prepared from 4-mil SiC fibers preimpregnated with resin. The SiC was characterized by X-ray diffraction, metallography, and mechanical test. Since each batch varied in properties, each sample was made from a single SiC spool. Composite preparation is described. Fibers were surface treated by thermal oxidation or pyrolysis. Shear strength and modulus, flexure strength, and tensile strength and modulus were determined. Properties were about comparable to B-resin composites. Surface treatments of the fibers improved strength in both 0 and 90° tests on bidirectional laminates. Shear modulus was unaffected by fiber treatment, and depended only on % fiber.

Fiber	Matrix
SiC	Epoxy

195. Composites With High-Temp Capabilities Are Being Sought

Mathews, D.

***So. Calif. Ind. News*, Vol. 19, No. 46, p. 13, June 26, 1967**

Report of address by C. T. Lynch at Los Angeles Chapter meeting of ASM is given. Ceramics reinforced by metals have limited potential because of differences in coefficient of thermal expansion, which causes cracking. Compatibility between fiber and matrix is a major problem in metal-matrix composites. Coatings and diffusion reactions need much more study. Some of the systems currently being studied are noted, and problem areas such as cost, reliability, fabricability, and reproducibility are discussed.

196. Structural Applications for Advanced Composite Materials

Mayer, N. J.

N66-33172, July 1965

NASA, Washington

Problems and approaches in utilization of composites in spacecraft are discussed. Usefulness of the analytical approach to prediction of composite properties is mentioned. Possible reinforcement forms are listed. Without specifying the application, it is only possible to demonstrate that various composite systems provide a measure of reinforcement, as predicted analytically. Beyond this, functional and environmental factors must be specified; a team approach to design should be used, with both end uses and material properties considered.

197. High Modulus-to-Density Fiber Reinforcements for Structural Composites

McCandless, L. C., et al.

AFML-TR-65-265, Part II, Sept. 1966

General Technology Corp.

Various filaments were prepared by vapor deposition, usually on a 1-mil W substrate. SiC and B_4C have been produced in continuous form with good properties, particularly SiC. Initial work was in a batch apparatus, in which feasibility and some process parameters were studied, followed by scale-up into a continuous system. Details of the deposition systems used are given. Best results were obtained when the spent gases were vented to an exit line rather than to an adjacent deposition chamber, and a high H_2 to plating gas ratio was used. Use of N_2 rather than A as a carrier gas may improve UTS. Optimum deposition temperature is 1150–1200°C. Other important factors are linear filament speed, chamber design, purity and ratio of plating compounds, and electrode design. Lower deposition temperatures favored amorphous deposits. There was little change in UTS at temperatures up to 1250°C. Several reactants and reactant combinations were used to prepare TiB_2 filaments, but there was great difficulty in all cases with temperature measurement and control. Modulus was about 70×10^6, but UTS was low, usually under 0.1×10^6 psi. Mo rather than W substrates gave much higher UTS and about 10% higher moduli. Several methods were used to deposit B_4C; properties varied with the source gases, and best results occurred with ethane and BCl_3. UTS decreased as plating temperature increased over 1100°C. AlB fibers were made by deposition from $AlCl_3$. Neither adequate method of temperature monitoring or control nor metering gas flow was found. Preliminary UTS values are rather low. $TiSi_2$ filaments had low UTS and moduli; it was very hard to keep uniform filament temperatures during deposition. No further work on this system is suggested. B-Si was prepared by several methods, but none showed any evidence of compound formation. There was wide scatter in the results, and none of the UTS values exceeded 0.1×10^6 psi. Doping SiC with B gave lower UTS but slightly higher moduli. Higher C ratios gave higher modulus; higher Si gave lower modulus. Attempts to form BeO fibers by oxidization of 5-mil Be wires were unsuccessful. Pyrolysis of C on Be wires also was unsuccessful. Formation of BeB both by codeposition and deposition of B on a Be wire was unsuccessful. UTS and moduli were poor. Composites of SiC-epoxy were prepared and tested in 3-point bending to provide a comparison of shear strengths with other composites. Shear strengths were comparable, but tensile values did not follow Rule of Mixtures and were less than predicted.

Fibers	Matrix
SiC, TiB_2, BeB, AlB_2, B_4C, BeB_2, Be_2C, B-Si, $TiSi_2$, SiC:B, BeO	Epoxy

198. Survey of the State of the Art of Ceramic and Graphite Fibers

McCreight, L. R., Rauch, H. W., Sr., Sutton, W. H.

AFML-TR-65-105, May 1965 (AD-464318)

General Electric Space Sciences Lab

This survey is the one mentioned in AD-615662 report. This survey contains sections on applications, factors affecting strength, results of visits and questionnaires, evaluation and discussion, conclusions, patents, and bibliography. There is too much material for reasonable abstraction.

Fibers	Matrices
All	All

199. Stress-Strain Behavior of Tungsten-Fiber-Reinforced Copper Composites

McDanels, D. L., Jech, R. W., Weeton, J. W.
NASA TN D-1881, Oct. 1963
Lewis Research Center

Continuous and discontinuous W fibers were formed into composites by Cu infiltration. Fabrication and test methods are described. Four stages of composite failure are identified: elastic deformation of fiber and matrix; elastic deformation of fiber, plastic deformation of matrix; plastic deformation of fiber and matrix; and failure of both fiber and matrix. Cyclic loading composites causes a small amount of permanent set due to differences in the deformation behavior of the components. Primary and secondary elastic moduli may be identified in stage II; the secondary modulus may be a better design value than the primary one. Both strength and modulus follow a Rule of Mixtures curve. Yield strength is hard to determine, since two values will be found, one for each slope. The differences in slope, and yield strength, decreases for increasing fiber content. The secondary modulus line may be more reasonable for yield point determination, since it has a longer slope and is more easily measured. Failure is by random fiber fracture until a local area accumulates fractures, and no longer can support the load. Delayed fracture of the composite occurs when the matrix continues to strain after all the fibers have broken, provided that there is good fiber-matrix bonding. Both continuous and discontinuous fibers followed Rule of Mixtures behavior, although the reinforcement mechanisms differed. In discontinuous composites, good bonding is required during all four deformation stages, not just stage IV, as for continuous fibers. A critical fiber length-to-diameter ratio must be exceeded in order for the fiber to break rather than pull out. Factors affecting composite properties include: bonding and aspect ratio, especially for discontinuous reinforcement; interfiber spacing; properties of fiber and matrix, as they affect critical aspect ratio; scatter of fiber strength; fiber orientation; and fiber content. Composite elongation was greater than that of individual fibers. Discontinuous fibers with L/d of 75 or more were as efficient in reinforcing Cu as continuous fibers. Generalizations from this work may or may not be applicable to other systems. Metal reinforced composites have more uniform and reproducible properties than glass-reinforced composites, due to the lower scatter of fiber properties.

Fiber	Matrix
W	Cu

200. Stress-Rupture Properties of W Wire From 1200–2500°F

McDanels, D. L., Signorelli, R. A.
NASA TN D-3467, July 1966
Lewis Research Center

They determined the stress-rupture properties of 5-mil W wire from 1200 to 2500°F, and compared the data with that for other forms of W, Mo, TZM, and Ni-base superalloys, in both cast and wrought form. At 2000°F, there was a transition from ductile to brittle fracture which was associated with recrystallization of the wire. This caused a slight decrease in stress-rupture life. Above 3000°F there is no difference between the tensile strengths of wire, rod or W sheet. W wire had higher 100 h stress levels than Mo, TZM, or the Ni-base alloys, particularly above 2000°F.

Fiber
W

201. Electrical Resistivity and Conductivity of W-Fiber-Reinforced Cu Composites

McDaniels, D. L.
NASA TN D-3590, Aug. 1966
Lewis Research Center

Electrical conductivity and resistivity of vacuum-infiltrated Cu-W composites were determined. Resistivity followed a hyperbolic function of W content, while conductivity followed a linear function of W content. Such materials may be useful for high strength conductors of controlled resistivity or conductivity. Electrical properties may be useful as a means of determining fiber content in W-Cu composites.

Fiber	Matrix
W	Cu

202. Measurement of the Strength of Whiskers and Their Role in Reinforcing Experimental Composite

Mehan, R. L., Sutton, W. H., Herzog, J. A.

GE Reprint No. 349, July 1965 (AD-465992)

General Electric Space Sciences Lab

Principles of fiber reinforcement are discussed, and equations governing fiber and composite properties are given. Measurement of mechanical properties of fibers is described; the importance of determining both the strength and variation of strength of whiskers is emphasized. Hand selection of whiskers for testing tends to preselect only the stronger ones, and biases test results. Composite properties will depend on overall whisker strengths and lengths. Ceramic whiskers seem to have more potential than metal ones, because they are less reactive and resist dislocation movement at much higher temperatures than metals; they do present the problem of bonding to the matrix, which usually requires precoating. Experimental determination of whisker strengths is hard, because of the irregular and hard-to-measure cross-sections, particularly for small whiskers. Also, it is hard to separate data scatter caused by surface imperfections and experimental difficulties. Large amount of scatter is in data when size decreases; chance of defects is approximately proportional to length. Bend tests are usually inaccurate because loads are low, thus small errors become significant, and it is hard to position the load points accurately; irregular cross-section makes it impossible to determine the proper moment of inertia of the beam. As scatter in whisker strength increases, anticipated composite strength will decrease for bundles of fibers, since weak ones will break at low stresses and overload the stronger ones. Modulus is not affected much by scatter in whisker properties, since most whiskers have similar moduli for particular materials, while strength is affected by surface defects.

Fibers
Al_2O_3, Fe, SiC, B_4C, Cu, Ni, Co

203. Evaluation of Sapphire Wool and Its Incorporation into Composites of High Strength

Mehan, R. L., Feingold, E., Gatti, A.

GE Reprint No. 356, Aug. 1965 (Covered also in AD-468533 and AD-469986)

General Electric Space Sciences Lab

Sapphire wool is a shorter and finer version of sapphire whiskers. Preparation of the wool is discussed; testing, and problems encountered are covered in some detail. Separate appendixes are included on effect of whisker length and apparent modulus, on modulus determination and replication techniques, and on the effect of varying cross-section on modulus. Examination of whiskers by X-ray diffraction and electron microscope is described; they were found to contain stacking faults, dislocations, overgrowths, etc. Liquid infiltration of wool coated with Ni over Ti gave erratic results, and low densities. When Ti was omitted, wetting was poor. Use of Pt and Ni coated wool and powder metallurgy techniques gave little increase in strength, mainly due to the low volume fraction of whiskers. Some practical problems involved in use of sapphire wool and the fabrication methods are discussed. Al_2O_3 whiskers were found to have a tensile strength of 1.6×10^6 psi, $E = 60\text{–}70 \times 10^6$ psi.

Fiber	Matrix
Al_2O_3	Al

204. Metal Fiber Reinforced Ceramic Composites

Miller, D. G., Singleton, R. H., Wallace, A. V.

***Bull. Am. Ceram. Soc.*, Vol. 45, No. 5, pp. 513–517, May 1966**

Allison Div./General Motors

The 50-μm wires of W and Mo were chopped to 1500 μm and coated with 2 to 3 μm of disilicide by pack diffusion, and vacuum hot pressed in Mullite or Zirconia to more than 98% density. Flexural modulus of rupture was increased 90% with 20% Mo or W at room temperature. Coated wires were less effective than uncoated ones. Thermal shock resistance was markedly improved using both fibers. Oxidation at 1200°C for 30 min caused complete failure of W-reinforced composites, and loss in strength of the Mo-reinforced composites. The higher than anticipated increase in modulus of rupture was attributed to micro-cracking of the matrix, although none was found by electron microscope examination.

Fibers	Matrices
W, Mo	Mullite, Zirconia

205. Investigation of Fiber Systems of Ablative Materials

Miller, G. H., Robinson, G. C., Jr.

NASA CR-54722, Sept. 30, 1965

Texaco Experiment

Various fibers were prepared as candidates for reinforcement in ablative rocket nozzle liners in H_2/F_2 or B_2H_6/OF_2 environments. Fibers were made in static chambers by deposition from a number of chemicals at various temperatures and times. Substrates included W, Ta, and Zr wires. Preparation of TiC by deposition from $TiCl_4$ and CCl_4, acetylene, C_4H_{10}, C_5H_{12}, and C_2H_5Br were unsuccessful. Depositing B from BCl_3 on 9-mil Zr wire resulted in hollow ZrB_2 fibers which were very weak. Codeposition of B and Zr was successful, but stoichiometric ratios could not be obtained. Deposition of B and conversion to ZrB_2 gave a weak, poorly adherent deposit. Graphite was deposited on W from acetylene. Adding B to the deposited layer from BCl_3 improved strengths. W was deposited on B from WCl_6, WF_6, and $W(CO)_6$. Best results were with WF_6. TiB_2 was deposited from $TiCl_4$ and BCl_3 successfully. Best fiber properties were those of the TiB_2 and graphite. Deposition conditions were not optimum, i.e., deposition temperature were not known within 50°C. The batch process did not produce really good or consistent fibers. Plasma arc testing resulted in melting of the W-coated B (probably by formation of W_2B_5). TiB_2 fibers had erratic test behavior. TiC had good resistance to BF_3, while graphite had the best resistance. In plasma containing H_2O, the graphite, TiB_2 and ZrB_2 completely eroded. Design criteria for reinforcing materials in rocket nozzles are given, and test techniques are described.

Fibers
TiB_2, TiC, ZrB_2, graphite,
ZrO_2, W on B, B on C

206. Growth of Composites From the Melt, Part I

Mollard, F. R., Flemings, M. C.
(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)
M. I. T.

A theoretical discussion is given on the conditions necessary to grow eutectic-like fibers in a matrix by directional solidification. They suggest it is possible to grow composites over a wide range of compositions, perhaps over the entire composition range. This structure is achieved by very slow (1 cm/h) growth rates and high temperature gradients (400°C/cm) with a planar front and no convection. Convection was eliminated by working with Pb-Sn alloy which rejected Pb at the interface, thus no density gradients were set up in the vertical furnace. The interface is held at the eutectic temperature, and liquid composition fluctuated about eutectic composition. Computer program was developed to predict effects of changes of growth rates, composition transients, using Fick's second law. Initial transient is about D/R:D = diffusion coefficient; R = velocity.

207. Growth of Composites From the Melt, Part II

Mollard, F. R., Flemings, M. C.
(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)
M. I. T.

Paper presents experimental confirmation of Part I theory. Pb-Sn alloys with from 12 to 26% Pb were unidirectionally solidified to form fibers in a eutectic-like matrix. Breakdown to cellular growth was about as predicted by constitutional supercooling theory. Changes in growth rate caused changes in fiber spacing and average composition of the solid. They took composition samples at various places along the solid, and found little deviation from nominal composition. By proper selection of G/R, they got rods of Sn to grow in the Pb-Sn alloy, without longitudinal segments.

208. C Fibers of High Strength and High Breaking Strain

Moreton, R., Watt, W., Johnson, W.
***Nature*, Vol. 213, pp. 690–691, Feb. 18, 1967**
Royal Aircraft Establishment

Heat treatment of C fibers at temperatures of 1200–2500°C gave higher strengths, but lower modulus and higher breaking strains. Use of 1-cm gauge length gave higher breaking strains. Use of 1-cm gauge length gave higher strength than 5-cm gauge lengths (up to 0.745×10^6 psi for 1 fiber treated at 1600°C). No maximum in modulus as a function of treatment temperature was found, but strength peaked at 1500°C. No evidence of surface flaws was found.

Fiber
C

209. Fiber Reinforced Metals

Morley, J. G.
***Sci. J.*, Vol. 2, pp. 42–47, Nov. 1966**
Rolls Royce

General review of composites, particularly stressing metals, is given. Advantages of whiskers, metal matrices, and disadvantages of composites, such as wire-reinforced metals and eutectic alloys, also are covered. Little or no increase in specific stiffness occurred in wire-reinforced and eutectic composites. Rolls Royce is working mainly with glass in Al, and C in resins. Obtained appreciable improvement in properties. Article is a good, but not comprehensive, general summary.

Fibers	Matrices
C, B, wires, glass	Al, resin, metals

210. Fatigue Behavior of Tungsten-Reinforced Silver and Steel-Reinforced Silver Composites

Morris, A. W. H., Steigerwald, E. A.

(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)

TRW

Improved fatigue properties were obtained when 20-vol% steel was added to Ag; tempering at 700°C gave better fatigue strength than at 300°C. A 4-mil W in Ag gave a drop in the elastic limit/UTS ratio above 12% W, probably because fracture becomes localized. At high loadings the force fields interact (spacing = 0.6 diam at 30%), causing premature failure. The mode of fatigue loading is very important, and must be considered when examining results. Each system will be different due to changes in fibers, matrix, process, and loading. Fiber-matrix bond also is important.

Fibers	Matrix
W, steel	Ag

211. An Investigation of the Fatigue Behavior of W-Reinforced and Steel-Reinforced Ag Composites

Morris, A. W. H., Steigerwald, E. A.

***Trans. AIME*, Vol. 239, pp. 730–739, May 1967**

TRW

This article is previously abstracted in a paper given at AIME meeting Feb. 1967 (entry No. 210). Composites were prepared by vacuum infiltration with Ag and tested in tension-tension fatigue. Both continuous and discontinuous 4-mil diam W wires were used; 15-mil diam steel wires were continuous, but tempered at 300 and 700°F to get different reinforcement strengths. All reinforced systems had improved fatigue strength, with higher strength for greater volume fractions of fibers. Each system had unique fatigue properties dependent on the elasticity of the components, but independent of surface condition. Fatigue failure mode for W-reinforced systems is sensitive to fiber spacing, and occurs in the fibers for more than 10% reinforcement due to interaction of the stress fields around the fibers. Steel reinforced Ag failed by matrix fatigue cracking followed by localized fiber failure. When the volume % W fiber was low, fiber failure tended to be at $L/d = 5$ (critical length?). Suggested that stress concentrations at fiber ends or breaks may be enough to cause adjacent fibers to break when spacing is close enough, i.e., % fibers is high enough.

Fibers	Matrix
W, steel	Ag

212. Filament-Wound Structures

Morris, E. E., Sanger, M. J., Darms, F. J.

***Space/Aeronaut.*, Vol. 47, No. 2, pp. 86–92, Feb. 1967**

Aerojet-General Corp.

Article cites advantages of composites over metals for specific applications, such as tanks, rotor blades and hubs, wings, and rocket cases; these include higher strength/density ratio, ease of fabrication, lack of plastic fiber deformation, improved reliability due to redundant design, short lead times, and use of noncritical materials. New, high strength, high modulus fibers are most useful when loads and structure can be designed for uniaxial stress paths. This requires an interactive design and fabrication method, including load analysis, selection of fiber and matrix, specifying orientation and composition of the components, and fabrication control. Relative structural efficiencies of different metals and composites combinations are given, and projected future yield strength/density curves are presented. Glass is the material mentioned most, although graphite is discussed. Present graphite fibers are porous and absorb moisture, and they may need surface finishes and protective coatings. Resins matrix properties which must be considered are viscosity and pot life, control of interim cure, fiber wettability, processing and curing temperatures, shrinkage, liberation of volatiles, shelf life, and toxicity. Interlaminar shear strength, elongation, and notch toughness also are important. Composite properties are affected by the shape of the object; winding path must hold the fibers in place to avoid slippage or bridging of the fibers. Fiber orientation must be maintained during service. To eliminate the problem of permeability in resin matrix tanks, liners must be used, and should be as low in weight as possible. If heavy wall liners are used, the tank becomes glass-fiber reinforced, and the metal may be strained plastically, with the fibers holding it in shape. Some advantages and applications of composites, such as wing boxes and rotor blades, are noted.

Fibers	Matrix
Glass, B, C, SiC, graphite	Resins

213. Growth and Characterization of Beta-Silicon Carbide Single Crystals

Nelson, W. E., et al.
AFCRL-66-579, May 1966
Stanford Research Inst.

SiC dendrites were grown from a melt containing excess Si by controlled freezing. Variables, such as thermal gradient, impurity concentration (mainly Fe), stirring rate, position in the crucible, and dendrite size, were studied. Low stirring rates and Fe concentrations gave longer dendrites; relatively abrupt and steep thermal gradients aided dendrite formation, as did adding 0.1% Ta. Additions of Y, Al, Au, Cr, Ni, Pt, Ag, Mo, Co, Pb, and Sm were tried, but with less success.

Fiber
SiC

214. Pyrolytic Deposits of SiC

Noone, M. J., Roberts, J. P.
***Nature*, Vol. 212, No. 5057, p. 71, Oct. 1, 1966**
Univ. of Leeds

SiC was grown from CH_3SiCl_3 vapor by H_2 reduction on graphite and pyrolytic graphite in an alumina tube. Deposit form was independent of temperature between 1200–1700°C, but it was dependent on gas flux (velocity times concentration). At fluxes above 1650 cm/min, layers were formed; below this level, whiskers were formed (with longest and largest whiskers formed at lowest fluxes).

Fiber
SiC

215. Design of an Aircraft Utilizing Fiberglass Reinforced Plastic Primary Structure

Noyes, J. V.
***J. Aircraft*, Vol. 3, No. 5, pp. 436–442, Sept.–Oct. 1966**
Douglas Aircraft

An analytical design study is made of the structural features and technical approach to show the feasibility of building a low-wing, light airplane from reinforced plastics. Fluted core and honeycomb core sandwich panels were analyzed. Advantages included fail-safe ability after damage, not notch sensitive, minimum number of assemblies, ease of field repair, low thermal conductivity, no corrosion, high impact resistance, lower flight drag, and reduced noise and vibration transmission.

Fiber	Matrix
Glass	Resin

216. Kossel Studies of Iron Whiskers

Ogilvie, R. E., Jansen, C. H.
RTD-TDR-63-4198, Part II, June 1966
M. I. T.

Whiskers may be grown from the vapor phase, from the bulk, or by decomposition of metal salts. Most whiskers are grown by climb of screw dislocations, which may be identified by the twist of the whisker, known as Eshelby twist. Kossel techniques were used to examine Fe whiskers grown by halide decomposition to identify growth mechanisms. No twist was found, indicating that Fe whiskers cannot be said to grow from screw dislocations. It is possible that no twist occurred because the dislocations climbed out of the whisker during or after growth, twist may have been present below the calculated amount and been too small to see, or there might have been paired dislocations, giving no net twist. Growth by nucleation of whiskers on impurities is ruled out because of the high purity of the materials. The Kossel methods are described and discussed.

Fiber
Fe

217. Composite Materials

Outwater, J. O., et al.
***Mech. Eng.*, pp. 32–39, Feb. 1966**
Univ. of Vermont

General survey of the field of composites from a mechanical viewpoint is given. Advantages of different types of composites and properties of each are discussed. Importance of designing each system and fabrication method for particular applications is emphasized. Form parts, not material for later fabrication when using fibers, is recommended. Wide range of properties are available. Data relating tests on samples to actual components under service conditions is needed. Present theories do not permit extrapolation of uniaxial loading to multiaxial loading conditions. Analytics models now available are inadequate. Failure modes are not known.

Fibers
Particles, layers, fibers

218. Fiber-Reinforced Metals and Alloys

Parikh, N. M.

ARF-2193-6, Feb. 1961 (AD-255992)

IITRI

Random felts were reinforced with wires. Thick wires had little strengthening effect, as did less than about 16% reinforcement. Up to 40 vol% strengthening was apparent. Higher concentrations were not tested. The felt did not have randomly oriented fibers, but had more aligned in the x-direction than the y-direction. Short fibers had little strengthening effect. Notch toughness and elongation were reduced in all systems. Inter-fiber spacings of 0.8mm or less seemed necessary.

Fibers	Matrices
W, Mo, steel, 403SS, 17-4PH	Ag, Cu

219. Deformation and Fracture in Composite Materials

Parikh, N. M., Warwick, D. N.

IITRI B-6073-6, Mar. 31, 1966 (AD-480418)

IITRI

The effect of microstructure on fiber reinforced composites is described. Mechanical strength is affected by fiber length, diameter and spacing, and method of composite fabrication. Critical lengths of fibers were determined for various conditions. They state that a ductile or fiber pull-out failure is desirable, while a brittle fracture of the fibers is undesirable; when L/d was more than 5, failures were brittle and weaker. They also note that, if L_c is about equal to interfiber spacing, g strengths are a maximum; but, if L_c/g is less than 1, the composites are low in strength and ductile. They did not find that strength increased with increasing fiber content according to Rule of Mixtures. This work is at variance with the NASA Lewis effort, which indicated that fiber pull-out was an indication of poor interfacial bonding and inadequate L/d. They also report that W in Ag, when strained, causes strain hardening of the matrix which peaks 2–3 d from the fiber end, and falls to almost nothing at the center. Data were obtained by microhardness and metallographic examination. Composite elongations were strongly affected by fabrication methods. Higher sintering temperatures gave higher strength interfacial bonds and better stress transfer. If interfiber spacing is too large, strengthening is ineffective.

Fibers	Matrices
W, steel	Ag, Pb-Sn

220. Whisker Reinforced Plastics and Metals

Parratt, N. J.

***Chem. Engr. Progr.*, Vol. 62, No. 3, pp. 61–67, Mar. 1966**

Ministry of Aviation Walleton Abbey

Discussion of whisker formation and theoretical aspects of reinforcement are given. Separation of whiskers was made from as-formed mats. Random mats of Si_3N_4 in Ag did not improve elastic limit, and a strain of 2% was required to develop maximum stress. After 100 h at 1100°C, Si_3N_4 whiskers in Ni spherodized. At the same temperature in Ni and Co, this occurs in a few hours. Al_2O_3 and SiC seem to behave similarly.

Fibers	Matrices
Si_3N_4, Al_2O_3, SiC	Ag, Ni, plastic, Co, Ni-Cr

221. Advanced Composites for Structures

Peterson, G. P.

(AIAA Paper 65-760 presented at the AIAA Aircraft Design and Technology Meeting, Los Angeles, Calif., Nov. 1965)

AFML

Review of programs in advanced composites for structures is given. This paper is abstracted in following entry No. 222.

222. Advanced Composites for Structures

Peterson, G. P.

***J. Aircraft*, Vol. 3, No. 5, pp. 426–430, Sept.–Oct. 1966**

AFML

Review on the status of composite materials and on the Air Force programs in the field is given. Development of high strength, high modulus fibers and their use in structures is a major advance. Additional development of design and fabrication is needed. Demonstration components are being developed to show utility of composites in high payoff areas.

223. Alloying Effects on Tungsten-Fiber-Reinforced Copper-Alloy or High-Temperature-Alloy Matrix Composites

Petrasek, D. W., Weeton, J. W.

NASA TN D-1568, Oct. 1963

Lewis Research Center

The effects of alloying additions with differing solubility in the W fiber were studied, using Cu as the matrix and alloy carrier. The strength and microstructure of composites as a function of reaction with the W was found to correlate with solubility of the elements in W. Sample preparation by infiltration is described. Co and Al in the Cu caused recrystallization of the W; Ti, Cr, Nb, and Ni-Fe formed varying amounts of alloy with the W. The Nb-Ni did not cause the W to recrystallize, while Ni in Cu or alone will; however, the Ni-Nb matrix is brittle, and did crack the W wires when the matrix cracked. Plating Cr on W and then infiltrating with Cu gave a barrier layer of Cr after infiltration. The Co-base alloys attacked and partly dissolved the W. Solubility of the alloying element in W seems most important in weakening and recrystallizing W. Al, Ni, and Co in small amounts lowered composite strength; Ti and Zr had less effect, and larger amounts were needed to do the same damage. Formation of brittle matrices, as in Ti-Cu and Zr-Cu, also lowered composite strengths. As the amount of alloying element increased, W damage increased; diffusion and recrystallization were more deleterious than alloying or reaction without recrystallization. High melting point materials caused the fibers to dissolve during infiltration. Good composite strength was associated with good ductility.

Fiber	Matrices
W	Cu, Cu-Ni, Cu-Co, Cu-Al, Cu-Zr, Cu-Nb, Cu-Ti, L-605, S-816, Ni-30Fe, Nb-48Ni

224. Elevated Temperature Tensile Properties of Alloyed W Fiber Composites

Petrasek, D. W.

NASA TN D-3073, Oct. 1965

Lewis Research Center

Material was also reported in *Trans. AIME*, Vol. 236, (entry No. 225). Additions of Ni formed a brittle interface which reduced composite strengths at all temperatures. Ductility of Ni alloys was better above 300°F than at room temperature. Both Cr and Ni additions reduced composite strengths and ductility, but Cr was less severe than Ni, since the diffusion zone was thinner. At low fiber contents, the Cu-Cr composite was slightly stronger than W-Cu composites, because Cr strengthened the matrix. Ni appears to diffuse into W along grain boundaries, reducing the effective fiber diameter and forming a brittle zone which acts as a notch. At 1500°F and above, there is little difference in the strengths of Cu and Cu-Cr matrix composites.

Fiber	Matrices
W	Cu, Cu-2% Cr, Cu-10% Ni

225. Elevated Temperature Tensile Properties of Alloyed Tungsten Fiber Composites

Petrasek, D. W.

***Trans. AIME*, Vol. 236, pp. 887–896, June 1966**

Lewis Research Center

Alloying between matrix and fiber reduced composite strengths. Binary alloys with the second component soluble in W were used. Ni formed a brittle phase with the W, and greatly reduced both composite and fiber strengths; Cr formed a ductile phase, and slightly improved composite strengths at 1200°F and above.

Fiber	Matrices
W	Cu, Cu-2% Cr, Cu-10% Ni

226. Metallurgical and Geometrical Factors Affecting Elevated-Temperature Tensile Properties of Discontinuous-Fiber Composites

Petrasek, D. W., Signorelli, R. A., Weeton, J. W.

NASA TN D-3886, Mar. 1967

Lewis Research Center

Composites were formed by vacuum infiltration around bundles of 5-mil W wire and tested at 300, 900, and 1500°F. As temperature increased, L_c increased. Fiber orientation also was important, especially at higher temperatures. As volume % fiber increased, less and less misalignment could be tolerated for a given L/d ratio. For a given misalignment and % fiber and as L/d increased, composite strength increased. For very small misalignments, there was some strengthening of the composites. More than 3° misalignment caused severe loss of strength, and was most noticeable for highest temperatures and lowest L/d. Adding Cr gave a reaction zone in the fiber, and reduced its strength but gave net increases in composite strength due to improved matrix shear strength. Load transfer between fiber and matrix is a function of matrix shear strength. Greater matrix shear strength also allowed greater misalignment without strength reduction.

Fiber	Matrices
W	Cu, Cu-Cr

227. Plastic Deformation and Failure of Silver-Steel Filamentary Composites

Piehler, H. R.

***Trans. AIME*, Vol. 233, pp. 12–16, Jan. 1965**

M. I. T.

Steel wires were Ag plated, placed in Ag tubes, and drawn. Tensile strength and elongation were determined and compared to Rule of Mixtures predictions; strength followed the predicted values, but elongations were about twice the expected amounts. This probably is due to matrix restraint of wire necking, which allows greater deformation before fracture. This restraint is increased as fiber packing density is increased. Composite failure is by successive wire fracture.

Fiber	Matrix
Steel	Ag

228. A Theory of Fiber Strengthening

Piggott, M. R.

***Acta Met.*, Vol. 14, pp. 1429–1436, Nov. 1966**

Atomic Energy of Canada

Fiber strengthening theory is developed, taking into account both elastic and plastic stress transfer between fiber and matrix. Only limited strengthening occurs under elastic conditions, so some stress transfer must be by plastic deformation of the matrix. Both non-work hardened and work hardened materials are considered. The minimum critical fiber length in W-Cu composites is reduced by about a factor of 3, if the relationships developed in the paper are applied. Stress transfer at the fiber ends is unimportant for large L/d ratios. Curves are given for effect of aspect ratio on strengthening factor without plastic deformation. Curves for strengthening factors as a function of the proportion of fiber stressed by the matrix in W-Cu and silica-Al also are given. Low fiber-matrix bond strength is not a problem for large L/d, and it may even be advantageous for fracture toughness limited applications. The theory is only applicable for d appreciably greater than matrix grain size; stress transfer to nearby fibers is neglected, as is stress transfer at fiber ends.

229. The Design and Construction of Filament-Reinforced Composite Rotor Blades

Pinckney, R. L., Hoffstedt, D. J.

(Paper C-4, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Boeing Vertol

Paper summarizes problems found using present helicopter blade materials and design, including: weight, dynamic balance, cost, maintainability, fatigue failure, corrosion, leading edge erosion, and notch sensitivity. Programs to design and build improved composite blades are noted and discussed. Use of composites permits improved blade configuration, more complicated shapes at no more cost, less notch sensitivity, better performance, lighter weight, and less corrosion susceptibility. Blade frequencies can be adjusted by varying stiffness. Problem areas are: lack of data on fabrication and on performance of advanced composites; and little design and use experience. Cost of B reduces its usefulness. Full-scale glass-epoxy blades have been made and tested successfully. Flight tests of advanced design glass-epoxy blades are scheduled for early 1968; flight tests of similar B-epoxy blades are planned for early 1969.

Fibers	Matrix
Glass, B	Epoxy

230. Control of Composite Microstructure Through the Use of Coated Filaments

Porembka, S. W., et al.

(AIAA Paper No. 67-175, presented at AIAA 5th Aerospace Sciences Meeting, New York, N. Y., Jan. 23–26, 1967)

Battelle Memorial Inst.

Graphite and Thornel 25 yarns were precoated with Ni and Co by electroless plating followed by swaging to densify the composite before gas pressure bonding for 1 h, 10,000 psi at 700 to 1000°C. Pretreating the graphite with stannous chloride solution to activate the surface before plating was essential. Good densification in Ni composites was only possible when 60% Ni was deposited. Co could be densified with as little as 23% Co, but better results were obtained at above 40% Co. The Ni composites were brittle because P was absorbed from the plating solution. This limits the usefulness of the composite to below 875°C; Co was not as badly embrittled, and is usable to 1020°C. Ti powder was put on B by coating with glycerine, then powder, outgassing in vacuum at 600°F, and then gas pressure bonding. All samples gave good control of fiber spacing and dense structures. Fiber integrity and alignment was maintained in the composites.

Fibers	Matrices
B, graphite, Thornel 25	Ni, Co, Ti

231. The Strength of Cd and Zn Whiskers

Predvoditelev, A. A., Zakharova, M. V.

***Sov. Phys. — Solid State*, Vol. 7, No. 2, pp. 305–310, Aug. 1965**

M. V. Lomonosov State Univ./Moscow

The strength of Cd and Zn whiskers is proportional to L/d^2, and may be due to whisker orientation, and dislocation density. Dislocations increase approximately as d^2. Surface or volume defects act as sites to nucleate dislocations.

Fibers
Cd, Zn

232. Preparation and Properties of Fiber-Reinforced Structural Materials

Price, D. E., Wagner, H. J.

DMIC Memo 176, Aug. 22, 1963

Battelle Memorial Inst.

This survey includes composites with the fiber or matrix being metallic; areas reported are fiber preparation, materials used, composite preparation, and properties. Powder metallurgy and melt infiltration are the compositing methods discussed. Vacuum infiltration of prepressed fiber felts followed by rolling has been successful for Ag with 1/2- and 1-mil steel-alloy fibers. Additives have been used in the matrix to improve wetting to the fibers. Elastomers and plastomers have been reinforced with chopped steel wires. Increases in strength and modulus of the composites were a direct function of the amount of fiber added; this is true at room and elevated temperatures. Changes in matrix composition can have significant effects on composite properties. Metal reinforced ceramics have not shown much improvement in properties, but some improvement in crack resistance was noted. Effects of process variables, such as fiber alignment, diameter and concentration, changing pressing and sintering conditions, rolling, etc. are noted. Problems, such as fiber-matrix bonding, best fiber length, fiber properties, effects of orientation, processing conditions, etc., need more study.

Fibers	Matrices
W, 316SS, Mo, Al_2O_3, 410SS, Ta, steel, Nichrome, 430SS, L-605	Co, Al, Cu, Ti, Ag, Ni, Al_2O_3, 316SS, plastics, ZrC, ThO_2, C

233. Carbon Yarn Reinforcements in an Epoxy-Anhydride Binder

Quackenbush, N. E., Sterry, J. P.

(Paper A-6, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Aeronutronic

Filaments formed from rayon were tested bare and in an epoxy composite. The strength and modulus values of the strongest C fibers in epoxy compared favorably with glass and B composites. C fiber is weakened by absorption of water, but other absorbed gases seem to have little effect. It is necessary to pretreat the fiber to remove water, and process the fiber into a final shape without storage. Otherwise, low and erratic properties were obtained.

Fiber	Matrix
C	Epoxy

234. Studies of W Composites Containing Fibered or Reacted Additives

Quatinetz, M., Weeton, J. W.

NASA TN D-2757, Apr. 1965

Lewis Research Center

This report is the basis for the paper abstracted in following entry No. 235. Preparation procedures, process variables, and properties are discussed. Extrusion pressures for HfO_2 additions did not correlate directly with composition. Increasing oxide content of ThO and ZrO_2 samples from 8 to 15% seemed to increase L/d ratios, but at 20.5 to 25.5%, agglomeration occurred. Best fibering of the refractories was HfN; worst fibering was HfB. For the same additions of ZrO_2, increased extrusion ratios gave increased L/d ratios. Property data are tabulated. Results are given in another abstract (entry No. 236). HfB appears to have reacted with the W. The method prevents handling and oxidation damage to the fibers, enhances bonding, and permits deformation of relatively hard and brittle materials into fibers.

Fibers	Matrix
HfO_2, HfB, HfN, HfC, TaC, ZrO_2, ThO, YO	W

235. Investigation of Extruded Tungsten Composites Containing Fibered or Reacted Additives, Part I

Quatinetz, M., Weeton, J. W., Herbell, T. P.

***Int. J. Powder Met.*, Vol. 2, No. 1, pp. 21–28, Jan. 1966**

Lewis Research Center

This paper was previously abstracted as NASA TN D-2757 (see entry No. 234). Fine powders of W and the desired additive were blended and hot extruded to obtain fiberization of the additives. Fabrication techniques are described. Tests included stress-rupture, hardness, step-load creep, density, grain size, and fiber length to diameter ratios. Composition and process history had a notable effect on properties. Test results, particularly stress-rupture, are given as a function of additive. Oxides fibered more than refractory compounds.

Fibers	Matrix
ZrO_2, ThO_2, YO_2, TaC, HfO, HfB, HfC, HfN	W

236. Investigation of Extruded Tungsten Composites Containing Fibered or Reacted Additives, Part II, Analysis of Data

Quatinetz, M., Weeton, J. W., Herbell, T. P.

***Int. J. Powder Met.*, Vol. 2, No. 2, pp. 51–64, Apr. 1966**

Lewis Research Center

Mixtures of oxides and refractory compounds were extruded at 4200°F, 8:1. Fiberization of oxides ranged from 7 to 23:1; for others, it ranged from 1 to 18:1. Appreciable increases in stress rupture and creep occurred when TaC, HfB, and HfC were added in amounts of 8%. Reactions between W and these additives occurred, and might have contributed to increased strength, in spite of the lack of fibering. Oxides showed more fibering, but little increase in strength. Additions of over 15% were not too effective, and agglomeration increased. Stress-rupture of oxides decreased with increasing content above 16%, but these samples were hard to machine, and sample preparation problems may have given low results. Sintering W in H_2 and then in vacuum improved stress-rupture life, but variables such as extrusion ratio, canning material, or use of high-energy presses had little effect. Grain size of W was reduced appreciably with all additives. All oxide additions increased matrix hardness, HfN increased hardness, and TaC decreased it.

Fibers	Matrix
ThO_2, ZrO_2, HfO_2, YO_2, HfB, HfC, TaC, HfN	W

237. Colloidal Asbestos Fibrils as Reinforcement for Polymeric Structures

Rader, C. A., Schwartz, A. M.

Final report on NASW-1420, May 1967

Harris Research Labs, Washington

Various resins were reinforced by colloidal asbestos fibrils, using method described in the report. Strengthening obtained depends on chemical nature of the matrix and the amount and type of surfactant used. Small amounts aid in preventing rubbing and breakup of fibers, but too much interferes with bonding. Phenolic resins, ice, and carbolyxalated resins were strengthened; nylon, gelatin, polyurathanes, and polypropylenes were not strengthened (but % fibers in nylon and polypropylene was low, probably less than critical %). Orientation of fibers increased composite strength. Methods of orientation are given. As fiber diameter decreased, strength seemed to increase. Phosphatesters were found to be satisfactory surfactant.

Fiber	Matrices
Asbestos	Resins

238. An Evaluation of New High Performance Fibers for Structural Composites

Rauch, H. W., Sr., Sutton, W. H.

ASM D5-14.2, Oct. 1965

General Electric Space Sciences Lab

Review of fibers, whiskers, and composites is presented. Comments are given on high potential strengths as compared to bulk materials, reasons for strength increases as fiber size decreases, forming methods, high temperature strength retention, problems of fiber-matrix compatibility, and fiber types (such as glass, polycrystalline nonmetals, metal wires, carbon/graphite fibers produced from organic yarns and whiskers). Their conclusions for high-performance fiber development are: to examine factors of fiber formation that affect strength, to learn to put fibers into matrices successfully, to develop understanding of interfaces between fiber and matrix, to protect the fibers in the matrix, and to reduce the costs of the reinforcements and processing into composites.

Fibers
B, B_4C, SiC, BeO, SiN_4, Al_2O_3

239. Survey of Ceramic Fibers and Fibrous Composite Materials

Rauch, H. W., Sr., Sutton, W. H., McCreight, L. R.

AFML-TR-66-365, Oct. 1966

General Electric Space Sciences Lab

Report gives an excellent overall survey of effort on fibers, whiskers, composites, testing, etc., both in the U.S. and Europe. Not all the organizations and programs are listed, but the vast majority are noted or discussed in some detail. It is divided into sections on: (1) summary of technical status, (2) lists and abstracts of over 200 patents, (3) reports on 95 personal contacts with active organizations, and (4) a bibliography of 797 items, reports, papers, etc., mostly published during 1965–1966. The text covers discussions of reinforcements, including characterization and preparation, properties, and applications. Methods for fiber preparation are melt drawing, wire drawing, bundle drawing, Taylor wire, extrusion, Rayon spinerette, conversion, and chemical vapor deposition. Active organizations are identified, and typical properties are quoted. Test methods, problems, and sources of error are discussed in some detail. Major sources of error are handling and aligning the fibers and especially the whiskers, measuring deflections and areas accurately, and accounting for statistical and structural variations in individual fibers. Gauge length has an effect on apparent UTS and modulus. Bend tests are useful to find the quality of fiber or whisker batches, but they correlate poorly with tensile properties and should not be used to establish anticipated composite behavior. Dynamic tests, such as torsion, vibrating reed, standing wave, and pulse propagation, are described. Versatility and type of properties obtainable in composites are listed and commented on. Effects of fiber length, diameter, orientation, volume fraction, fiber shape, constituent behavior, and composition are noted. The importance of mechanics and interface properties is emphasized, and use of additions to aid wetting and bonding are illustrated. Critical volume and fiber length are described. Resin-matrix composite fabrication methods, such as spray up, hand layup, filament winding, and preforms, are noted with applications given. Metal-matrix fabrication methods include infiltration, powder blending, vapor plating, electroplate, melt drawing, diffusion bonding, extruding, and directional solidification. Advantages of metal matrices are cited. Most of the work in the past was on model systems to define important parameters in strengthening behavior. Properties of various systems are given, both at room and elevated temperatures. Applications noted are aircraft engines, wing boxes and leading edges, turbine blades, rocket nozzles, cables, high temperature bearings, pressure vessels, castings, and deep submergence structures. Ceramic matrix composites are discussed also. Strengths are more limited than in metal matrix parts, but thermal properties such as conductivity and shock resistance can be improved. Fabrication techniques include powder metallurgy, slip casting and sintering, melt casting, and prepregnation layup. Limited applications include radomes, printed circuits, dies, insulators, and antenna windows. Advantages, disadvantages, costs, and maximum temperatures of use are discussed and tabulated. Each reinforcement has specific advantages, disadvantages, and attractive uses, as does each reinforcement-matrix combination. Areas needing further work are identified. Notable trends are availability of vapor deposited fibers and preparation of demonstration components, the rapid progress in new types of reinforcements, availability of fine metal fibers, rapid growth of interest in metal matrix composites, and recent emphasis on whisker reinforced composites.

Fibers	Matrices
All	All

240. Preparation of B Filaments on Fused Silica

Reeves, R. B., Gebhardt, J. J.

(Paper D-2, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

General Electric Space Sciences Lab

B is deposited on a C-coated silica substrate using diborane. Dilute solutions in A gives a diffusion limited reaction; pure diborane at 0.03 at. is surface diffusion limited, and about 10 times as fast. The effects of temperature, reactor design, concentration, and filament speed were studied. Deposition temperatures are about 1300°F vs about 2000°F for BCl_3 on W, and the substrate density is lower. Cross flow reactors with Hg seals and electrical contacts were found most effective. A cold wall reactor is needed to prevent premature decomposition of the diborane. Maximum deposition rates were obtained at low concentrations, moderate flow rates, and temperatures between 500 and 750°C. There seems to be no correlation between filament properties and flow rate or drawing speed, but both temperature and concentration have strong effects. Increasing the deposition rate above about 120 mil/h requires either greater concentration or temperature, which lowers modulus and tensile strength, respectively, in the dilute system. The low pressure system gave the same dependence of modulus on process conditions; strength is increased at lower feed rates. Reactor geometry had no effect on modulus, but a 6-stage reactor improved modulus and strength. Routine production material has an average tensile strength of 0.35×10^6 psi, modulus of $52–55 \times 10^6$ psi, density of 0.085 lb/in.^{3}. Composites made with these fibers retain more strength after 2 h in boiling H_2O than those made with B on W. There is no B-core reaction, and residual stresses are beneficial rather than detrimental. Substrate surfaces are smoother, giving better B surface smoothness.

Fiber
B

241. Structure and Properties of Al_2O_3 Whiskers

Regester, R. F., Gorsuch, P. D., Girifalco, L.
***Mater. Res. Stand.*, Vol. 7, No. 5, pp. 203–206, May 1967**
DuPont, General Electric Space Sciences Lab

The mechanical properties of alumina whiskers were studied as a function of diameter and surface area. The tensile testing device and techniques are described. Strength increased as diameter decreased. Fracture generally occurred along the $(\bar{1}2\bar{1}0)$ plane. UTS was based on actual measured whisker cross-sectional area. Whisker circumference and area were plotted against UTS, and both were straight lines with experimental points in good agreement with theory. It is concluded that strength is thus a function of perfection of the surfaces, not internal defects.

Fiber
Al_2O_3

242. Evaluation of the Potential Structural Performance of Composites

Riedinger, L. A.
(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)
Lockheed Missiles and Space/Sunnyvale

The potential of composites used in box beams and cylinders is noted. Both laminated orthotropic materials and box beam sections are considered. Comparisons were made of the compressive buckling efficiency of various materials, showing that Be is better than almost all composites. Be is superior for lifting and re-entry bodies, but composites are more efficient for submersibles. The effects of layup on the critical axial loading of cylinders was shown for B-epoxy and B-Al. The best winding angle was found to be 15°. Theory indicates that, for Al honeycomb with a B-epoxy cover on a box beam, a joint overlap of 3 in. is needed. Experiment tends to bear this out. Joints are a major problem in composites, and need much more study. Other major problem areas that need more effort are compressive behavior and structural design. Metal-matrix composites are better for buckling applications than resin-matrix ones. Twelve different composites were studied and compared, some with varying layups. Comparison also is made between composites and metals in box beam bending and cylinder buckling at room temperature and 1000°F. Joints can become complex, and can add significant weight if there are a number of them, or high reliability is needed, or a large safety factor must be used.

243. Adherence and Wettability of Ni, Ni-Ti Alloys, and Ni-Cr Alloys to Sapphire

Ritter, J. E., Jr., Burton, M. S.
***Trans. AIME*, Vol. 239, No. 1, pp. 21–26, Jan. 1967**
Cornell Univ.

Study of the effect of alloying additions and test atmospheres on wetting and bonding of Ni to Al_2O_3 is given. This study is similar to work previously done by Sutton, and both came to the same general conclusions. Pure Ni contact angle was unaffected by atmosphere, but increasing impurity content from 16 to 31 ppm increased shear strengths. Maximum shear strength for Ti additions occurred when no oxidation of the Ni drop occurred. Roughening of the interface occurred and probably caused improved shear strength. Wettability increased as a function of Ti content up to 0.97%, the highest tested. Wettability of Cr-doped Ni also improved as a function of alloy content, but above 10.5% no improvement was noted. Excess oxidation of Cr-doped Ni gave poor shear strength. For a given addition, contact angle decreased in A compared to H_2. Contact strength of pure Ni was best in impure A or vacuum and was worst in pure A and H_2. For best adherence of Ti-Ni alloys, limited reaction at the interface is required.

Fiber
Al_2O_3

244. Prospects Stepped up for Fiber-Reinforced Metals

Ronnholm, P.
***Metals/Mater. Today*, Vol. 60, No. 7, p. 46, July 1967**

Report of address by W. H. Sutton to Hartford chapter of ASM is given. More progress has been made in composites in the past two years than at any other comparable time. Fiber reinforcement is described, and some of the problems such as bonding, compatibility, and fabricability are stated. Advantages, such as strength retention at high temperature, are described. Fiber-reinforced metals soon will compete with conventional materials where their specific properties are useful.

245. Non-metallic Composites—Systems, Fabrication Techniques, and Properties

Rosato, D. V.

ASM Report C6-7.3, Oct. 1966

A general survey is given on composite systems, particularly non-metallics, with major emphasis on applications, types of matrix systems, fillers, fabrication techniques, and some property data. Metal reinforcements and matrices are lumped with plastics as a special case. Growth of industry use of plastics and future usage are discussed. Composites permit tailoring of materials to uses; wide variety of fabrication methods possible, each having advantages for certain applications or materials.

246. A Note on the Failure Modes of Filament Reinforced Material

Rosen, B. W.

GE Reprint No. 295, Feb. 1964

General Electric Space Sciences Lab

Presents an analytical study of the effects of constituent properties and geometry on elastic constants and failure modes of composite laminates, shells, and filament wound cylinders. A cylindrical shell in axial compression could have wide variations in filament orientation with only a small change in performance. An isotropic laminate was the most efficient system studied. Hollow glass fibers are about twice as efficient as solid fibers.

247. Mechanics of Composite Strengthening

Rosen, B. W.

GE Reprint No. 289, Nov. 1964

General Electric Space Sciences Lab

Analytical models are developed for behavior of composites loaded in the direction of uniaxially aligned fibers. Failure criteria are maximum tensile or compressive loadings. Experimental and analytical data are compared, and indicate that compressive failure is limited by shear stress in the matrix. Matrix materials with moderate rather than low moduli compared to the fibers are indicated as better in compression. Compression instability in composites seems to be analogous to column buckling. Expressions for the effects of component properties are given.

Fiber	Matrix
Glass	Epoxy

248. Tensile Failure of Fibrous Composites

Rosen, B. W.

***AIAA J.*, Vol. 2, No. 11, pp. 1985–1991, Nov. 1964**
(GE Reprint No. 302)

General Electric Space Sciences Lab

An analytical study was made on the failure mode of composites stiffened by uniaxially aligned fibers. Limiting strength factor was thought to be fiber defects which caused fracture of the fibers until individual fibers were less than critical length, at which point matrix shear failure occurred. Failure mode in the tests was by an accumulation of fractures and final matrix failure rather than excessive shortening of the fibers due to fracture. Failures of fibers due to surface imperfections probably will be much more important in glass-reinforced composites than when fibers are metallic or ceramic whiskers. Discontinuous fibers may co-exist with continuous fibers at a lower stress level; this will be well below maximum load on the continuous fibers. Matrix properties, particularly efficiency of stress transfer around a fiber break, will have a significant effect on composite strength. The model had admitted deficiencies.

Fiber	Matrix
Glass	Epoxy

249. Influence of Constituent Properties Upon the Structural Efficiency of Fibrous Composite Shells

Rosen, B. W., Dow, N. F.

GE Reprint No. 329, Apr. 1965
(Also NASA-CR-63452)

General Electric Space Sciences Lab

An analytical study is given on various fiber-composite systems for use as shell structures, particularly for launch vehicles. Monocoque construction does not utilize composite properties well, but sandwich designs do. Elastic buckling is the prime consideration for elastic shells. Isotropic fiber orientations gave best efficiencies. Hollow E-glass fibers are too weak in transverse shear to be useful, but hollow B fibers theoretically would be a big improvement due to high modulus. For high load intensities isotropic orientations impose weight penalties, and uniaxial alignment would be preferred. Low volume fractions of very stiff fibers would replace metals when loading is light. For composite shells, fiber modulus is relatively unimportant; but for isotropic sandwiches, increasing modulus is almost as good as lowering density.

Increasing binder modulus or decreasing density improves buckling efficiency. Relation between buckling efficiency and fiber property varies with configuration. Efficiency depends on modulus and strongly on density. Binder properties affect efficiency much less than fiber properties. Failure criteria are inadequately known.

Fibers	Matrices
B, steel, E-glass, Al_2O_3, asbestos	Mg-Li, Al, epoxy, B, Be, assumed alloys

250. Survey of Composite Materials Failure Mechanics

Rosen, B. W.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

General Electric Space Sciences Lab

The Rule of Mixtures is a poor approximation of the anticipated strength of composites, since the fiber properties used in the rule are based on a very wide range of properties. A statistical method should be used to determine properties of the fibers before applying the values. Other variables also should be considered, such as fiber length and the coefficient of strength variation. Different tensile failure modes were shown and discussed; these included bundle, cracking, and statistical failure. An average stress concentration factor can be calculated for breaking different proportions of fibers; as the number of fiber breaks goes from 1/n to 1/3, the factor goes from 1.33 to 1.50 in a 2-dimensional composite. In 3 dimensions, this factor is greatly reduced, as shown by experimental data. Failure modes for glass-epoxy composites are more like the probability of having two adjacent breaks which grow together to form a crack, rather than cumulative damage or a single bad break. This failure mechanism is demonstrated by comparison of data with theoretical curves. The normalized composite strength for discontinuous fibers as a function of fiber length is essentially that of continuous fibers as fiber length approaches 100 diameters. The composite is more forgiving of weak fibers than the fiber bundle; thus, fiber strength alone is insufficient to predict composite strength. Compressive strengths of composites can be postulated to be very high. Some work on composite compressive strength was discussed. It was emphasized that composites should be designed and used in uniaxial loading. Improvements in matrix properties will make a major contribution to improved composite properties.

251. The Statistical Breaking Strength of a Bundle of Classical Fibers

Rosenbaum, B. M.

NASA TN D-3137, Dec. 1965

Lewis Research Center

Mathematical expressions are given for the statistical distribution of breaking strength of a bundle of ideal fibers. One can predict the load carried and number of fibers still active on a probability basis. Variance of fiber strengths can be calculated.

252. Symposium on Fibrous Materials

Ross, T. H., editor

ASD-TDR-62-964, Jan. 1963, (AD-299030)

Directorate of Materials and Processes, AFSC

Fibers as small as 0.0005 in. were drawn from 0.005-in. commercial wire by using diamond dies. Die wear was excessive for all but Elgiloy and Hastelloy B. Fibers were drawn into yarn, and were tested both as fibers and yarn. A. D. Little studied strengths of superalloys and refractory filaments at elevated temperatures. At 1800°F, Ni-base alloys crept severely. Hot-dip Sn-Al on W protected against oxidation in air at 2000°F as long as 23 min. Electrodeposition of Ni formed fine fibers almost as good as wire drawn ones.

Fibers
Hastelloy B, Rene 41, A-286, Elgiloy, M-252, Udimet 500 and 700, Waspalloy, W, Mo

253. Nuclear Magnetic Resonance (NMR) as a Quality Control Tool

Rosten, F.

(Paper H-5, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Coast Manufacturing and Supply

NMR can be used to identify various resins and to follow the effects of storage, volatiles content, etc. Theory behind the test is given, as are test methods and sample preparation. Test results are summarized. The applications and properties studied are not considered to be the only useful tests which can be made using NMR techniques. Some potentially useful areas include optimizing cure cycles and detection of fiber prefinishes.

Matrix
Resin

254. Re-entry Vehicle Application of Advanced Filament Reinforced Structural Composites

Saffire, V. N., Shenker, L. H.

(Paper C-5, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

General Electric Missile and Space Division

Paper describes program to form cylinder with hollow, integrally stiffened ribs designed by advanced analytical techniques. The structure was characterized by shell diameter and length, ring spacing, ring thickness, ring depth and width, fiber pattern, and shell thickness. Ribs were wound over an eutectic alloy which melts at 285°F; this alloy was melted out of the cylinder after resin cure. Winding was in a helical pattern. Main problems were variations in prepregnated tape strength, width, resin staging and content, and broken fibers. B fibers were used successfully with no major problems. Testing was on flat plates for preliminary data, and on the cylinders to full design loads.

Fiber	Matrix
B	Resin

255. Whisker Composites by Unidirectional Solidification

Salkind, M. J., et al.

***Chem. Eng. Progr.*, Vol. 62, No. 3, pp. 52–60, Mar. 1966**

United Aircraft

This article summarizes the work done by United Aircraft in formation of eutectic, monotectic, and eutectoid alloys. Very good elongation is obtained; some alloys have been cold worked successfully and strengths approached those predicted by the Rule of Mixtures. Whisker precipitate gave better strengthening than lamellar precipitates which have imperfections on the blades. Materials were limited to particular alloys and % of whiskers was limited by phase diagram of system investigated. Bonding is excellent because of the low surface energy between fiber and matrix; this makes such alloys stronger than comparable composites. They can be heat treated without spherodizing whiskers. Also, controlled porosity bodies or controlled electrical properties can be made.

Fibers	Matrices
Al_3Ni, Ta_2C, $CuAl_2$	Al, Ta

256. Eutectic Composites by Unidirectional Solidification

Salkind, M. J., et al.

(Paper F-5, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

United Aircraft

Brief review of unidirectional solidification of composites is given. Rods are formed when composition of phases is uneven; lamellae form occurs when they are about equal. Process can be zone melting, slowly withdrawing one end of a sample from the furnace, or heating one end and cooling the other. Advantages of process are elimination of whisker handling, accurate spacing and alignment, good interfacial bonding, low interface energy, and stability at high temperatures. Strength of eutectic composites at high temperatures exceeds that of other alloys of the same matrix material. Creep behavior of composites reinforced by whiskers (rods) and plates differs with plate composites having more rupture ductility. Samples solidified at higher rates have smaller, closer spaced rods, and greater creep resistance than those with greater rod spacings. Notch impact strength as a function of orientation has been studied; they were found to have good notch toughness. Low cycle fatigue strength seems to be affected by fiber shape; the lamellae are more fatigue resistant. Transverse tensile strength is higher than the matrix strength. Composites were rolled successfully perpendicular to the fiber direction; they also have been swaged successfully. Diffusion bonding has been used to form cross-plyed laminates of Al-Al_3Ni composites.

Fibers	Matrices
Al_3Ni, $CuAl_2$, Ta_2C, Cb_2C, NiMo, Cr	Al, Cu, Ta, Cb, Ni, Cr-Cu

257. Fatigue of Eutectic Composites

Salkind, M. J., Bayles, B. J.

(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)

United Aircraft

Eutectic composites of Al-$AlNi_3$ and Cu-$CuAl_2$ were tested for fatigue in tension-tension. Both fiber and lamellar composites were tested; lamellar samples seemed more fatigue resistant. Higher strains may be possible in fatigue than simple tension, since in tension when the fibers become overloaded they fail and transfer all the load to the weaker matrix. Fatigue allows slow crack growth, and more strain. The endurance limits for both composites is about 10,000 psi and 10^8 cycles. Fatigue behavior of both the fiber and the composite should be considered. There is an apparent stress-corrosion effect; the endurance limit is higher in A than in air. Photomicrographs showing fibers turning aside cracks and holding together broken pieces of matrix were shown. Another method of reinforcement is to lower the strain energy. Reinforcement shape controls dislocation motion and fatigue behavior. Lamellae are more effective, in spite of greater interfiber spacing.

Fibers	Matrices
$AlNi_3$, $CuAl_2$	Al, Cu

258. Impact Behavior of Al_3Ni Whisker-Reinforced Al

Salkind, M. J., George, F. D.

(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)

United Aircraft

The impact behavior of 10-vol% Al_3Ni fibers and blades eutectically grown from Al was studied. The effect of orientation on Charpy impact strength was examined by cutting samples in six directions in relation to fiber growth direction. Four of six fiber orientation samples withstood testing by bending rather than breaking; five of six blade samples broke instead of bending. Toughness was excellent, even at liquid N_2 temperatures. Toughness was anisotropic. Cu-$CuAl_2$ blade samples had very poor impact resistance and low Charpy strength.

Fibers	Matrices
Al_3Ni, $CuAl_2$	Al, Cu

259. Metals with Grown-in Whiskers

Salkind, M. J., Lemkey, F.

***Int. Sci. Tech.*, No. 63, pp. 52–64, Mar. 1967**

United Aircraft

Article discusses composites formed by unidirectional freezing of eutectics, advantages, disadvantages, and some properties. Only eutectic or monotectic compositions can be used. Rods or whiskers will form only when the energy balance favors it; otherwise plates or lamellae will form. Above about 28%, rods seldom will be stable. If more reinforcement than this is needed, it will have to be as lamellae. Eutectics very near 28% may go either way depending on solidification conditions. If both phases have low entropy, controlled microstructures will form perpendicular to the solid-liquid interface; if entropy of fusion of one or both phases is high, controlled microstructure cannot be maintained. Strengthening is comparable to fiber reinforced composites. Rods are more efficient than plates, but both work. Eutectics are strong in only one or two directions, but are tough if tested in the proper orientation. Eutectic composites can retain strength and microstructure better at high temperature than plain matrix materials, thus better creep strength, e.g., Ta-Ta_2C is stronger above 2000°F than Ta-10W. Turbine blades have been cast directly from the melt in Ni-NiMo composites. Other applications are electrical or magnetic, where an inert phase is grown in an active matrix, such as NiSb in InSb. This type of material could be used as a brushless contact, since it can be electrically or magnetically actively in one direction only. Another application is freezing monotectics, leaving liquid in the fiber position. The liquid is blown out or otherwise removed, leaving aligned, uniformly spaced holes.

Fibers	Matrices
Al_3Ni, Cb_2C, $CuAl_2$, NiMo, TaC_2, NiSb	Al, Cb, Ni, InSb

260. Composites—Where do They Stand?

Salkind, M. J.

***Metals/Mater. Today*, Vol. 60, No. 7, pp. 29–31, July 1967**

United Aircraft

Review of advantages and problem areas in composites is given. Fabrication difficulties, such as uniform fiber distribution, joining, and interface reactions are touched upon. Fabrication techniques are described briefly, with the most emphasis on unidirectional solidification. Understanding mechanics of composites and developing testing techniques also are problems. Some applications are suggested; changes in roles of designers, fabricators, and material producers are projected.

261. Testing of Fibers

Schmitz, G. K., Metcalf, A. G.

***Mater. Res. Stand.*, Vol. 7, No. 4, pp. 146–152, Apr. 1967**

Solar

The effects of test techniques and meaning of strength values are considered for E-glass and S-glass. Important test conditions include gauge length, temperature, humidity, and strain rate. For example, gauge length increases cause decreases in strength, but not as a continuous function. A straight line relationship indicates control by a particular defect; changes in slope show changes attributable to a different type of defect as controlling strength. The slope of the line measures defect severity. A statistical analysis of test results is given, showing the effects of gauge length. Changes of strength as a function of strain rate and annealing also are discussed. Static fatigue data also are given.

Fiber
Glass

262. A Compatibility Study of SiC and B Fibers in Be, Fe, Cv, and Ni

Schneidmiller, R. F., White, J. E.

SSD-TR-66-220, Oct. 1966 (Also, Report TR 1001 (2250-10)-1 of Aerospace Corp., and Paper E-5, presented at 10th SAMPE Meeting on Advanced Fibrous Composites, San Diego, Calif., Nov. 9–11, 1966)

Aerospace Corp.

Composites were prepared by hot pressing powders and fibers at 5000 psi. Be matrix samples were pressed at 1000°C; Ni, Co, and Fe were pressed at 600°C. The samples were then exposed up to 100 h at temperatures from 600 to 1000°C. B reacted most with Ni, least with Be; Co was less stable than Fe, which was less stable than Be. Ni reacted with B during pressing at 600°C, while none of the others did. There was extensive interdiffusion of Ni and B after 100 h at 700°C. The B concentration in the reaction zone tended to stabilize at 8–10%, indicating compound formation. Fe and Co showed similar compound forming tendencies after short periods at 700°C. Reactions were greater for Fe and Co in the direction of pressing, which gave better conditions for interface formation. Interdiffusion was identified metallographically by microprobe and microhardness. Diffusion of B into Ni, Fe, and Co increased matrix hardness, but had no effect on Be. There was no indication of diffusion of Fe or Co into the B. Be seems to diffuse into the B; after 100 h at 1000°C, the B fiber had doubled in size. SiC is much less reactive in all matrices. Ni, Si, and C interdiffuse after 100 h at 700°C to form a compound. Co behaves the same way, but more slowly. Be diffused into SiC to form a compound after 24 h at 1000°C. The reaction zones between SiC and Fe or Co are much harder than the fiber when reacted at 700°C, but at 800–900°C the hardness is less than the fiber. The Be-B interface was a smooth transition, showing good bonding. Co-B after hot pressing and Fe-B after pressing at 700°C had interdiffusion zones. Be-SiC composites had a distinct reaction zone extending into the fiber. Co-SiC and Fe-SiC had poor bonding between matrix and reaction zone. Porosity formed at the reaction zone by thermal exposure can be reduced by hot pressing under the same conditions.

Fibers	Matrices
B, SiC	Fe, Ni, Co, Be

263. Compressive Strength of Boron-Metal Composites

Schuerch, H.

NASA CR-202, Apr. 1965

Astro Research Corp.

Compressive failure in laminates and metal matrix composites were studied from a theoretical standpoint. Metal matrix composites are limited by anelastic crippling; high yield strains will improve compressive strengths. Yielding in the ductile metal matrix occurs at a small fraction of ultimate failure stress. Fatigue applications should await better knowledge of behavior under cyclic stress. Several samples of Mg-B composites gave ultimate strengths in good agreement with theory.

Fiber	Matrix
B	Mg

264. A Contribution to the Micromechanics of Composite Materials—Stresses and Failure Mechanisms Induced by Inclusions

Schuerch, H.

ARC-R-210, Feb. 1966

Astro Research Corp.

Effects of various types and shapes of *inclusions* in composites were studied. Stress interaction effects, loading patterns, and shape factors were investigated. Best shape for fibers is rhombohedral (ellipse is next best) to avoid harmful interactions and to obtain maximum strengthening effects. Use of different sizes of fibers also improves strength properties. Transverse reinforcement in glass-resin systems improves composite strength.

Fiber	Matrix
Mathematical treatment of reinforcement micromechanisms	

265. Elevated Temperature Behavior of Fibers

Schulman, S.
ASD-TDR-63-62, Apr. 1963
ASD, Wright-Patterson AFB

Various fibers were tested at temperatures up to 1700°F for tensile properties, with a comparison of room temperature and high temperature properties. Fibers were heated 10 min at each test temperature before testing. W had no strength at 1400°F; Au plating W had increased strength by 3 times at 1200°F.

Fibers
Glass, PBI, nylon, Au on W, Au on Mo, Rene, Hastelloy, W, Chromel A

266. Single and Multi-fiber Interactions in Discontinuously Reinforced Composites

Schuster, D. M., Scala, E.
(Presented at AIAA/ASME 8th Structures and Materials Conference, Palm Springs, Calif., Technical Papers, pp. 80–87, Mar. 1967)
Cornell Univ.

This paper describes the study of discontinuous fiber reinforcement of photoelastic resins with B. The critical elastic aspect ratio was found to be 20; critical fiber length to fracture the B is about 5 times L/d for the elastic critical length. The effects of fiber spacing and overlap for 2-fiber composites was studied. To reduce stress concentrations in 2-fiber systems below the stress in single fiber systems, the overlap must be 1/2 the elastic critical L/d or more. High shear stresses result when overlapping fibers have a narrow spacing. Stresses increase rapidly for fiber spacings less than 1 diameter for no overlap or full overlap. For 50% overlap the stress concentration is less than for a single fiber. When fibers have close spacing and 100% overlap, stress concentrations in the matrix occur. Thermal prestressing the fiber reduced stress concentrations at room temperature when loaded in tension. Matrix weak spots and locations of stress concentration are dependent on fiber spacing and overlap. The ratio between fiber and resin modulus in this program was 125:1, which is comparable to that of actual composite systems. At spacings of 5 or 6 diameters, there is little interaction between fibers, and the amount of overlap has no effect.

Fiber	Matrix
B	Resin

267. The Reinforcement of Polymeric Structures by Asbestos Fibrils

Schwartz, A. M., Rader, C. A., Davis, A. E.
Final Report on Contract NASW-1183, May 1966
Harris Research Lab

Asbestos fibers 250–500Å in diameter with varying lengths were made from macroscopic asbestos fibers by ball milling in liquid, and stirring or mixing in a blender. Thin sheets up to 10-mil thick were formed by pressing to give isotropic fiber alignment. The effects of fiber size and surface wetting agents, such as oleate soap or linolic acid, were studied. Highest strengths were obtained with composites formed from long ultrafine fibers or a mix of long ultrafine fibers and macroscopic fibers. Poorest strength was obtained with short ultrafine fibers of almost all macroscopic fibers. Some papers had 80% voids, and none had over 55% fibers. It was hard to get large amounts of fibers in the papers and to obtain high densities. None of the samples tested were as strong as commercial asbestos papers, either as papers or in resin composites. Up to 20% surfactant had no effect on strength. Dispersing the asbestos directly in the resin and then making paper mats did give somewhat improved composite strength, indicating that a better bond was formed between fiber and matrix. Less than 5% of theoretical ultrafine fiber strength was utilized in the composite. Oriented fibers would give higher strengths.

Fiber	Matrices
Asbestos	Phenolic, polyester

268. Radar Reflection, Absorption, and Transmission of B Fiber Reinforced Plastics

Schwartz, H. S., Bahret, W. F.
MAN-AFML-66-11, Project 7340, July 1966
AFML, Avionics Lab

Radar characteristics of B on W and silica substrates were determined. B on W gives essentially the same radar reflection as metals, making it unacceptable as radome material. B on silica gives less radar reflection than fiberglass for internal reflections, but more than fiberglass for external reflection. Transmission through the B-resin composites was appreciably less than through fiberglass (~50%), making it unsuitable as radome material.

Fiber	Matrix
B	Resin

269. Mechanical Behavior of Be Wire Reinforced Plastic Composites

Schwartz, H. S., Mahieu, W., Schwartz, R. T.
(Paper A-5, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)
AFML, Univ. of Dayton

Both ingot and powder derived Be wire were tested, and composites of Be-epoxy were made with the ingot Be (because of availability). Uni-, bi-, and tri-directional composites were prepared and tested. No surface treatment besides cleaning the Be was needed to get good bonding. Composite failure was by plastic deformation. Both the 5-mil wire and the composites had low proportional limits, about 1/3 ultimate. Ingot wire had rougher surfaces and more large inclusions, higher strength, and lower elongation, compared to powder wire. High efficiencies of reinforcement and good quality composites were obtained; composites had stress-strain behavior like the Be wire. Modulus of both wire and composite were less than expected.

Fiber	Matrix
Be	Epoxy

270. Mechanical Behavior of Beryllium Wire Reinforced Plastic Composites

Schwartz, H. S., Schwartz, R. T.
AFML-TR-66-404, Jan. 1967
AFML

Fabrication of Be wire and composites is described, as are test methods and results. Laminates in two and three directions were made to check the interlaminar shear strength and effects of wire crossover. Tensile, compression, flexure, and shear tests were made, using straight-sided tensile bars and NOL rings. Composite efficiencies probably were in error due to problems in determining true % reinforcement. This was partly due to residual Ni clad on the Be. High efficiencies of reinforcement were obtained. Tensile samples tended to break near the grips; compression samples failed in buckling; interlaminar shear strength was good. Be composites undergo plastic deformation before failure. The quantative effects of this should be studied further. Good fiber-resin bonding was obtained by solvent cleaning the Be. Specific strength and modulus of the composites was very good. Tests of fatigue behavior and impact resistance would be useful.

Fiber	Matrix
Be	Epoxy

271. Characteristics of B Fibers and B-Fiber-Reinforced Plastic Composites

Schwartz, R. T., Schwartz, H. S.
***AIAA J.*, Vol. 5, No. 2, pp. 289–295, Feb. 1967**
AFML

Review is given on advantages of B fibers compared to glass with typical properties available now from fibers, and 1-, 2-, and 3-dimensional laminates as compared to glass fiber laminates. The 1- and 2-dimensional laminates have anisotropic strength and modulus, but 3-dimensional laminates are reasonably isotropic. Unidirectional laminates had 3% of the oriented strength in the transverse direction; bi-directional composites had less than 1/3 the strength when tested at 45°F. Fiber strength is reduced by the presence of a weaker WB_2 core, and by the presence of flaws in the fibers. There is little loss in strength after holding 1000 h in air at 500°F; after the same treatment at 700°F, there was a 90% loss in strength. Pretreatment of fibers is mentioned. Resins with higher modulus gave better composite properties. Problems were noted in gripping and testing the samples.

Fiber	Matrix
Be	Epoxy

272. Development of Structural Plastic Composites Incorporating New High Modulus Reinforcements

Schwartz, H. S., Spain, R. G.
In *Proceedings of the 8th AIAA Structures Conference*, pp. 65–79, Mar. 1967
AFML

Brief discussion of composites and reinforcements is given. They studied new fibers to determine fabrication and process parameters, evaluate composites, determine reinforcement efficiency, and investigate failure modes. Graphite composites were formed by laminating narrow uni-directional tapes; both uni-directional and bi-directional composites were tested. Results were encouraging. The C fibers with UTS of 0.3×10^6 psi and modulus of 50×10^6 would be competitive with B or glass fibers, but they are not available in production yet. Need for coupling agents is unknown, but boiling in H_2O has slight effect on composite strength. More work is needed in this area. SiC-epoxy was tested as narrow uni-directional tapes. There was considerable data scatter. Bi-directional 8-ply tapes also were tested, and gave 84–94% of Rule of Mixtures strengthening. Both tensile and compressive properties were equivalent to an equal vol% of B in epoxy, but the SiC is denser. The effects of various surface treatments were studied, and several useful ones were found. Interlaminar shear and flexure strength in particular were improved. Be wires 5 mil in diameter from ingot rod were laminated into uni-, bi-, and tri-directional plates and uni-directional rings, and were tested both for static mechanical properties and ballistic impact. Essentially all the expected strength of Be wires was realized in composites. Water boiling for 2 h did not affect their strength. Ballistic impact resistance was appreciably higher than glass-epoxy composites.

Fibers	Matrix
Graphite, Be, SiC	Epoxy

273. Applications of Reinforced Plastic Composites in Aircraft

Schwartz, H. S.

(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)

AFML

Plastics are used for complex pipes and tubes, wing and rudder sections, radomes, helicopter rotor blades, propellor and compressor blades, rocket motor cases, and gas storage bottles. Newer high-modulus fibers, such as B, have been used in rotor blades and as a 2/3-scale rudder section for the F-111. The Air Force is very interested in applying composites to aircraft and missile structures.

274. Statistical Theory of the Tensile Strength of Laminates

Scop, P. M.

(Paper G-3, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

National Research Corp., M.I.T.

Statistical theory for the strength of laminar composites is given. The physical model assumed is described, and mathematical descriptions are given. The effect of the glue bonding the layers is very great, since failure will occur only when all sheets are cracked within the same strip. This failure applies only to brittle sheets whose strength is governed by severe flaws. Laminate strength is given by flaw strength distribution, number of sheets, sheet dimensions, and glue shear strength, of which glue strength is the most important. Examples of the importance of each are given. Experimental results of glass-epoxy laminates are presented and found to agree very well with theory.

Fiber	Matrix
Glass	Epoxy

275. SiC Whiskers

Shaffer, P. T. B.

***Ceram, Age*, Vol. 82, pp. 46–50, May 1966**

Properties of SiC whiskers and their use are discussed. Modulus values are uncertain and range from 13 to 125×10^6 psi. A rough estimate of 70×10^6 was used. Reactivity with matrix may be a problem.

Fiber
SiC

276. A Feasibility Study on the Preparation of Boron Carbide Filaments, Whiskers, and Plate-Like Films

Shapiro, I.

AFML-TR-66-231, July 1966

Aerospace Chemical Systems

B_4C fibers were deposited on 10- to 20-mil W wire at 900–1025°C and on Mo wire at 850–1400°C. Deposits on Mo were uneven, thicker in the center where temperatures were higher, and with uneven lumps and modules. Deposits on W were more even, with less pronounced thickening in the center, and large overlapping sections on the wire ends. A number of cracks were seen in the coating. Sheets were deposited on Ta plates at about 975°C; they had a mosaic appearance. Pyrolysis in sealed 304 stainless tubes resulted in formation of Fe, Ni, and Mn borides; this method is not satisfactory for B_4C formation. Carborane-10 does give B_4C deposits, but the process is not developed.

Fiber
B_4C

277. Whisker Composite Technology

Shyne, J. J., Shaver, R. G.

(Paper B-6, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

General Technology Corp.

Major processing problems and some solutions are discussed. Whiskers tend to bunch up, and are very hard to align. Mixing in a blender gives good 2-dimensional mats; liquid infiltration into a capillary tube gives reasonable unidirectional alignment, but it is of limited practical use. Best results are obtained when whiskers are more than 3/8 in. long, and can be combed or carded. This method may induce residual stresses in the whiskers, and result in less than nominal composite strength. Metal matrix composites can be made by normal processing techniques without severely damaging the whiskers. Whisker vol % and *L/d* ratio must be adjusted to permit filling of voids around the whiskers, or increasing the fractions of whiskers will give reduced strengthening of the composite. Low efficiency glass-SiC whisker composites have been made by drawing down glass tubes containing whiskers. Electroforming Ni around SiC whiskers that were hand spun over a fine Ni wire produced composites which had an uneven, generally cylindrical surface, but with very good strength and modulus properties. In metals, whiskers can be used to control nucleation of alloys, reduce high temperature creep, and serve as a dispersion hardening medium.

Fibers	Matrices
SiC, Al_2O_3	Al, resin, Be, Fe, Ti, Ni

278. Interface Reactions in Composite Materials

Signorelli, R. A., Petrasek, D. W., Weeton, J. W.
NASA TM X-52168, 1966
NASA

This report is a proposed chapter on interfacial reactions in *Composite Materials* book, discusses interface reactions, and gives examples of experimental results. Reactions between fiber and matrix may increase, decrease, or leave unaffected the strengths of each phase or overall composite; any reaction is most likely to lower strengths. Reaction effects can be reduced by inserting a diffusion barrier on the fiber, alloying, adding a coating to the fiber to aid bonding and wetting, or changing fabrication methods. For W fibers in Cu, adding elements with low solubility in W and small atomic radii resulted in diffusion and recrystallization in W being greatest and loss of strength being greatest. Solid solubility of the alloy in W seemed more important than solubility of W in the matrix. Formation of two-phase zones at the interface or solid solution alloying did much less damage to the fiber. At elevated temperatures, fibers with recrystallized surfaces are weakened less than at room temperature. Use of powder processes or coating the fibers with thin diffusion barriers improved composite properties. Alloying the matrix before infiltrating ceramic fibers improves wetting and bonding, but also weakens fibers and reduces properties. Additions tend to segregate at the interface. Coating glass with Al before forming an Al matrix composite greatly increased composite strength. Control of interfacial reactions by alloying in the matrix, selection and careful control of fabrication processes, and proper selection and application of fiber coatings should permit maximum composite properties to be obtained.

Fibers	Matrices
W, glass, Mo, Al_2O_3	Cu, Cu alloy, Ni alloys, Ni, Ag, SS, Ti, Al

279. Carbon Fiber Composites

Simon, R., Prosen, S. P., Duffy, J.
***Nature*, Vol. 213, pp. 1113–1114, Mar. 18, 1967**
U.S. Naval Ordnance Lab

Preliminary data on C fibers indicate attractive strength and modulus values are possible, but the true value will be as a composite. There is an inverse relationship between fiber modulus and composite shear strength. The epoxy wets C fibers, but shear strength is low. Treating the C with HNO_3 increases shear strengths by 50%, but they still are too low. Fatigue resistance of C-epoxy is governed by type of test. The C fiber composites are resistant to H_2O degradation, and they will not need surface finishes as do glass-epoxy composites.

Fiber	Matrix
C	Epoxy

280. Development and Evaluation of the Diffusion Bonding Process as a Method to Produce Fibrous Reinforced Metal Matrix Composite Materials

Sinizer, D. I., Toy, A., Attridge, D. A.
IR-8-355(III), May 1965
North American Aviation

Be composites were made by orthogonal winding on Al and diffusion bonding. Up to 35% Be was added; UTS and modulus followed the Rule of Mixtures at all levels of reinforcement. Properties at both room temperature and 400 and 600°F were better than for controls. Compressive yield strengths up to 600°F were about the same as UTS. Off-axis UTS was reduced 10% or less at 30, 45, and 60°. Modulus was essentially the same. Failures were along the axis of filaments. No reaction between Al and Be was found after 1000 h at 600°F. The Ag plate on the Al, used to get better matrix-matrix bonding, did diffuse into the Al, but not much into the Be. Decreases in composite properties after 1000 h at elevated temperature are due to matrix softening, and can be recovered by heat-treatment of the matrix. Bending of the composite sheet around a 2T radius without cracking is feasible. Ti-B composites had lower UTS than controls at all % B and test temperatures up to 1000°F; modulus and compressive strengths were higher than controls up to 1000°F. Low properties of Ti-B composites were thought to be possibly due to EDM machining or embedding of the Al_2O_3 stopoff in the Ti. Changes to normally machined specimens showed no difference, and change to another stopoff reduced Ti embrittlement, but composite properties were still lower than the controls at all loadings and temperatures. Actual decreases in UTS with filament volume were found for Ti-B up to 1000°F. Filament alignment is good in both systems, and uniformity of spacing is fair to good. A study of the economics of Al-Be composites was made.

Fibers	Matrices
B, Be	Ti, Al

281. Development and Evaluation of the Diffusion Bonding Process as a Method to Produce Fibrous Reinforced Metal Matrix Composite Materials

Sinizer, D. I., Toy, A., Attridge, D. A.
IR-8-355(I), Nov. 1965
North American Aviation

Composites were formed by diffusion bonding filament wrapped foils in vacuum; filaments were wrapped over a mandril on a lathe. B-Ti layup was made by weaving B fibers over a Ti-6-4 foil on a special loom, followed by cleaning the mat. Properties of the filaments and matrix materials were determined, and initial bonding parameters were established.

Fibers	Matrices
B, Be	Ti, Al

282. Development and Evaluation of the Diffusion Bonding Process as a Method to Produce Fibrous Reinforced Metal Matrix Composite Materials

Sinizer, D. I., Toy, A., Attridge, D. A.
IR-8-355(II), Feb. 1966
North American Aviation

Bonding of Al and Be at 3600 psi for 4 h at 850–950°F gave improved modulus and UTS, and good Al-Al bonds at 10–17% Be. Properties of the composites were improved at 400 and 600°F also. Surface finish of the panels was improved by using hard anodized Al on the dies instead of plasma sprayed Al_2O_3. Close filament spacing in the Al-Be composites is possible while maintaining good matrix-matrix bonding. A brittle TiB_2 interface forms in the Ti-B composites; UTS for the reinforced composites was less than for controls made at the same time. Modulus was increased at all reinforcement levels. B-Ti bond cycles were 1550–1650°F, 3600 psi, for 3 to 27 h. A feasibility study which purports to show that the Ti-B composite fabrication process is economically feasible is given. Some of the cost assumptions for future years seem unrealistic, and the capital costs of one key process are taken as $1 million, although it is admitted that they may actually be as much as $10 million. Two processes are assumed to be practical at reasonable cost; however, they have not yet been demonstrated even on a laboratory scale. The Ti-B reaction during bonding is not considered. This economic study is considered very optimistic.

Fibers	Matrices
B, Be	Ti, Al

283. Some Tensile Characteristics of Ultrafine Be

Soltis, P. J.
(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)
U.S. Naval Air Engineering Center

Purpose of this program was to determine the size effect in Be wire. A 5-mil wire was chemically etched to 1-mil thickness. The wire had an unidentified brittle surface layer; removal of this layer considerably improved UTS and elongation. Modulus went from 30 million to 40 million after etching. Etching rates were very slow – 1 mil per day. The scale must be removed to get good etching. Some room temperature failures were ductile, but high temperature failures were brittle due to a surface scale. UTS peaked at 2 mil with about 0.22×10^6 psi. At 1- to 1.5-mil strength drops, again, due to uneven etching and some notches. At elevated temperatures, cyclical yielding was observed, with a maximum at 1000°F in the etched wire; there was a plateau in UTS at 500–850°F. Be brittleness is thought to be due to impurities. Control of impurities may permit us to get ductile wire which yields gradually. The cyclical yielding may be caused by strain aging or C diffusion.

Fiber
Be

284. Graphite Fiber Reinforced Composites

Spain, R. G., Schwartz, R. T.
(Paper A-3, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)
AFML

Tests of four types of graphite fibers were made, both on as-received fibers and in resin composites. The effect of surface finishes was not studied. Some difficulty was experienced in obtaining high fiber loadings. There was good transfer of fiber properties into the composite, 80–90% usually, sometimes over 100%. Mechanical properties of fibers now available in quantity are lower than desired, but better fibers are in the laboratory stage.

Fiber	Matrix
Graphite	Epoxy

285. Graphite Fiber Reinforced Composites

Spain, R. G.
AFML-TR-66-384, Jan. 1967
AFML

Report is a briefer version of paper given at AIAA meeting in Palm Springs, Mar. 1967. Several types of graphite fibers in yarn form were tested, with Thornel 25 receiving the most attention. Narrow unidirectional composites were formed and tested. Stress strain curves were linear up to fracture, densities were low, and good utilization of fiber strength was realized. Eight-ply bidirectional laminates were made and tested with less scatter than unidirectional samples. Water boiling for 2 h had little effect on strength. The need for coupling agents is not yet known. Higher volume content composites and higher strength fibers are needed before this need can be evaluated. More extensive evaluations including more types of static tests plus dynamic tests are needed, as are studies of the effect of graphite fiber anisotropy on the composite.

Fiber	Matrix
Graphite	Epoxy

286. Thermal Conductivities of Unidirectional Materials

Springer, G. S., Tsai, S. W.
***J. Compos. Mater.*, Vol. 1, No. 2, pp. 166–173, Apr. 1967**
Univ. of Michigan, Washington Univ.

Thermal conductivity of unidirectional composites was studied and equations derived for conductivity parallel and perpendicular to the filament. It was assumed that the composite is homogeneous, as are the matrix and fibers; that thermal contact resistance between fiber and matrix is negligible; and that fibers are in a periodic 2-dimensional array. A shear bonding analogy was used and was found reasonably good for less than 60-vol % fibers. Filament shape, vol% and thermal conductivity as compared to matrix conductivity were examined. An idealized thermal model also was used and was found satisfactory.

287. High-Temperature Polyimide Reinforced With Silica Fiber

Standage, A. E., Turner, W. N.
***J. Mater. Sci.*, Vol. 2, pp. 103–111, 1967**
Rolls Royce

Composites of glass-reinforced resins were prepared using aromatic polyimide polymers. Methods of fabrication and test are described. Laminates were made by wrapping tape on a drum, and cutting the sheet into strips. The strips were laminated and precured in the mold by pressing only, then cured under carefully controlled conditions. Testing was at room temperature and up to 400°C. The fabrication technique eliminated porosity in the composite. Poor fiber matrix bonding was obtained; tensile fractures were fibrous. Coupling agents probably would not be effective, because they have limited high temperature capability, and could not wet the silica glass surfaces.

Fiber	Matrix
Glass	Resin

288. Metal Matrix Composites: Air Force Materials Lab Report to NASA Research Advisory Committee on Materials

Standifer, L. R.
AFML Report to NASA, Jan. 1967
AFML

Air Force interest in metal-matrix composites is discussed, and programs getting under way to develop useful structures and applications are specified. The effects of secondary fabrication techniques, such as forming and joining, will be studied, as will the structural efficiency and properties of actual hardware. Active programs sponsored by AFML are listed and described briefly.

There are 29 programs, with 18 different organizations (primes only).

Fibers	Matrices
B, SiC, Be, W, TiB_2, SiB_4, Al_3Ni, B_4C	Ti, Ni, Al, Mg, Al-Si, Fe-Cr-Al, Ni-Cr, Co, Cu-Al, Co, Ag

289. Surface Finishes for B Filaments

Starks, D. F., Hough, R. L., Golf, L. C.

***AIAA J.*, Vol. 4, No. 10, pp. 1818–1821, Oct. 1966**

AFML

Vapor deposited protective coatings on B fiber were made by glow discharge. Ti, TiB_2, Si, SiC, and TiN were deposited from halides; Ni was deposited from the carbonyl; and 1 μm coatings were produced. B_4C, TiB_2, and Ni gave poorer oxidation resistance at 700°F than uncoated B; Si and SiC were much better, and TiN was slightly better. Si, SiC, and TiN also gave much better adhesion shear strengths in Epon 828 resin, both dry and after 2-day water soak, than did uncoated B or any of the other coatings. X-ray diffraction indicated that all but SiC coatings were amorphous even after aging at 700°F in air.

Fiber	Matrix
B	Epon 828

290. Fiber Reinforced Composite Metals

Steg, L., Rosen, B. W., Sutton, W. H.

***Israel J. Tech.*, Vol. 5, No. 1–2, pp. 71–98, Feb. 1967**

General Electric Space Sciences Lab

Survey of composite technology is given. Functions of fiber and matrix, i.e., load bearing member, binder, and protection for fiber, are mentioned. Properties and potential are described. Fiber characteristics and preparation methods are briefly covered, including advantages, disadvantages, and problem areas of various methods. Specific problem areas, such as reactions between the C or B fibers and metal matrices, are noted. Whiskers offer the greatest potential from the viewpoint of highest strength and modulus, but there are many fabrication and handling problems to overcome. An increasing effort is being directed towards metal-matrix composites. A number of advanced reinforcement materials are becoming available, and are being applied to structure. Much work on the mechanics of composites is being done, but there is room for more and for developing better understanding of the micro-mechanics of behavior. A rather extensive discussion of the micro-mechanics of composites is given, including various approaches to elastic behavior, effects of geometry, relative volume fractions, viscoelasticity, internal stress field, and failure mechanisms. It is important to consider the statistical distribution of fiber strengths and lengths when studying failure modes and strengthening efficiency. Structural evaluations are conducted on a structural efficiency basis by using laminate analysis to couple from constitutive relations to specific structural applications. Examples given are boost vehicle cases, box beams, and wing boxes; comparisons are made between various metals and composites. If properly designed, composite structures offer appreciable weight savings over metals. Understanding of structural behavior is still rather meager, and more work must be done on materials properties, failure mechanics, plastic behavior, wave propagation in heterogeneous media, thermostructural response, energy absorption, and design consideration, particularly joints.

291. Properties Determination and Process Control of Boron Filament Composites Using Non-destructive Test Methods

Stinebring, R. C., Zurbrick, J. R.

(Paper H-4, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

AVCO Corp.

Nondestructive tests on composites have been made and correlated successfully with test results. They have been used to find material properties, and to monitor and adjust material production. Microradiography and stereoradiography are used to eliminate fiber breaks and crossovers in composites. Dye penetrant shows delaminations caused by cutting. Ultrasonic velocity correlates well with interlaminar shear strength. Microwave absorption is affected by moisture content. Filament diameter is monitored continuously by passing the fiber between parallel plate capacitors. These various tests are capable of detecting sample variations, identifying out of control process steps, monitoring the process and detecting the effects of surface treatment and water pickup.

Fiber	Matrix
B	Epoxy

292. Development of Ultra-High Strength, Low Density, Aluminum Plate Composites

Sumner, E. V.

NASA CR-62395, Nov. 1964

Harvey Aluminum

Composites of Al were prepared by hot extruding and hot rolling; obtained appreciable strengthtening. Also, report is abstracted in AFML-TR-65-207 (see entry No. 144).

Fiber	Matrix
Steel	Al

293. Development of Ultra-High Strength, Low Density, Aluminum Plate

Sumner, E. V.
HA 2208, Jan. 1966
Harvey Aluminum

Wires used to reinforce Al are those from Nat-Std. referenced (entry No. 13). Sheets were made by extrusion, pressing, and rolling. Best results were obtained by powder pressing the composite, and hot rolling. Best Al-Al bonding was obtained by diffusion bonding. Formation of alloys between wire and matrix lowered composite strengths 40%. Heat treating or cold rolling to raise matrix strength appreciably increased composite strengths. At 27:1 L/d, UTS was determined by wire UTS. Notched tensile and Charpy data also were obtained. At $-320°F$, impact strength was up 50%.

Fibers	Matrix
AFC77, 302 SS, 355 SS, steel	Al

294. Development of Ultra-High Strength, Low Density, Metal Matrix Composites

Sumner, E. V.
HA 2236, Apr. 14, 1966
Harvey Aluminum

Steel-Al composite plates were prepared up to 1 × 8 ft and 0.035- and 1/4-in. thick. Also, smaller samples between 0.023 and 3/4 in. were made and tested. The large plates showed some bunching of the wires during fabrication; it was reduced by putting 0.003-in. shims between the wires during layup. Al shims were too soft, so steel shims were used with better results. Plates were made by diffusion bonding, pressing, and rolling. Cold and hot rolling after diffusion bonding was found to increase UTS. Up to 4% cold reduction was used. Al-Al diffusion bonding was best at 900°F and 14,000 psi. Alloying between wire and Al reduced UTS by 40%. Hot rolling up to 10% improved flatness, while cold rolling had more effect on properties. Electron beam welding was unsuccessful; joints could be made by etching the Al from the wires, cleaning the wires, and filling with 95% Zn, 4% Al, and 1% Cu by vacuum infiltration. A 54% joint efficiency was obtained. Heat treatment of the matrix to increase its strength also increased the composite strength. Notched tensile samples with $K_t = 6.2–7.2$ had slightly less UTS than unnotched ones. Charpy values at room temperature, $-97°F$, and $-320°F$ were comparable to the matrix Al. Both B and Be fibers were tested in Al matrix, and were found to have almost full reinforcement efficiency. Also, 49-vol% fibers were used.

Fibers	Matrix
NS 355, Be, B	Al

295. Development of Ultra-High Strength, Low Density, Al Plate Composites— Final Report

Sumner, E. V.
HA 2263, July 1966
Harvey Aluminum

The composites followed the Rule of Mixtures; efficiencies were nearly 100% for the diffusion bonded plates. Residual stresses in the matrix were found to affect the stress-strain curve appreciably. Joints made by liquid infiltration were only 54% efficient; to get high efficiency joints, the joint design must be part of the original composite fabrication. Plates as thin as 0.023 in. and as thick as 1/4 in. were prepared in sizes up to 1 × 8 ft. Wire distribution in 1 × 2 ft plates was much more uniform than in the longer plates, but was satisfactory in each case. Charpy samples had about the same impact strength as parent alloy; notch-unnotched strength ratio was 95% for $K_t = 6$. The Al was degreased, caustic etched, and wire brushed just before the steel wires were added, then they were diffusion bonded inside a 1020 steel can filled with A, and then evacuated. Pressure was 14,000 psi at 900°F for 20 min, followed by 10% reduction at 700°F, solution heat treat, and roll 1% age 10 h at 375°F. Formation of an Al-steel alloy reduced composite strength 40%. Electron beam welding and use of steel inserts did not provide good joints. Bibliography of 58 items with brief abstracts of each is included. Course notes from UCLA short course, Mechanical Behavior of Fibrous Composite Materials by H. T. Corten, given Apr. 26–30, 1965, also are included.

Fibers	Matrices
Steel, NS355, NS302, AFC 77	Al-2024, 1100, 2219,
B, Be	3003, 5052, 5456,
	7075, 7178

296. Effect of Residual Stress and Cold Work on the Load Deformation Curve of Al Matrix Steel Fiber Composites

Sumner, E. V.

(Paper F-2, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Harvey Aluminum

The effect of strain, resulting from thermal stress and 2% cold work on the load deformation curve, was studied. The composite of 25-vol% 9-mil wires was diffusion bonded at 900°F, 6 ton/in.^{2} for 1/2 h. The four stages of deformation are elastic-elastic, elastic-plastic, plastic-plastic, and failure. The theoretical composite shows all four stages; annealing gives residual tension in the matrix from different coefficients of expansion, and almost totally suppresses stage 1; heat treating and aging also suppresses stage 1, and expands stages 2 and 3; heat treating and cold working expands stage 1, shortens stage 2, and changes the slope. Residual stress can have an appreciable effect on primary and secondary modulus and primary yield strength. Composite tensile strength is increased due to matrix strengthening by heat treating and working. When the fibers are brittle, putting the matrix in compression can cause loss of composite strength.

Fiber	Matrix
NS355 steel	2024 Al

297. Development of Composite Structural Materials for Space Vehicles Applications

Sutton, W. H.

***ARS J.*, Vol. 31, No. 4, pp. 593–600, Apr. 1962 (GE Reprint No. 53)**

General Electric Space Sciences Lab

This is an early paper in which the idea of using Al_2O_3 whiskers is presented and discussed. Rule of Mixtures strengthening is predicted, and greatly improved strength to density and high temperature properties are predicted. Problems mentioned include obtaining stress transfer between fiber and matrix, obtaining good fiber-matrix bonding, selecting only strong whiskers, and fabricating composites to get good orientation and bonding. Composites were made up with to 9-vol% ZrO_2 without reinforcement. The 17% fibers did increase composite strength. In most cases, too little fiber addition was made to strengthen the composite appreciably. High temperature strengthening is also predicted.

Fibers	Matrix
Al_2O_3, ZrO_2, glass	Al

298. Investigation of Metal-Whisker Composites

Sutton, W. H.

GE Reprint No. 54, June 1962

General Electric Space Sciences Lab

Early discussion of the concept of whisker reinforcement, some of the problems involved, and results of using Al_2O_3 in Ag are presented. Fabrication was by vacuum infiltration. Use of Al and Al alloys was unsuccessful because of matrix metal oxidation. At 19% fibers, there was little strengthening, but at 32% fibers, some strengthening was noted. Effective reinforcement by whiskers was demonstrated. Metallizing the whiskers by sputtering Ni onto them gave much better bonding and composite strengths. The whiskers were not attacked by the molten Ag. It is difficult to get packing densities of 50% or more, particularly if interfiber contact is to be avoided. Other problems noted include obtaining a supply of high strength whiskers of sufficient L/d, maintaining whisker strength during fabrication, getting a uniform, parallel whisker distribution, and wetting and bonding to the whiskers.

Fiber	Matrices
Al_2O_3	Ag, Al

299. Sapphire Whiskers for Structural Reinforcement

Sutton, W. H.

GE Reprint No. 55, Dec. 1962

General Electric Space Sciences Lab

Growth and properties of Al_2O_3 whiskers is discussed. Effects of growth variables such as direction of gas flow and supersaturation ratios is given. Comparative strengths of various fibers and whiskers is given. Growth of desired whisker types can be controlled and reproduced; continuous whisker growth appears feasible; and high strength is a function of diameter and surface perfection. Four types of whiskers (hexagonal and rhombohedral with 3 different l/h ratios) were tested. Strengths varied from 0.900×10^6 to 2.8×10^6 psi. The whiskers were successfully used to reinforce metals both at room and high temperatures, but more extensive study is needed before the feasibility and amount of strengthening of plastics can be determined. Other whisker materials, such as B, Be, B_4C, SiC, and C, are suggested as theoretically better than Al_2O_3 in strength or modulus.

Fiber	Matrix
Al_2O_3	Ag

300. Development of High-Strength, Heat-Resistant Alloys by Whisker Reinforcement

Sutton, W. H., Chorne, J.

***Mater. Eng. Quart.*, Vol. 3, No. 1, Feb. 1963, (Also GE Reprint No. 193)**

General Electric Space Sciences Lab

Paper presents concept of whisker reinforcement of Ag to obtain better high temperature strengths, using Ag as a model system. Factors affecting fiber strengthening are discussed, but not extensively elaborated upon. Whisker growth was by vapor deposition; composite fabrication was by vacuum infiltration of Al_2O_3 whiskers which had been coated with Ni by vapor plating. Comparisons of dispersion strengthened and whisker reinformed metals are made up to 1400°F. Whisker reinforced composites should have better UTS and stress rupture strength, particularly at higher temperatures. The improvement in high temperature strength of Ag demonstrates that this is so.

Fiber	Matrix
Al_2O_3	Ag

301. Investigation of Bonding in Oxide-Fiber (Whisker) Reinforced Metals

Sutton, W. H.

AMRA-CR 63-01/5, Sept. 1963 (AD-427056)

General Electric Space Sciences Lab

Additions of 1-at.% Ti, Zr, and Cr aided wetting between fiber and substrate. Ti was more active than Cr. Al, Cu, and In had little effect on wetting and bonding.

Fiber	Matrices
Al_2O_3	Ni, Ni alloys

302. Whisker-Reinforced Plastics for Space Applications

Sutton, W. H., Rosen, B. W., Flom, D. G.

GE Reprint No. 257, May 1964

General Electric Space Sciences Lab

Ceramic fibers appear more attractive than metals as reinforcements because of higher strengths and moduli, less handling damage, and oxidation. Mechanics of whisker reinforcement are discussed. Stresses are highest at fiber discontinuities and ends. Epoxy selection, interface properties, and coupling agents are mentioned. Experimental data indicated appreciable reinforcement can be obtained. Many problems remain to be solved.

Fibers	Matrix
Al_2O_3, B_4C, SiC	Resins

303. Wetting and Adherence of Ni/Ni Alloys to Sapphire

Sutton, W. H.

GE Reprint No. 265, June 1964

General Electric Space Sciences Lab

The effects of additions of impurities to Ni on wetting of Al_2O_3 are studied. Properly controlled chemical reactions appear to give best interfacial bonding. Excessive reactions give poorer bonding. Al, In, and Cu in small amounts did not segregate at the interface, or increase adherence. Trace amounts of Ti, Zr, and Cr segregate at the interface and lower contact angles between Ni and Al_2O_3. Ti was most active. Ti and Zr caused high internal stresses at the interface on cooling, but lowering Ti content from 1 at.% to 0.01 at.% increases shear strength 300%. The 1-at.% Cr seems to give highest shear strength. Ni with 31 ppm impurities had better wetting and shear strengths than Ni with 15 ppm. Absorption of additions at the interface is not related to °F of oxide formation, as had been expected. Chemical reaction with Al_2O_3 seems to govern wetting.

Fiber
Al_2O_3

304. Investigation of Bonding in Oxide-Fiber (Whisker) Reinforced Metals

Sutton, W. H.

AMRA-63-01/2, Jan. 1965

General Electric Space Sciences Lab

Additions of Ti, Zr, and Cr were found to preferentially segregate to the metal-oxide interface and promote wetting by attacking the oxide surface. Continuation of the work in AMRA 65-01/3 showed that wetting of the same type occurred with as little as 20 ppm Cr and Ti. The greatest bond strength was for Cr, and the least with Zr.

Fiber	Matrix
Al_2O_3	Ni

305. Potential of Oxide-Fiber Reinforced Metals

Sutton, W. H., Chorne, J.
GE Reprint No. 335, Jan. 1965
General Electric Space Sciences Lab

Paper reviews and summarizes advantages and problems of reinforcing metals with oxide fibers in terms of strengthening principles, types and strengths of available fibers, practical limitations, and properties of experimental composites. Considerable development is still needed, but considerable improvement in materials properties, particularly at high temperatures is possible. Improved yield strengths and moduli also are possible, as are lower densities and higher strength-to-weight ratios. Utilization of fiber composites requires more understanding of their properties and of the technology required. This includes fiber growth and processing, handling and coating, techniques for composite fabrication, particularly metal matrix joining methods, and understanding physical and mechanical response of the materials. Experimental data for several systems are given. Metal-metal composites effectively utilize the reinforcement, and are relatively insensitive to process variables. Problem areas such as weakening caused by direct fiber-fiber contact, inadequate wetting and bonding to the matrix, and sensitivity of ceramic fibers to process variations are discussed.

Fibers	Matrices
Glass, Al_2O_3, steel, W, SiO_2, Si_3N_4	Al, Ag, Cu, Pb, Ni, nichrome, Ni-Pd

306. Investigation of Bonding in Oxide-Fiber (Whisker) Reinforced Metals

Sutton, W. H.
AMRA-CR-65-01/4, Aug. 1965 (AD-630359)
General Electric Space Sciences Lab

When Ni samples with Cr, Ti, and Zr additions were heated in H_2, the relative shear strength of Ni-Cr alloys was lowest and Ni-Ti alloys was highest, opposite to the results without H_2 treatment. Ti and Zr alloys had higher shear strength as alloy level decreased. Characteristic fracture occurred for each addition. Additive concentration and heat treatment must be optimized to get maximum wetting and bonding with minimum weakening of the substrate.

Fiber	Matrix
Al_2O_3	Ni

307. Role of Interfacially Active Metals in the Apparent Adherence of Nickel to Sapphire

Sutton, W. H., Feingold, E.
GE Reprint No. 358, Aug. 1965
General Electric Space Sciences Lab

Study of the effect of adding 1 at.% of various interfacially active metals (Cr, Ti, and Zr) on the wetting and adherence of Ni to Al_2O_3 to improve interfacial bond strength in composites is given. Testing was by use of doped sesile drops to determine contact angle and bond strength and failure mode in a simple shear test. Use of pure Ni and Cr-doped Ni gave best adherence, while Ti and Zr attacked the substrate and gave weaker bonds. It is suggested that the active elements segregate at the interface, and preferentially react with and diffuse into the Al_2O_3. First, bond strength increases due to improved wetting and bond and, then, decreases as the substrate is attacked and weakened, or as harmful interface reaction products are formed. Interfacial reactions must be limited to prevent excess attack of the substrate. This limitation can be done by control of composition and amount of additive, bonding time, temperature, and atmosphere.

308. Role of the Interface in Metal-Ceramic (Whisker) Composites

Sutton, W. H.
AD-477422, Feb. 1966
General Electric Space Sciences Lab

The importance of the interface in stress transfer between fiber and matrix is discussed and illustrated with data from the Ag-Al_2O_3 system. The interface provides the common boundary between fiber and matrix, and is an important factor is composite performance. Reactions between matrix and fiber must be sufficient to wet the fiber without attacking it and weakening it. In liquid infiltration of fibers, the surface energy of solid-vapor must exceed solid-liquid and liquid-vapor surface energies for adequate wetting. This may be achieved by plating the fiber with another metal, as Ni on Al_2O_3. Wetting should be uniform along the fiber length to permit effective use of the entire fiber and to obtain large L/d ratios. The L/d needed to develop 95% of the fiber strength in the composite is about 10× the critical L/d, if there is no strain hardening in the matrix. If considerable matrix hardening occurs, this can be reduced by 2–10×. The weaker the matrix, the larger L/d is critical. Interfacial bonds should be stronger than the matrix shear stress and than any residual stresses caused by fabrication operations.

Fibers	Matrices
Al_2O_3, SiC, Si_3N_4	Ag, Cu, Ni

309. Review of Current Developments in New Refractory Fibers and Their Utilization as High Temperature Reinforcements

Sutton, W. H., Rauch, H. W., Sr.
(Paper B-1, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)
General Electric Space Sciences Lab

Paper surveyed refractory fibers and their use as composites and discussed results of literature and patent search, questionnaires, and plant visits here and abroad. Parameters influencing fiber strength, composite properties, and fiber preparation and properties are noted. Fiber requirements for efficient reinforcement in high temperature matrices are covered. Mechanical properties of composites at room and high temperatures are given and compared. Critical problem areas are identified as fiber handling and orientation, composite fabrication, bonding, strength retention at high temperatures, and cost. Much more emphasis is being put on continuous fibers than short ones, and on ceramics and carbon than on metals. The advantages and disadvantages of various fiber types are tabulated.

Fibers	Matrices
All	All

310. Fabrication of Beryllium Oxide Radome by Pyroletic Deposition

Taebel, W. A., Hoekstra, G.
AD-638219, June 1965
National Beryllia

Report describes chemical vapor deposition techniques designed mainly to produce massive BeO shapes, but also short fibers and whiskers. Composition of the reaction chamber atmosphere was found to be critical. Reduction of furnace components such as Al_2O_3 gave products which prematurely oxidized the Be metal, and prevented fiber formation. Fibers were formed at 1650°C on a graphite substrate, and at 1300°C on a 310 SS substrate. Each succeeding run gave lower yields; attack by liberated H_2O is one possible cause. The amount of oxidant in the chamber must be closely controlled within a narrow range which has not yet been defined.

Fiber
BeO

311. Summary of Composites Research in Japan

Tao, T.
(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)
Nagoya Aircraft Works

Summary of composites research in Japan is given. Most of the effort is in glass fiber-reinforced plastics (FRP). They are used in the fabrication of airplane parts, sounding rockets, and pressure vessels, including rocket nozzles, tanks, and rocket chambers. Fundamental research includes a new ISAS test ring for hoop strength evaluation, investigation of mechanical joints and joint strength, effect of bonding between fibers and matrix and between laminate layers, effect of cyclic fatigue where 80% and 90% preloading of the composite is applied, and general fatigue strength. It was found that preloading above 80% of the ultimate is harmful, that rigidity in fatigue decreases as frequency increases, and that temperature increases proportional to the stress range and rev/min. Below 3000 rev/min, fatigue strength at 10×10^6 cycles was unaffected by rev/min, and air cooling was ineffective; below 1×10^6 cycles, fatigue strength was affected by rev/min, and air cooling helped. Most FRP had fatigue limits above 10×10^6 cycles. Effects of winding angle on the properties of filament wound structures have been studied, using cylinders. Fracture toughness studies showed the reinforced material is superior to unreinforced material. Photoelastic studies have been made on flat plates with holes. Polyimide resins and C and graphite cloths were studied as possible new composite materials. They also are interested in B.

Fiber	Matrix
Glass	Resins

312. Mechanics of Strengthening and Fracture in Composite Materials

Tetelman, A. S.
NASA CR-60123, Nov. 1964
Stanford Univ.

Report presents the study to determine if critical stress or strain concentration causes crack neucleation when brittle particles are dispersed in ductile matrix. Fine holes drilled in plates show little effect when hole size is about equal to grain size.

Fiber	Matrix
TiC	Ni-Mo

313. Mechanisms of Strengthening and Fracture in Composite Materials

Tetelman, A. S.

NASA CR-77451, July 1966 (N66-35175)

Stanford Univ.

Progress in four programs generally in the composite area is described. Spherodized quenched and tempered steels are treated as composite systems. The effect of particle spacing on fracture behavior is being studied. TiC-Mo_2C-Ni composites were made by pressing and sintering at 2500–2560°F, and preliminary tests were started. Theoretical aspects of fracture are being studied from the viewpoint of pileup of screw and edge dislocations. The effect of drilling small holes in Fe-3%Si alloys is under investigation. The C content affects the reduction in ductile-brittle transition temperature in Charpy samples; it is most effective at 0.1–0.25%C.

314. Vertol to Build, Flight Test B Filament

Thomas, B. K., Jr.

***Aviat. Week*, pp. 40–46, Aug. 29, 1966**

Vertol will build helicopter blades with a combination of glass and B fibers in epoxy, with Al honeycomb core. Because there are problems with natural frequency, bending moments, and stiffness, it will take several years of iterative effort to solve all the problems. Wide use of B blades as standard items is expected in 5 years. General Dynamics/Ft. Worth is using B-epoxy on wing boxes and rudders for F-111.

Fibers	Matrix
B, glass	Epoxy

315. Development and Evaluation of the Diffusion Bonding Process as a Method to Produce Fibrous Reinforced Metal Matrix Composite Materials

Toy, A., Atteridge, D. G., Sinizer, D. I.

AFML-TR-66-350, Nov. 1966

North American Aviation

Plates up to 12 × 18 in. were formed by diffusion bonding woven mats of fibers or winding fibers over matrix plates. Up to 20 layers of B-Al, 40 layers of Be in Al, 20 layers of B in Ti, and 21 layers of SiC in Ti were formed. Up to 35-vol% Be in Al plates were made successfully; tensile strength and modulus increased linearly with fiber content. Elevated temperature properties were significantly improved, as were stress-rupture life and fatigue strength. Corrosion resistance in high humidity and salt spray was equal or better than bare Al. Testing at angles to fiber direction showed little decrease in strength. Compressive yield strength is about the same as tensile strength up to 600°F, and is superior to unreinforced Al. After 1000 h at 600°F, there was no reaction between Al and Be, but the matrix was annealed; matrix strength could be recovered by heat-treatment. Composite plates can be formed at room temperature by hydroforming and brake forming; bending is more critical in the fiber direction. The same process was successful in forming Al-B plates up to 9 × 12 in. with 24-vol% fibers. Both room temperature and 500°F tensile and compressive properties were improved significantly, as was fatigue strength. Tests of extracted B and Be fibers showed no loss of strength due to fabrication. Ti-B had decreasing tensile strength with increasing fiber content due to reaction between B and Ti. Fiber strength was reduced by 3–4 times, thus, the weaker fibers were less than the critical reinforcement volume; weakening was noted at all temperatures up to 1000°F. Compressive yield strength and modulus did increase with increasing fiber content up to 1000°F. SiC in Ti did not show degradation of fibers due to processing. Little increase in yield or tensile strength was noted up to 18-vol% reinforcement. Samples up to 4 × 6 in. were made. Lack of reinforcement was attributed to failure to exceed the critical volume fraction reinforcement. Moduli were increased with added fiber content. The Be-Al system is considered sufficiently advanced for scale-up at this time; joining is not considered to be a real problem. Ti-B composites might be prepared satisfactorily by lower bonding temperatures and higher pressures. Major economic problem in Al-matrix composite costs is the Ag plated on the Al to aid bonding. Major cost limit for Ti-B composites is the raw materials.

Fibers	Matrices
B, Be, SiC	Al, Ti, Ti-6Al-4V

316. Mechanics of Composite Materials, Part I: Introduction

Tsai, S. W.

AFML-TR-66-149, Part I, June 1966

Washington Univ.

Principles of mechanics and their applications to understanding the behavior of composites are discussed. The behavior model used depends on whether a macroscopic or microscopic viewpoint is taken. Macromechanics assumes that the composite is homogeneous; micromechanics assumes that it is heterogeneous. The two approaches may be related mathematically. Stress is defined as a tensor; scalar, vector, and tensor are described. Equations governing macroscopic stress-strain behavior of orthotropic composites are given and explained. Examples of the effect of fiber orientation on composite strength of B and glass reinforced composites are given, and the effects were found to agree with experimental data; this confirms that the assumptions are correct. Elastic moduli of laminated composites can be derived theoretically. Composite strength is more complex, and requires a micromechanical approach, including anisotropy and behavior in complex stress fields. Equations for maximum tensile strength as a function of fiber orientation are given for uniaxial tensile strength, maximum stress, and maximum strain. Theory of elasticity is used for micromechanics analysis, since the assumption of boundary conditions can be expressed precisely and mathematically. Equations for axial and transverse properties are derived and the assumptions are discussed. Modulus values can be predicted with reasonable accuracy, but transverse strength and shear strength cannot be predicted as yet. Equations which describe homogeneous materials cannot be applied indiscriminately to composites. Test methods are noted; standardization of test methods should not precede understanding what properties should be evaluated. Understanding mechanics will aid testing and evaluation. Samples should be tested under homogeneous stress and strain; short samples should be avoided. Introduction of defects makes data reduction more complex. Intrinsic and extrinsic properties should be determined separately.

Fibers	Matrix
B, glass	Epoxy

317. Mechanics of Composite Materials, Part II: Theoretical Aspects

Tsai, S. W.
AFML-TR-66-149, Part II, Nov. 1966
Washington Univ.

Mathematical treatment of principles of mechanics applicable to composites is given. Elastic modulus of an anisotropic body is a fourth rank tensor. Tensor analysis is described with emphasis on indexical notation and operations. Equations are derived and examples are given. Plane stress and strain, elastic moduli of laminates, strength of unidirectional composites, material symmetries, and engineering constants are covered.

318. A Test Method for the Determination of Shear Modulus and Shear Strength

Tsai, S. W.
AFML-TR-66-372, Jan. 1967
Washington Univ.

An experimental method is given for determining shear modulus and shear strength, using simple specimens rather than torsion tubes. Flat plates are used for modulus, columns for compression, and dog bones for tension. Mathematical derivations of elastic equations are given and discussed. Uniaxial alignment of fibers at 0, 45, and 90° in the sample is sufficient to establish composite properties. It is recommended that three-element strain rosettes be used on the samples, and that both tensile and compressive data be obtained to determine the variability of shear strength.

319. Survey of Environmental Interactions

Tsai, S. W.
(Presented at the International Conference on Mechanics of Composite Materials, Philadelphia, Pa., May 8–10, 1967)
Washington Univ.

We are unable to predict the properties of composites in adverse environments, under different loads, and over extended periods. The mechanisms acting under different conditions are important, and should be studied and understood. Properties, such as anisotropy and viscoelastic behavior, are not understood. Confidence must be developed in the composite reliability, and the nature and extent of degradation in different environments should be predictable. Glass fibers are attacked by water in a way analogous to stress corrosion; the fiber-matrix interface also is attacked by the water. Shell Chemical has shown that rapid passage of water along the interface can occur. Other environmental effects are time and temperature, and moisture and temperature. The effects of environmental attack on longitudinal and transverse properties cannot be compared accurately. Some attack mechanisms are void formation in the resin and plasticizing the matrix. Modulus values of glass-epoxy systems can follow the curves for good adhesion even when no adhesion was present, but for the same systems, UTS was greatly affected by adhesion, increasing with increasing adhesion. Transverse UTS is greatly affected by void content; for B-epoxy, UTS decreases by about 1/2 with 14% voids; longitudinal strength was not affected. Exposure to 50% relative humidity and to water did not indicate any improvement when the glass fibers were thinly coated with resin or coupler. There is still no really reasonable mathematical treatment of viscoelasticity.

320. Strength Properties of Fiber-Reinforced Composites

Vasilos, T., Wolff, E. G.
***J. Metals*, Vol. 18, No. 5, pp. 583–592, May 1966**
AVCO Corp.

This review article presents the various work done by the authors in the systems metal fiber-metal matrix, ceramic fiber-metal matrix, and metal fibers ceramic matrix. It covers theories of strengthening, results of tests on a number of systems, and 69 references.

Fibers	Matrices
All	All

321. Review of Recent Developments—Fiber Reinforced Metals

Wagner, H. J.
DMIC Letter, June 24, 1966
Clevite

Processing method greatly influenced reinforcement obtained. The 23-vol% 10-mil wire increased high temperature strength 2–3.5 times at 1800 and 2000°F, but hot rolling reduced strengthening to 1.5–2.5 times. Extruded Waspalloy with 30% W was unstrengthened.

Fiber	Matrices
W	Waspalloy, Hastelloy C, Ti-6Al-4V

322. Review of Recent Developments—Fiber Reinforced Metals

Wagner, H. J.
DMIC Letter, June 24, 1966
General Technology Corp.

Electroformed Ni around 3-mil SiC fibers gave 0.106×10^6 psi for 40-vol% fibers. Cr-coated fibers did not react up to 1200°C. No reaction was found between B and Mg or Al to near matrix melting points. Some indication of reaction occurred with molten Al, but not with molten Mg. Diffusion barrier in contact with Ni is needed. Al and Ag reinforced with B may be usable for short times up to 900°C.

Fibers	Matrices
SiC, B	Ni, Al, Mg, Ti

323. Review of Recent Developments—Fiber Reinforced Metals

Wagner, H. J.
DMIC Letter, June 24, 1966
North American Aviation

Composites were made by solid state diffusion bonding at 900°F, 4 h, 3600 psi. The 10% Be wires gave appreciable increases in modulus with lesser increases in yield and tensile. B in Ti degraded while processing, and was contaminated during EDM machining.

Fibers	Matrices
B, Be	Al, Ti

324. Metal Filaments for Composite Systems

Weber, H. H.
(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)
Brunswick

To weave wires for textile-like fabrics, 6- to 8-μm wires are needed. A proprietary multi-end process is used to form 12-μm wire for less than $50/lb. There are both process and material dependent variables; strength decreased as size decreased below 25 μm and as gauge length increased; length effect are noted for both 7- and 12-μm wires. Cleaner metal showed a greater gauge length effect. Defects were both inclusions and instabilities (thin sections) in the wire. They are also drawing Al alloys, Ni, and refractory alloys.

Fibers
SS, Ni, Al alloys

325. Fiber-Metal Composites

Weeton, J. W., Signorelli, R. A.
NASA TM X-52123, Aug. 1965 (Also presented at the 12th Sagamore Army Materials Research Conference, Roquette Lake, N.Y., Aug. 24–27, 1965)
NASA

Rule of Mixtures relationships for composites are discussed. Deviations were considered in terms of synergistic reactions, damage, and intermediate amounts of damage or reinforcement. Development and potential of composites were reviewed. Tabulation of W reinforcement, graphs of ultimate tensile, fiber deformation, modulus, and yield strength of composites were given. Potential strengths of fiber-superalloy composites, superalloys, and dispersion hardened alloys were compared. Tensile strengths of amorphous and polycrystalline fibers were graphed.

Fiber	Matrices
W	Cu, Ag, W, superalloys

326. Fiber-Metal Composite Materials

Weeton, J. W., Signorelli, R. A.
NASA TN D-3530, Aug. 1966
Lewis Research Center

Composite strengthening follows a Rule of Mixtures behavior at room and elevated temperatures. A number of systems were studied and evaluated. There must be sufficient fiber overlap to prevent shear failures and to allow stress transfer to fibers of more than critical length. Slight misalignment increases composite strength, but more than about 10° causes rapid drop in tensile strength. Four stages of fracture were identified: both fiber and matrix elastic; fiber elastic, matrix plastic; both plastic; and initial fiber failures. For W in Cu, minimum critical L/d was 8; for stainless in Al when L/d was 830, strength was essentially that of an infinite fiber. As temperature increases, L/d required increases. Additions to matrix which give brittle interfaces, as when 33% Zr was added to Cu with W wire reinforcement, reduced composite strength. Addition of 10% Ni caused recrystallization of the W wire, and decreased composite strength. Co had the same effect. The recrystallized surface probably caused local notches in the W. Stress-rupture strength and time to failure followed Rule of Mixtures curves. Reactions between fiber and matrix can cause lowered strength and high stresses in composite, as for W in Cb-Ni. This reaction can be minimized by changing fabrication methods. Adding 22–23% W to Nichrome, L605 or Co decreased RT strength, but increased high temperature strength. Co-extrusion of W and various oxides gave fibers with better interfacial bonds and improved stress-rupture strengths, even at 8–10 vol % reinforcement. L/d ranged from 12.7 to 23.3.

Fibers	Matrices
W, Al, Steel, Mo, ZrO_2, YO_2, HfO_2, ThO, HfN, SS	Cu, Cu-Ni, Ag, Cu-Co, Cu-Zr, Co, Ti, Ti-Al-V, Mo, Cb-Ni, W, Al, Nichrome, Cb, L605

327. 1968 Is Target Date for Air Force to Prove Worth of Composites

Wessling, J.
***Metalworking News*, Vol. 8, No. 367, pp. 9 and 20–23, Aug. 7, 1967**

Interviews with George Peterson and J. A. Herzog of AFML on programs and progress in composites are given. Applications are being emphasized; the goal of flight testing composite structures should be met by late 1968. This testing will demonstrate the utility and advantages of composites, and will allow designers much more flexibility and scope. Efforts are more in plastic matrices for more current applications, but much work on metal matrices is being done. Developmental work includes interface studies and compatibility, whisker incorporation in various matrices, and development of whiskers. Current and recently concluded Air Force sponsored programs on composites are listed and described very briefly, together with their current status; 16 programs with a budget of about $8 million are listed.

Fibers	Matrices
B, SiC, B_4C, TiB_2, Al_2O_3, etc.	Al, Ti resins, Ni alloys, etc.

328. Progress Report—Development of Advanced Composite Structures

Whipple, L. D.
(Paper C-2, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)
AFML

Paper presents a brief history of high modulus composite history and advantages, i.e., reduced weight and relief from design restraints. Program goals are: reliable and cost-effective use of composites; development of new designs and techniques taking full advantage of benefits, minimizing disadvantages; develop fabrication and test procedures; and design, build, and test full scale structures to demonstrate advantages of composites. Applications programs are described. An F-111 horizontal stabilizer has been made from B-epoxy skins, fiberglass honeycomb, and Ti end fittings. Tests were very successful. A T-39 wing box with B-epoxy face sheets on Al honeycomb, fiberglass spars, and stainless fittings failed at less than design loads due to failure at the fitting attachments. Helicopter tail rotors of B-epoxy were successfully made and tested, but a similar main rotor section was improperly fabricated, and failed prematurely. Integral compressor fan blades and stiffened cylinders also were made and tested successfully.

Fiber	Matrix
B	Resin

329. Elastic Moduli of Unidirectional Composites With Anisotropic Filaments

Whitney, J. M.

***J. Compos. Mater.*, Vol. 1, No. 2, pp. 193–199, Apr. 1967**

AFML

Elastic moduli are predicted for unidirectional composites with anisotropic fibers, using elasticity solutions of multiple-inclusion problems. The filaments are assumed to be transversely isotropic with the filament axis elastically symmetrical. Plots of theoretical and experimental properties are given for isotropic and anisotropic filament composites as a function of vol% fiber; properties include transverse tensile modulus and shear modulus. Effects of fiber orientation are given for moduli in tension and shear, Poisson's ratio, and shear coupling ratio; the limited data for graphite falls very nearly on the theoretical curves for anisotropic filaments. Such anisotropy introduces significant differences in elastic behavior of composites. Isotropy of fibers should be established before doing the micro- and macro-analyses of composites; this is not easy. If the filament Poisson's ratio is more than 1/2 or less than 0, it may be anisotropic. Certain B fibers have Poisson's ratios of 0.7 and, thus, may also be anisotropic.

Fibers	Matrix
Graphite, B	Epoxy

330. *Fibrous Reinforcements for Space Applications*

Whittaker Corp.

NASA CR-796, May 1967

The use of polymeric materials in fibrous form as reinforcements is examined. Only materials beyond the laboratory stage are included. Advantages, disadvantages, and ratings for such properties as strength, modulus, stability in vacuum or radiation, fiberability, etc. are given. Descriptions of some selected polymers are given, as are structural properties. Potential of these polymers is discussed. Some polymers have specific strengths equal to glass, but moduli are 1/2 that of glass. Little information is available on stability of most of the polymers in space environments. Applications may be dependent on load conditions. Standard test techniques are needed to correlate data from different sources. There has been little use of polymer fibers to date. Bibliography is included.

Fibers	Matrices
Polymers	Resins

331. The Fabrication of Preimpregnated Tape From Multiple Collimated Boron Fibers

Wilson, F. M.

(Paper B-2, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Whittaker Corp.

Paper describes techniques used to prepare B-resin prepregnated tape. Properties of B tape have been doubled in the past year due to improvements in B consistency, matrix materials, and tape preparation. Tape is prepared by maintaining a constant tension, precoating with controlled amounts of resin, and overwrapping layers of fibers. Fiber collimation and maintaining proper spacing are important also. Each B fiber is overwrapped with two glass fibers to control spacing. Control of the volatile content aids uniform resin distribution and reduces voids and gaps. Better resins also will increase composite properties.

Fiber	Matrix
B	Resin

332. Continuous Silicon Carbide Filaments

Withers, J. C., McCandless, L. C., Schwartz, R. T.

(Paper D-8, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

General Technology Corp., AFML

Batch studies were made on the effect of gas ratio, temperature, and flow rate on deposition of SiC by both codeposition and single source on 1-mil and 1/2-mil W. A variety of compounds and compound mixtures were used. Little success was obtained using codeposition, since hot spots developed in the wire, causing rapid burnout. Use of Mo, Ti, nichrome, and Pt-coated W did not prevent development of hot spots. The results obtained using various single source organosilanes are given; methyl and ethyl compounds gave the best results. A continuous reactor has been set up and is producing good quality fibers. The most important variables identified that affect tensile strength are purity of starting materials, temperature, reactant concentration, and gas ratio. At the gas ratios selected, the best temperature is 1200°C. Modulus varies from 60–75 × 10^6 psi, UTS is about 0.2 × 10^6 psi, and density on 1/2-mil W (4-mil fiber) is 3.35 gm/cm^3. Present systems allow deposition at about 2 ft/min. SiC produced recently is fairly uniform in properties, does not react with metals as readily as B, and retains strength at higher temperatures.

Fiber
SiC

333. High Modulus Filaments for Metal Matrix Reinforcements

Withers, J. C., Alexander, J. A.
(ASM Paper WES 7-75, presented at the Western Metal and Tool Exposition and Conference, Los Angeles, Calif., Mar. 13–17, 1967)
General Technology Corp.

Reactions between various fibers and matrices were studied. B reacts with everything but Al and Mg; at 1 h and 900°C, extensive reactions could be seen with Fe, Cb, Co, Ni, Ti, and Zr. If a reaction can be seen metallographically, it weakens the fiber. SiC is more stable than B in all matrices, and can be used with molten Al, which attacks B after 16 min. Diffusion bonding electroformed tapes or layers of fiber and foil are the most used fabrication method. Non-stoichiometry may give false indications of reactions. Better fibers do not react.

Fibers	Matrices
B, SiC, TiB_2, B_4C, MgO, Si_3N_4, Al_2O_3	Al, Mg, Ni, Fe, Co, Ti, Cb, Zr

334. High Modulus, High Strength Filaments and Composites

Witucki, R. M.
AFML-TR-66-187, May 1967
Astro Research Corp.

Internal stress distribution in B fibers was studied to determine the nature, cause, and distribution of the stresses, and to analytically predict stresses from the forming process. Most as-received fibers had residual curves after unwinding from the shipping drum; curved fibers had definite tendencies to split, while straight ones did not. Effects of various treatments on fiber curvature and splitting were examined in detail. Both outer skin and core were found to have residual axial compressive strains. Length of the W core increases during B deposition, due to conversion to borides. The B fiber also increases in length after an initial decrease when the surface is etched to remove successive layers of B. The final increase is not always seen. Thermal expansivity was determined between 50 and 1030°C. Creep was determined at 1070°C. Torsion modulus was affected by reaction time during fiber formation, and was lower for shorter times. Internal stresses are affected by the reaction time and the temperature and nature of reaction products. Volume change during formation of borides, differential thermal stresses during cooling, and strains inherent in the vapor deposition process cause internal fiber stresses. Examination by X-ray diffraction and electron microscopy to find the nature and extent of these strains is described. Strain relief in the core was observed on aging or etching. Stages of fiber formation and strains induced during each stage are described. Good agreement was found between experimentally determined and analytically predicted residual stresses. The nature and magnitude of stresses are derived mathematically. A small amount of similar study of SiC fibers was done. Appendixes give analyses of axial crack stability, and stresses from differential thermal expansion, Δ length, etc.

Fibers
B, SiC

335. Single Crystal Fiber Reinforcements

Wohrer, L. C., Economy, J.
(Paper B-7, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)
Carborundum Co.

Aligned composites of SiC whiskers were prepared by extruding a solution of whiskers in an organic matrix into a 1:1 solution of acetone and water, and then burning off the organic before adding a resin to the aligned fibers. As-extruded the whisker content was about 12–13%, but the composite had up to 50% whiskers. A similar technique was used for metals; a mix of organic polymer, whiskers, and metal powder is extruded, and the polymer burned off. This mixture is then hot pressed or sintered. No data on properties are given. Use of whiskers in continuous fiber composites is suggested to increase interlaminar shear strength by increasing matrix shear strength and modulus, and reducing crack propagation. The effects of such additions to epoxy-B and epoxy-steel systems are noted.

Fiber	Matrices
SiC	Resin, Cu, Al, Mg

336. Whisker-Reinforced Plastics

Wohrer, L. C., Frechette, F. J., Economy, J.
***Machine Des.*, pp. 138–141, Dec. 22, 1966**
Carborundum Co.

Thermoplastic resins were reinforced with whiskers by cold drawing, extruding, transfer molding, and injection molding. Cold-drawn samples with up to 7½% whiskers were weaker than the controls; extruded samples were weaker at 4½% and slightly stronger at 7%. Moduli of most resins were increased. SiC whisker yarns in phenolics utilized less than 1/5 the whisker strength and less than 1/2 the modulus. The importance of aspect ratio, matrix modulus, and critical volume fraction are mentioned briefly, as are anticipated trends.

Fibers	Matrix
SiC, Al_2O_3	Resins

337. Research on Boron Filament/Metal Matrix Composite Materials

Wolff, E. G., Hill, R. J.
AFML-TR-67-140, June 1967
AVCO Corp.

Study was made of the factors which affect physical, mechanical, and chemical properties of B-fiber metal-matrix composites, and on the optimization of process variables. Previous work on B-metal composites is surveyed, and a summary of fabrication techniques and results are given. Processes used for this program were liquid infiltration of Al, both vacuum casting and vacuum infiltration, and powder metallurgy of Al, Ni, and Cu. Vacuum casting gave low strength composites because the B was attacked by the molten Al. Fibers were continuously aligned, discontinuously aligned, and discontinuously random. Details of fiber alignment procedures, fabrication techniques, and results are given. Both Ni-coated and uncoated B fibers were used. Reactions between B and Ni occur at temperatures of 1300–1800°F, which is the range of interest for Ni composites, so further work with Ni was abandoned. Diffusion barriers for use with Ni matrices were studied. Cu applied by immersion or immersion and electroplating were ineffective; Al_2O_3 applied by electrophoresis was porous and not very effective; while dipping gave a better coating, but still not a satisfactory one. BN applied by electrophoresis was uneven, but dipping gave a more uniform coating which survived 48 h at 1650°F without reaction in Ni. Dip coatings of B_4C with Al_2O_3 did not provide good diffusion barriers. Other coatings which were tried and found ineffective were C, ThO_2, Re, W, SiO_2, ZrO_2, Al, and Pd. Coating with Al_2O_3 had no effect on the B tensile strength. B in Al was held at 932°F for 112 h without degradation. The corrosion behavior of B-Al in NaCl, $NiCl_2$, and buffered solutions of pH 4, 8, and 10 was studied. In general, the composites were not attacked more than plain Al. Corrosion of B-Cu in NaCl, FeCl, and HNO_3 was no worse than unreinforced Cu. B-Ni in NaCl had the same corrosion behavior as bare Ni; but in $Al_2(SO_4)_3$, the composite was attacked more severely than Ni. Mechanical properties of some samples before and after corrosion tests were made and little difference was found between B-Al and just Al. Vacuum infiltration of Al was most effective if the samples were water quenched after 2–4 min at 1330–1470°F. No change was noted in strength whether air or A was used as a cover gas. Both room and elevated temperature tensile tests were performed. Continuously reinforced samples of B-Al alloy had much better high temperature strength than discontinuously reinforced ones. Residual stresses are induced in B-Al composites during elevated temperature fabrication due to differences in coefficient of thermal expansion. These stresses can cause reductions of composite strength. Fatigue and creep strength of B-Al were determined. Composites had much improved fatigue and creep strengths. Thermal conductivity of continuous and discontinuous B-Al composites were determined up to 200°F. Thermal expansion of B-Al and B-Cu between 75 and 600°F was found, as were the densities. Tapes were made by drawing B through molten Al. The effects of process variables such as draw speed, temperature, alloy content, crucible material, preheat, length of hot zone, B surface, orifice size, and atmosphere were studied. The tapes were hot pressed and vacuum infiltrated to form larger bodies successfully. Samples made with these tapes had higher strengths than those made by the same consolidation method but with plain B fibers.

Fiber	Matrices
B	Al, 4OE Al, Cu, Ni

338. Ceramic-Metal Composites: Nucerite

Wood, W.
***Proc. Inst. Mech. Eng.*, Vol. 180, Pt. 3D,**
pp. 193–199, 1965–1966
Befour Great Eleven/Scotland

Properties and uses are described for Nucerite, a composite of metal and crystallized ceramic used mostly in chemical process equipment, such as reactor liners, manhole protection rings, baffles, and agitators. Surface preparation and cleanliness of the metal are vital. The ceramic is coated onto the metal and crystallized by heat treatment. The metal should have a higher thermal expansion coefficient than the glass to keep the glass compressed.

Fibers	Matrix
Steel, Inconel	Ceramic

339. Preparation and Properties of Eutectic Bi-MnBi Single Crystals

Yim, W. M.

(Presented at the 96th Annual AIME Meeting, Los Angeles, Calif., Feb. 19–23, 1967)

RCA

They grew single crystal Bi with 8-vol% MnBi filaments by zone melting in a magnetic field (500 Oe). The fibers were about 5 μm diameter by about 200 μm long, perpendicular to the trigonal axis. The eutectic was ferromagnetic and strongly anisotropic, but with the same electrical and thermal properties as Bi. Could not grow fibers in the absence of magnetic field.

Fiber	Matrix
MnBi	Bi

340. New Techniques for Making High Temperature Resistant Metal Matrix Composite Materials

Yoblin, J. A.

(Paper F-7, presented at the 10th SAMPE Symposium on Advanced Fibrous Reinforced Composites, San Diego, Calif., Nov. 9–11, 1966)

Whittaker Corp.

Cellular composites were formed by co-extrusion of core and cladding in a variety of shapes. Honeycomb allows the best packing densities; up to 95% reinforcement has been obtained. A variety of shapes and cladding thicknesses have been produced. Another technique is to metal-coat a core material and to co-extrude them, forming the fibers during extrusion, or elongating existing fibers.

Fibers	Matrices
SS, Zircaloy, W, Ta, Zr, resistant metal	Pyrex, Mo, SS, U, Ta, Al, Mg, Hastelloy X

341. Microstructure of Magnesium-Aluminum Eutectic

Yue, A. S.

***Trans. AIME*, Vol. 224, pp. 1010–1015, Oct. 1962**

Dow Chemical

The effect of solidification conditions on the structure of Mg-32%Al was studied. Temperature gradient across the freezing interface had no effect on interlaminar spacing, or the transition from lamellae to rods. At low freezing rates lamellae were formed, while at higher rates rods were formed. Lamellaer spacing was closer as freezing rate increased; number of faults in the lamellae also increased with increasing freezing rate. When rods are formed, increasing freezing rate increases the number of rods, but lowers their diameter. Mechanisms of eutectic formation and growth are discussed.

342. Development of Nondestructive Methods for the Quantitative Evaluation of Glass-Reinforced Plastics

Zurbrick, J. R.

AFML-TR-66-269, Mar. 1967

AVCO Corp.

Quantitative relationships between nondestructive tests and glass-reinforced plastic-laminate design properties were examined to develop ways of avoiding overdesign or performance variations. Test methods included radiometry, radiography, ultrasonic, low frequency and microwave dielectrics, visual appearance, and correlation with destructive tests on a statistical basis. Material preparation and test techniques are described. Radiometry was found useful for density measurements; radiography detected frayed and folded glass cloth, small bubbles, and inclusions. Ultrasonic velocity was used to determine modulus. Low frequency dielectrics were used to measure variability of the laminates, while microwave frequency dielectrics were used to measure dielectric constant and variability. Samples were prepared with intentionally varied resin and void content, and were tested destructively and nondestructively. Tests included density, resin content, porosity, dielectric properties, and tensile and flexural properties. Both resin-specific and general data crossplots were prepared as part of the correlation between NDT and mechanical data. NDT responses represent the true material, not a simplified model. Mechanical design parameters are based on assumed homogeneity, isotropy, and uniform stress distribution. Epoxy systems had a clear relation between ultrasonic velocity and % porosity and % resin. For other systems, the porosity relations were less clear; polyesters and silicones had a pretty definite relation between % resin and ultrasonic velocity, but the others had considerably less well established trend lines. Results were similar for relative capacitance as a function of resin content. It was concluded that elastic moduli are dependent on resin mechanical properties, particularly resin moduli. Apparent tensile modulus is affected by resin shear deflections, porosity between plys and within plys, and resin content. Macroporosity between plys has much more effect on ultrasonic velocity perpendicular to the plys than on moduli. Porosity within plys has a large influence on modulus and other mechanical properties. Ultrasonic velocity parallel to the laminates is influenced most by porosity and resin content within plys, and least by resin content or porosity between plys. Combining ultrasonics and radiation gauging permits identification of between-ply porosity and can predict elastic modulus. The geometry and the location of resin and porosity have great effects on ultrasonic velocity. Ultrasonic waves perpendicular to the plys can detect high interply void content and low interply resin content. Ultrasonic waves parallel to the ply can give tensile modulus to within 0.7×10^6 psi and flexural modulus to within 0.37×10^6 psi. More development is needed in low frequency and microwave dielectric measurements. Use of proper coupling agents results in in-ply microporosity being much less than between-ply macroporosity. Several techniques can predict densities accurately for various systems. Limitations of the equipment is discussed. Appendixes cover effects of porosity on ultrasonic velocity, laminate preparation, and NDT and destructive test procedures.

Fiber	Matrices
Glass	Epoxy, phenolic, polyester, silicone, polybenzamidazole

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