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# High Temperature Composites—Status and Future Directions

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# HIGH TEMPERATURE COMPOSITES - STATUS AND FUTURE DIRECTIONS


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## ABSTRACT

High temperature composites are being studied in the hope of creating a new class of materials with higher-use-temperature potential. The types of composites being explored include ceramic/ceramic, ceramic/metal, and metal/metal. This paper will review the general results obtained for each type with a view toward evaluation of their current status and suggestions for future direction of development.


## INTRODUCTION

Designers of heat engines, particularly aircraft gas turbine engines, can increase efficiency and reduce fuel consumption by increasing combustion temperature. Combustion gas temperatures for peak efficiency are typically well above those allowable by the maximum service temperature of current hot components. Materials research over the past thirty years on high temperature alloys, principally nickel and cobalt base, has provided ever-increasing component operating temperature limits which currently are 951-981 $^{\circ}$C (1751-1811 $^{\circ}$F). More recent alloy improvements also have permitted an increase in service life from a few hundred to several thousand hours. In addition, elaborate and ingenious air-cooling designs have allowed increased combustion temperatures without exceeding material use temperature limits. The success of these efforts has permitted remarkable growth in reliability and efficiency for aircraft gas turbines which has helped to revolutionize air transportation. However, the limited potential for further improvements in conventional alloys to permit higher use temperatures has fostered work toward alternate material approaches. Directional solidification of superalloys, both polycrystalline and single crystals, is being used to eliminate transverse grain boundaries as a source of weakness. Directional solidification of eutectic alloy compositions is controlled to form rod or lamellar phases with the strengthening characteristics of composites. Oxide dispersion strengthened superalloy composites, with oriented structures, are another approach being pursued for heat-resisting materials. Fiber composites fabricated by bonding together strong, refractory fibers into structural elements are still another promising family of materials.

The diminishing additional gains possible from conventional polycrystalline alloys also is influenced by the severe erosive and corrosive oxidation environment present in modern turbine engines. Refractory metal alloys have demonstrated significant strength and creep resistance at higher temperatures but with unsatisfactory environmental resistance. Coatings, the subject of intensive study, show indications of some success, but with inadequate reliability for the long service times required. The failure to provide refractory alloys with a satisfactory combination of properties for high-temperature service has directed increased attention to ceramic materials. Conventional ceramic materials such as oxides, carbides, and nitrides have a combination of high-temperature properties that make them potential candidates for turbine components. However, some significant property deficiencies, including low ductility and flaw tolerence, have restricted their application.

Composites offer a promising approach for achieving structural materials that combine the high temperature strength of refractory materials while preserving much of the toughness and flaw tolerance of metals. This paper will discuss those composites which appear to have high temperature potential. These include: brittle/brittle combinations, such as silicon carbide or carbon fibers in a ceramic or carbon matrix, brittle/ductile combinations, such as aluminum oxide fibers in iron or nickel alloys, and ductile/ductile combinations such as refractory metal alloy wire in iron or nickel alloys.

## BRITTLE/BRITTLE COMPOSITES

The high temperature strength of conventional monolithic ceramics is sufficient for a significant use temperature increase. Two ceramics studied for turbine applications are silicon carbide and silicon nitride. A combination of high temperature strength and thermal cycle resistance makes these materials logical choices for study. However, their low toughness 5alues make them vulnerable to catastrophic failure. Attempts to increase toughness by incorporating refractory metal wire mesh have met with some success. But cracks, which can not be avoided in the brittle matrix, may lead to severe oxidation of the reinforcement wire. Despite this, refractory wire/ceramic composites can be considered for short time applications.

Use of oxidation resistant ceramic fibers has been studied to add toughness without sacrificing oxidation resistance. A severe limitation to this approach has been the relatively small number of ceramic compositions available in fiber form. Silicon carbide and carbon fibers have been hot-pressed into ceramic matrix composites. The high processing temperatures required have caused a reaction between matrix and fiber which typically has severely degraded fiber properties. This problem has fostered approaches which use weak bonding between fiber and matrix. A good example is carbon fibers in glass. Small diameter fibers are preferred for such an approach because their large surface-to-cross-section area provides a small stress transfer length. Further, small diameter fibers can provide a small inter-fiber spacing which limits stress induced matrix crack progagation length. The potential of this type of composite for high service temperatures at moderate strength levels has fostered a renewed interest in ceramic matrix composites. However, the primary effort in high temperature composites over the past two decades has been directed at the ductile matrix type, both with a brittle ceramic filament and with more ductile refractory metal alloy filaments.

## BRITTLE/DUCTILE COMPOSITES

The initial impetus to study fiber composites was based on the strengths of ceramic fibers and whiskers which were well above the values possible with bulk forms of the same material. Other properties that make such reinforcements a leading choice for composite development are high modulus, good strength retention at elevated temperatures, excellent oxidation and corrosion resistance, and a density lower than that of conventional high-temperature alloys.

Single crystal aluminum oxide or sapphire whiskers, such as those in figure 1, have demonstrated tensile strengths approaching a theoretical value for cohesive strength, about 11 percent of Young's modulus. The scatter in strength is large, but small diameter whiskers, under one micrometer, display values to 41 $GN/m^2$ (6 million psi). Typical tensile values of 6.9 $GN/m^2$ (1 million psi) at room temperature decrease to 4.1 $GN/m^2$ (611,111 psi) at 1191$^{o}$C (2111$^{o}$F). Consolidation of whiskers with such high temperature strengths into useful metal matrix composites offers the potential for remarkable strength improvements. However, small size whiskers are difficult to fabricate into a composite. They must be reasonably well aligned, surrounded by the matrix, and bonded to the matrix to be able to transmit stress. The strength of the near-perfect crystal structure of whiskers is associated with very smooth, flaw-free surfaces which can be readily altered. Bonding is particularly troublesome because reaction at the matrix-whisker interface to achieve bonding can roughen the surface and drastically reduce strength. Fabrication methods also are troublesome because of the limited fiber-to-fiber distance necessary to achieve a reasonable fiber content. For example, with a 1 micrometer diameter fiber, less than a 1/2$\mu$m distance between fibers is necessary for a 51 volume percent fiber content. Liquid infiltration is a possible fabrication method, but poor wetting can inhibit matrix infiltration. Alloying with elements to improve wetting also tends to accelerate attack of the filament surface. Thus, opposing effects must be combined to achieve a difficult compromise.

Coatings have been tried on whiskers as a technique to improve bonding while resisting surface attack of the reactive matrix. However, the small diameter of whiskers limits coating thickness to achieve reasonable volume fiber content. But thick coatings typically are needed to promote matrix infiltration into the whisker array. For example, a 1/2 μm coating of tungsten or platinum metal was dissolved in a few seconds in an attempt to infiltrate liquid nickel matrix into alumina whiskers. Dissolution of the coating was followed by poor infiltration and whisker strength degradation. Powder metallurgy fabrication approaches also were unsuccessful because of the small interfiber distances with whiskers and because the hot pressing of the solid state powder/whisker billets caused whisker damage and fracture. In almost all cases, the high temperature properties achieved for whisker composites were disappointing compared with conventional superalloys.

A further problem encountered with composites of sapphire whiskers and iron, cobalt, or nickel matrix alloys is the large thermal expansion mismatch between fiber and matrix which generates internal stresses when composites are exposed to the typical thermal cycle of heat engines. The stresses can cause disbonding or fracture of fibers. The lack of success with sapphire-whisker fiber-reinforced metal composites, to a large degree, was heightened by the small sizes of whiskers.

In an attempt to circumvent these problems, a considerable effort was undertaken to explore brittle/ductile composites using larger diameter fibers. Flame polished single crystal rods, as well as single crystal continuous fibers, were produced. These fibers, with diameters from 100 to

350 μm (.004 to .015 inch), had tensile strength values lower than the higher strength whiskers but about equivalent to the average values of whiskers. Strength values were sufficient to encourage studies aimed at developing composites for high temperatures. The larger diameter greatly simplifies fabrication. Fiber coatings to promote wetting and inhibit surface attack can be applied with a thickness of several micrometers. Several candidate coatings, including refractory metals, carbides, and oxides were tried. Composite test specimens were fabricated using liquid and solid state fabrication incorporating 100–350 μm (.004–.015 inch) diameter sapphire fibers. However, long time exposure to service temperatures and thermal cycles indicated problems. A combination of surface attack and thermal cycle induced stress caused failures at disappointing strength levels. Relatively minor surface attack, when coupled with the stress from thermal expansion mismatch between fiber and matrix, was sufficient to cause failure by fiber fracture or disbonding. The current level of activity in aluminum oxide fiber/metal composites for high temperature use is very low because such problems were encountered. These problems while formidable are not unresolvable.

One limitation hampering development of brittle/ductile composites is the limited number of ceramics composition available in fiber form. A number of different fabrication processing methods have been studied to make it possible to fabricate a family of ceramic fibers for high temperature composites. One of the more successful methods developed uses a focused laser beam to melt the tip of a polycrystalline feed rod. The suspended molten drop is then contacted by a single crystal seed rod which is withdrawn at a controlled rate.

Continuous filaments with diameters from 50 to 500 μm (.002 to .020 inches) were grown in this way. This method offers the potential for growth of fibers of a wide range of high melting temperature ceramic composition without the limitation of crucible contamination. Improved fibers combined with a matrix composition to reduce thermal mismatch effects offers a potential means to overcome the problems that have prevented the achievement of the brittle fiber/metal matrix high temperature composites.

Silicon carbide shares with sapphire a decade or more of concerted study as a reinforcement for high temperature composites. Silicon carbide whisker/metal composites were studied and abandoned because of the same problems encountered with sapphire whiskers. Fortunately, silicon carbide polycrystalline fibers, produced by chemical vapor decomposition on a heated substrate, were available for use in studies of high temperature composites. These fibers, while not as strong at the intended service temperatures, $980^{o}C$ to $1200^{o}C$ ($1800$–$2200^{o}F$), offer a large advantage over the best superalloy candidates. The comparison of calculated, density compensated strength of SiC composites to similar data for typical superalloys, figure 2, shows what could be achieved if SiC fiber strength could be utilized in composites. SiC is readily wet by most candidate superalloys and there is no problem in obtaining bonding. However, the reaction-degradation of SiC filament with most candidate matrix alloys can quickly destroy the filament at temperatures above $980^{o}C$. Thus, coatings are necessary to achieve reasonable service lives. The micrograph in figure 3 shows the reaction obtained with an uncoated fiber as contrasted with a coated filament.

Refractory metal coatings can be effective, but the density increase from the coating thicknesses required, 20–50 μm, are prohibitive. Refractory carbide coatings have been shown effective, but a practical coating process to obtain long time diffusion barrier reliability has been elusive.

As with sapphire filaments, the thermal expansion difference makes the problem of retaining a stable coating between the high thermal expansion matrix and the lower expansion of the filament difficult. The brittleness of coatings and the coating/fiber interface further compounds the complexity. Recent studies in the US and USSR have addressed the reaction problem between SiC and Fe, Ni, and Co matrix materials. The results are consistent in indicating the need for a coating to reduce reaction at temperatures above $980^{o}C$ ($1800^{o}F$). There is some divergence of results at lower temperatures with the most pessimistic indicating reaction starting at $675^{o}C$ ($1250^{o}F$). However, other studies indicate relatively limited reaction to $925^{o}C$ ($1700^{o}F$).

## DUCTILE/DUCTILE COMPOSITES

The problems encountered in studies to develop the technology of brittle fiber/ductile matrix high temperature composites had thwarted efforts to achieve the high potential strengths theoretically possible. As discussed above, the strength limits obtained have been low because of a combination of fiber/matrix surface reaction and internal stresses from thermal expansion mismatch. Further approaches to overcome these problems should be undertaken. An alternate approach which has received continued effort is based on refractory metal alloy wire as the high strength phase. While the theoretical specific strength potential of this family of metal matrix composites is less than that of ceramic fibers, as shown in figure 4, the ductility of refractory alloy wire drastically reduces the severity of the two primary obstacles with ceramic fibers. As described above, ceramic fibers were notch sensitized by the surface roughening effect of reaction with matrix alloy elements. In contrast, fiber-matrix reaction with alloy wire causes a diffusion controlled, very gradual degradation of wire properties typical of the strength degradation of highly alloyed superalloys at their service temperatures. In addition, as will be shown subsequently, the temperature for such degradation of refractory metal alloy is above that at which superalloys are used. Further, the thermal expansion mismatch problem is more tolerable because of the ability of the fiber to relieve strains rather then to fracture. Thus, a ductile/ductile combination offers the practical potential for realizing a use temperature increase in the near term. This system also serves as a learning vehicle since many of the technology factors for high temperature composites are common to all systems and the refractory alloy wire/superalloy composite system offers the opportunity to address them.

Screening of Mo, Nb, Ta, and W alloy wire properties indicated the potential for several candidate systems. Since the creep rupture properties of composites are usually directly related to those of fibers, these data were evaluated first. Most of the studies were conducted using commercially available lamp filament and thermocouple wire. The need for higher strength wire was recognized and fabrication processing for producing wire from stronger alloys was undertaken. Representative data from that program are shown in figure 5. The 100-hour 1090$^{o}$C (2000$^{o}$F) rupture strength of the strongest wire produced, W-Re-Hf-C wire, was over 16 times that of superalloys and twice as high as commercial lamp filament. The properties of the W-Re-Hf-C wire were not optimized by a thorough thermomechanical fabrication study. Further, there has been no refractory metal alloy composition study conducted with the purpose of maximizing strength properties for composite application temperatures. Thus, the properties demonstrated thus far are merely indications of the properties of the first generation of fibers. Considerable further improvement in properties can be projected. Based on these strength data and other factors, tungsten wire was chosen for most composite studies. The acronym TFRS is used for tungsten fiber/superalloy composites.

Matrix alloy selection also plays a major role in TFRS properties. Matrix alloy composition must be compatible with fibers to minimize interdiffusion related strength degradation. The photos of figure 6 show the wide range of compatibility that variation in matrix composition can achieve. The photos in figure 6 a, b, d, and e show the varying degree of reaction with commercial tungsten lamp filament and the four alloys listed in Table I.

TABLE I

Nickel-Alloy Matrix Materials

Nominal Composition of Alloy (w/o)

| Alloy number | Al | Nb | Cr | Mo | Ni | Ti | W | Ta |
|---|---|---|---|---|---|---|---|---|
| 1 | -- | -- | 20 | -- | 55 | -- | 25 | -- |
| 3 | 2 | -- | 15 | -- | 56 | 2 | 25 | -- |
| 5 | -- | 1.25 | 19 | 4 | 70.5 | -- | 4 | 1.25 |
| 7 | 4.2 | 1.25 | 15 | 4 | 66.8 | 3.5 | 4 | 1.25 |

The almost complete destruction of TZM molybdenum alloy wire by all four alloys is typified by the results with alloy 1 in figure 6c. Data have been accumulated to indicate that matrix compositions can be selected to control reaction and bond the fibers into a useful structural member. However, other requirements influence matrix alloy choice. For example, oxidation and hot corrosion protection of the otherwise oxidation prone tungsten also is necessary. Also, matrix ductility is important to provide impact resistance at low temperature and to provide mechanical and thermal fatigue resistance. In general, relatively strong conventional nickel and cobalt alloys have been unsatisfactory as matrix alloys for TFRS because of fiber/matrix interdiffusion and property loss. These alloys also are less effective in resisting strain from thermal expansion mismatch. Weak, ductile, oxidation-resistant, iron-base coating alloys (Fe, Cr, Al, Y) have been the best compromise compositions identified to date.

One of the significant first accomplishments with TFRS was to evolve fabrication processing for production of test specimens in order to evaluate properties. As with many other composite materials, conventional fabrication processing methods such as casting, rolling, and forging are not ideal. Of the several fabrication processing approaches used, diffusion bonding of fiber arrays with matrix alloy powder or matrix foil has been the most effective method. A number of mechanical properties have been evaluated to permit an evaluation of the relative merit of TFRS compared with current high temperature alloys. The brevity of this review prohibits detailed discussion of the properties of TFRS. However, a brief review of some results is appropriate here to indicate the potential of the material and to identify the problem areas. The stress rupture data for 100 hours at 1090 $^{\circ}$C (2000 $^{\circ}$F) for representative TFRS and the strongest superalloys are shown in figure 7. The W-Hf-C/superalloy composite is more than three times stronger than the superalloys. Results obtained in creep rupture indicate the potential to increase use-temperature as much as 150 $^{\circ}$C (300 $^{\circ}$F) above that of the best currently identified superalloys for turbine blades. The properties measured included thermal stability, impact resistance, thermal fatigue, thermal conductivity, and thermal expansion. Screening of these properties has indicated that TFRS has promising potential for further development. However, one of the critical areas in need of further study is thermal fatigue, particularly with the fiber content and ply orientation for engine components, rather than with simple unidirectional test specimens used thus far.

One of the compensating benefits associated with the low thermal expansion properties of tungsten fibers is that TFRS composites have a low thermal expansion compared with conventional superalloys. Thus, low expansion thermal barrier ceramic coatings on TFRS composites would be expected to improve cyclic temperature service lives since the coatings would be less likely to spall from the TFRS substrate. Thermal barrier coating can effectively permit an increase in the allowable gas temperature without increasing material temperature. Alternatively, cooling airflow can be reduced at a given turbine gas temperature. Both are advantageous to increase performance and/or to reduce fuel consumption.

A further potential advantage that TFRS offers for cooled turbine components is a much higher thermal conductivity. Tungsten has about a three-fold higher thermal conductivity compared with superalloys. Thermal conductivity of TFRS with typical fibe content is about one and one-half to two times the thermal conductivity of superalloys. This higher thermal conductivity can significantly improve the potential use-temperature of air-cooled components. This advantage also can permit the use of a simpler, lower cost cooling geometry. For example, convention cooled blades with impingement inserts may be substituted for the complex geometries needed for film or transpiration cooling.

Fabrication processing of simple test specimen geometries has been accomplished using several solid state methods. The processing methods used to produce TFRS test specimens followed the techniques evolved for aluminum and polymer matrix composites. Pressure and temperature are applied to an assembled array of fiber/matrix composite plies. An advantage for TFRS is the ability to plastically deform the tungsten alloy wire at temperatures above about 370$^{o}$C (700$^{o}$F). Plastic deformation eases the problem of fiber cracking during fabrication and permits secondary plastic deformation of a composite billet. The fabrication of complex, hollow, air-cooled airfoils parallels the methods used to make fan and compressor blades with refinements to permit the hollow airfoil and film cooling holes near the trailing edge. The process shown schematically in figure 8 has been used to fabricate a prototype first stage turbine blade. The process uses diffusion bonding of monolayer composites along with steel core plies and unreinforced cover skin plies at the inner and outer surfaces. After diffusion bonding, the steel is leached from the airfoil leaving a hollow configuration. An impingement cooling insert can be inserted to improve the interior cooling airfoil path. The successful fabrication of the blade, figure 9, offers evidence that fabrication processing of complex components can be achieved. This accomplishment is to provide a basis upon which the manufacturing technology for TFRS components can be developed.

A further milestone has been reached as part of the prototype turbine blade fabrication, which addresses the concern of component density and weight for TFRS composites. The density compensated mechanical properties values of TFRS are used for comparison with superalloy properties for components, particularly rotating airfoils where component stress levels are density related. Since tungsten is twice the density of nickel, the concern has been that while the density compensated strength values are high, the component weight could be high also and affect the requirements for other components such as turbine disks. However, by varying the fiber content along the span length of the airfoil to match the stress and temperature requirements and by varying the hollow blade wall thickness, blade weight can be very similar to that of superalloys. The prototype blade weight with TFRS was within 10 percent of a conventional superalloy blade weight.

## SUMMARY

The study of high temperature composites, fostered by the desire to provide improved heat resistant engine components, has been rewarded with considerable success and some disappointments. Composites with brittle ceramic fibers such as $Al_2O_3$ or SiC, either whiskers or monofilament in a ductile metal matrix, have achieved strengths far below their promising theoretical potential. Reaction at the fiber-matrix interface degrades fiber strength by causing surface flaws which act as local stress concentrations. Thermal expansion mismatch between ceramic fibers and candidate matrix alloys can induce severe internal stresses. The combination of thermally induced stress and surface flaw stress concentrations leads to low strengths. Coatings and/or alternate fibers are needed to consider future work in this area promising for increased temperature components.

Composites of ceramic fibers in a ceramic matrix have had mixed results. Attempts to increase toughness for conventional high strength ceramics such as silicon carbide or silicon nitride by reinforcing with carbon or ceramic filaments have made limited gains. The high fabrication temperatures for producing the matrix body without destroying the filament presents a difficult challenge. An approach that has offered the potential for intermediate strength composites is to bond an array of fibers using only a mechanical bond or very weak chemical bond. Graphite/glass and carbon/carbon composites have shown useable properties at intermediate to high temperatures.

Efforts in ductile refractory metal fibers in a ductile metal matrix have focused on tungsten fiber reinforced superalloys. TFRS offers a promising potential for a use-temperature increase for turbine components of up to $150^{\circ}C$ ($300^{\circ}F$) above commercial superalloys. Also, a successful demonstration has been made of the feasibility to produce a hollow air-cooled blade with a weight approaching that of superalloys. However, the larger effort to develop manufacturing process technology is yet before us. A specific and complete data base must be accumulated upon which more detailed component designs can be developed. These data will include the effects of multiaxial stress and cyclic thermal conditions required in components.

An additional need that must be addressed for all high temperature composites, including FRS, is the development of failure models and associated analysis techniques to predict performance and aid in design. The lack of such theory and supporting data are a major obstacle to the acceptance of composites for high temperature applications. Similarly, fabrication of and simulated service evaluation of components are necessary to develop the confidence for service commitment. The revolutionary nature of composites, combined with the complex requirements for all high temperature service, presents a formidable uncertainty impeding acceptance by high level decision makers. The benefits to be gained are great, but the perceived risks must be reduced to gain wider acceptance.

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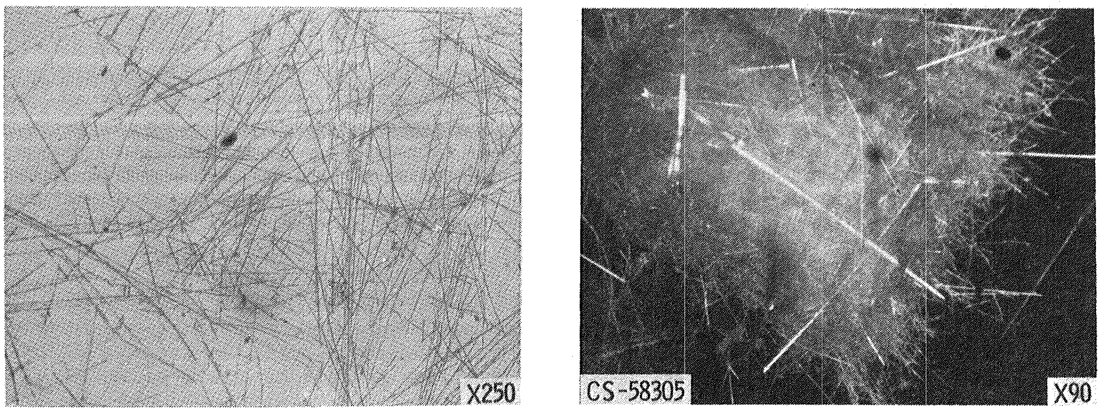



(a) Separated whiskers on a glass slide. (b) Section of whisker matt not separated.

Figure 1. - Typical appearance of separated and unseparated $Al_2O_3$ whiskers.

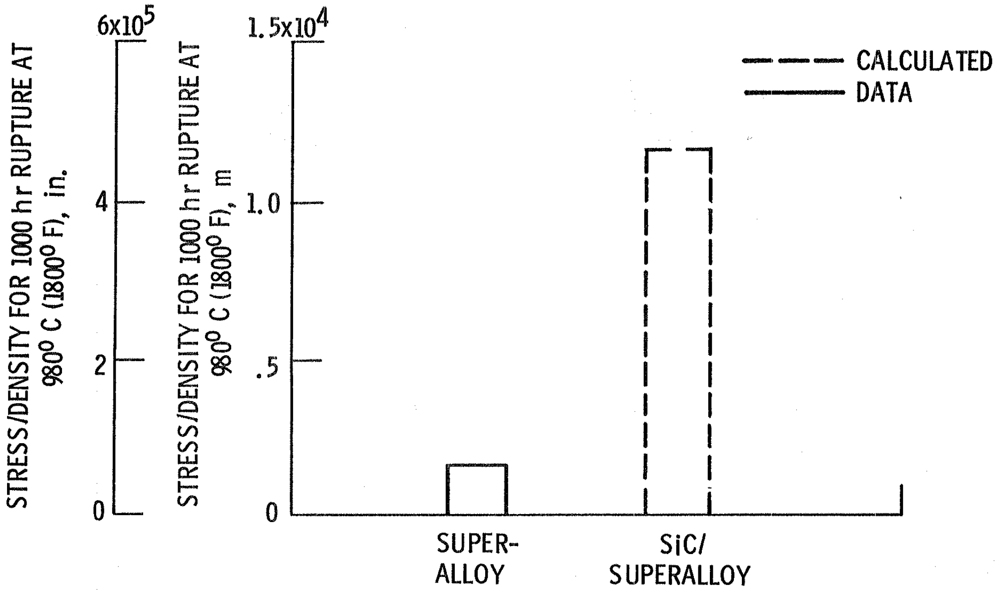



Figure 2. - Potential strength of silicon carbide/superalloy.

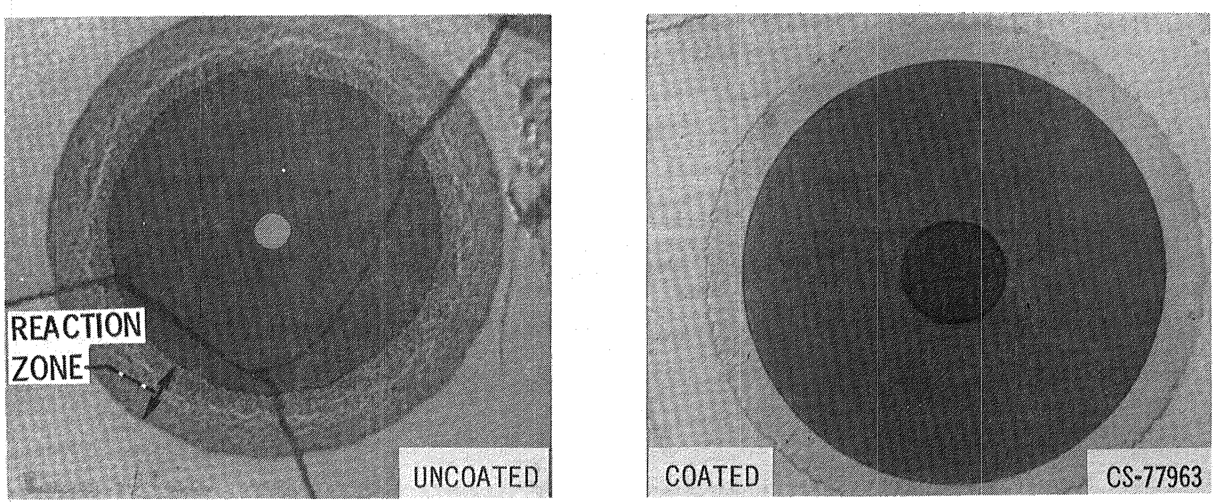



Figure 3. - Interfacial reaction of silicon carbide/superalloy.

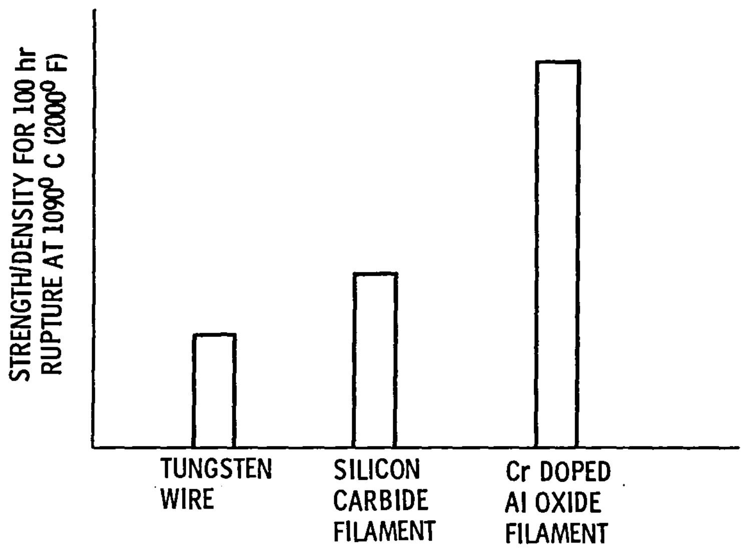



Figure 4. - High temperature fiber strength.

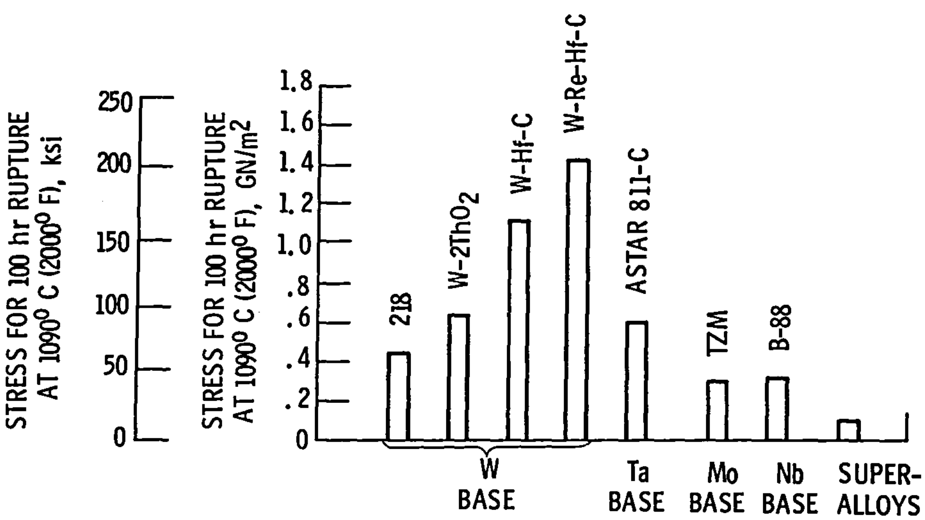
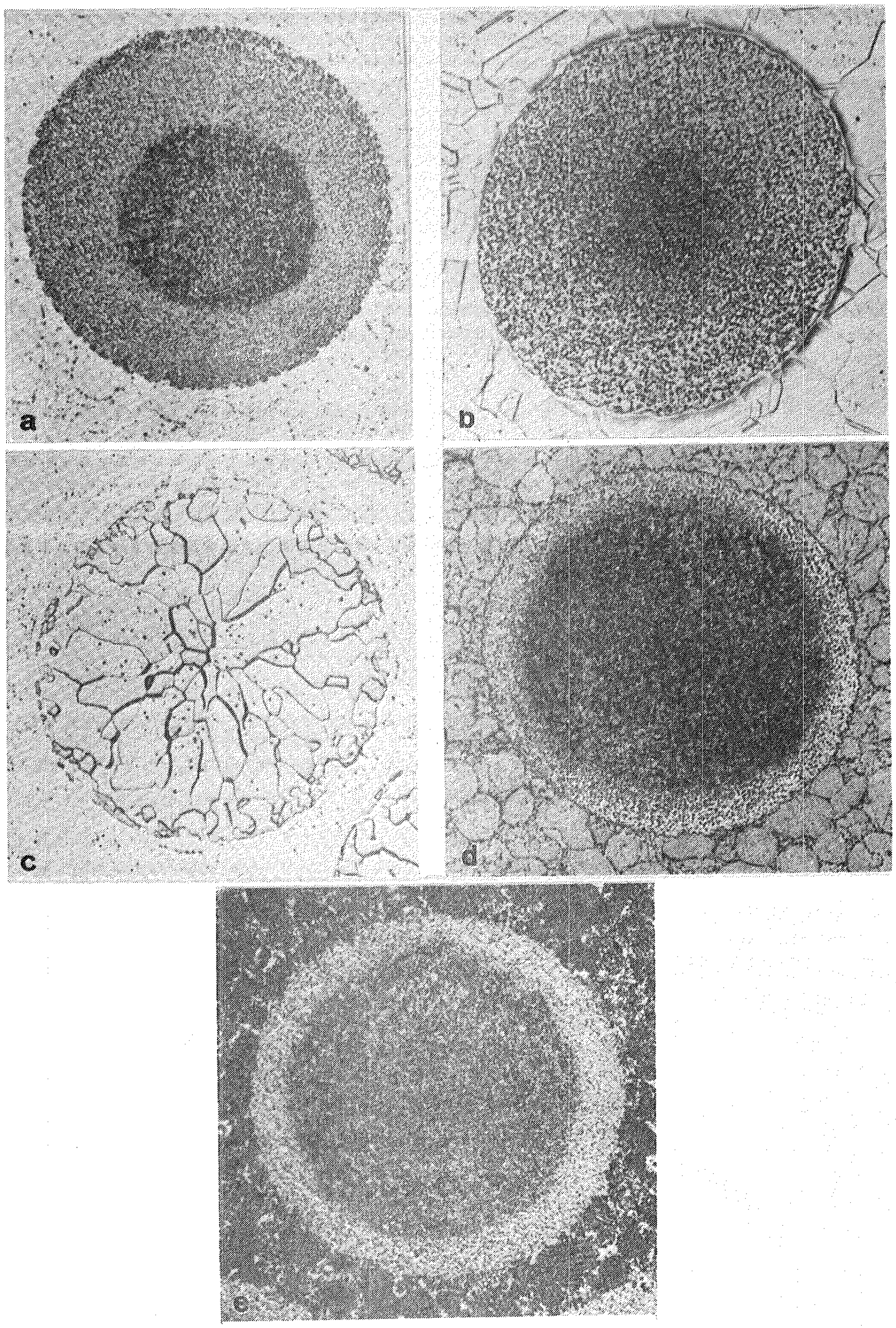



Figure 5. - Refractory alloy wire rupture strength at 1090° C (2000° F).




(a) Alloy 1/218 wire, (b) Alloy 5/218 wire,

(c) Alloy 1/TZM wire, (d) Alloy 3/218 wire,

(e) Alloy 7/218 wire.

Figure 6. - Refractory wire/matrix reaction.

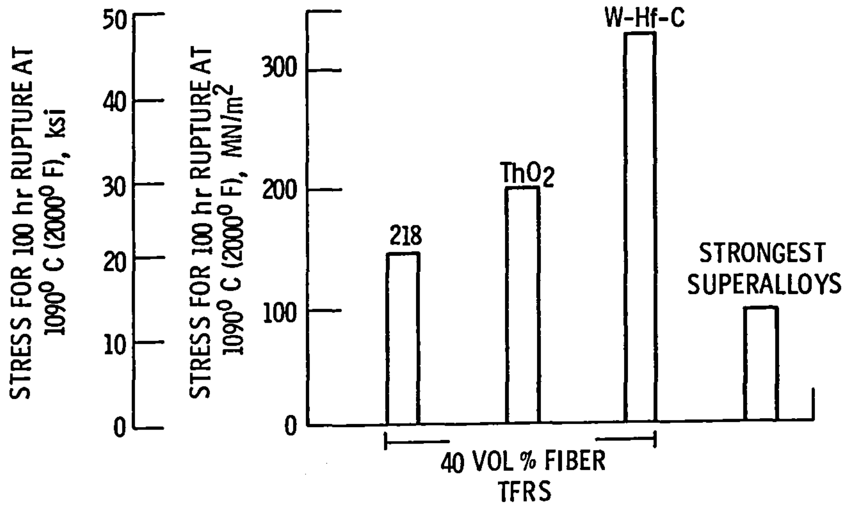



Figure 7. - TFRS composite rupture strength at 1090° C (2000° F).

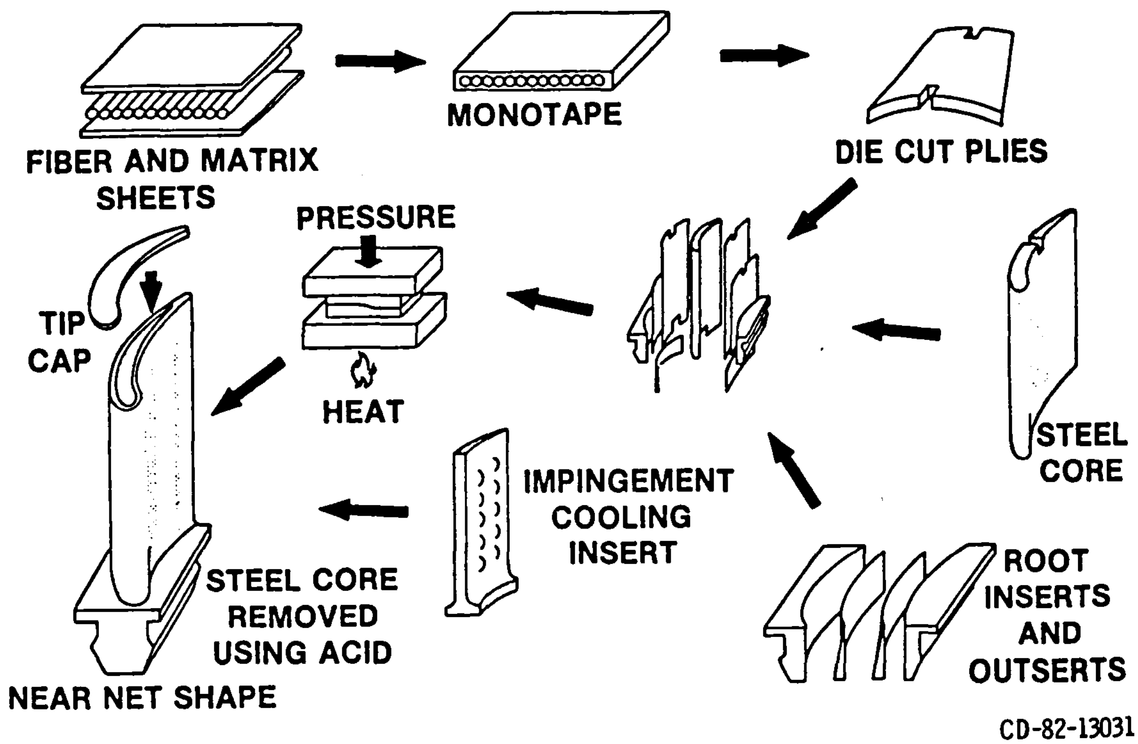



Figure 8. - Schematic of TFRS fabrication process.

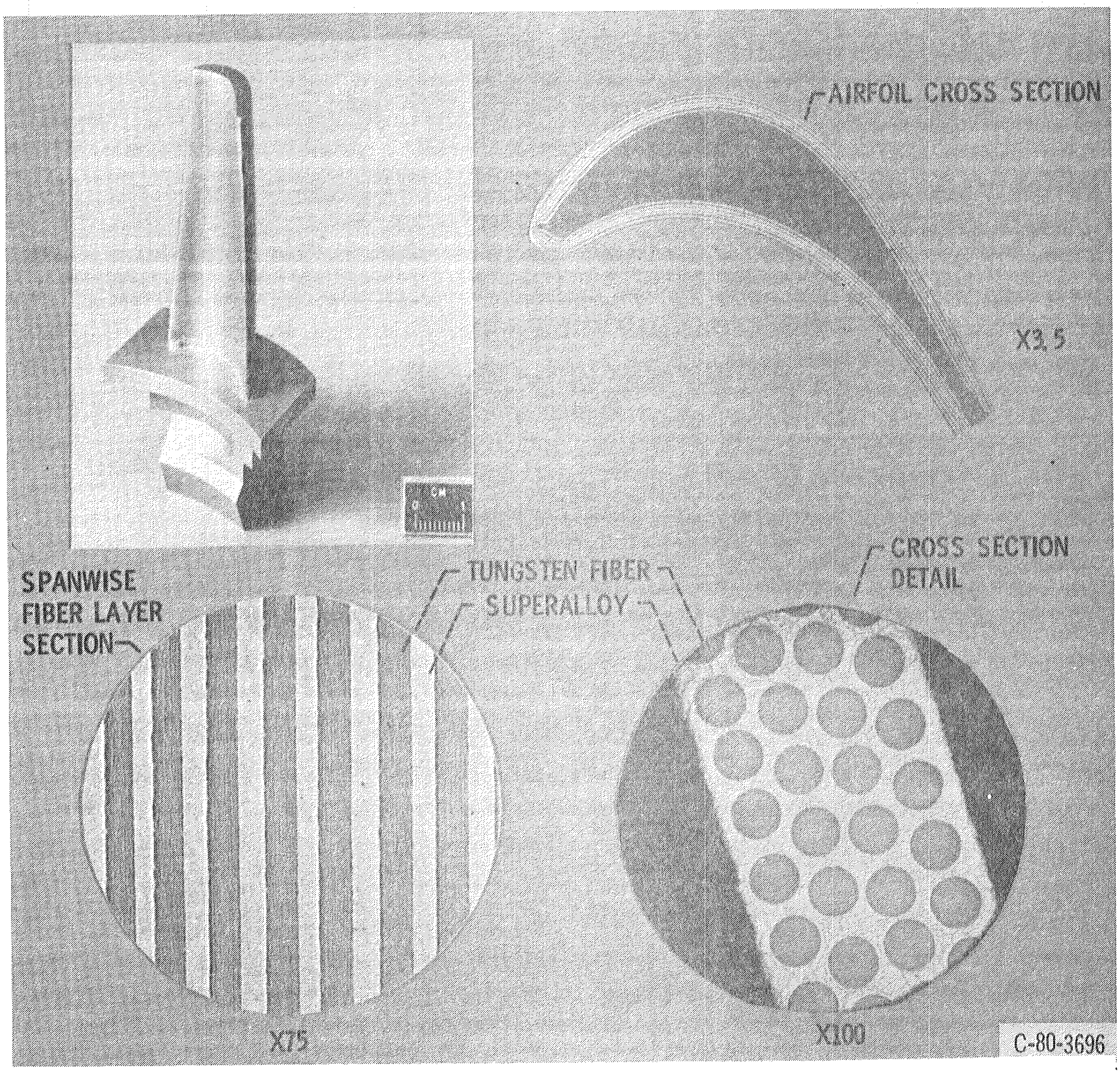



Figure 9. - Tungsten fiber/superalloy composite blade.

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16. Abstract

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