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Mechanical Behavior of Carbon-Carbon Composites

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SUMMARY

Mechanical testing and non-destructive inspection of carbon-carbon composite (C-C) materials is a concern of some researchers and this report reviews some techniques on coated and uncoated C-C at room and elevated temperatures. Also presented within this report are preliminary test results on a low modulus C-C material and a research plan to examine the effect of stress on the oxidation behavior of coated C-C. The tests reviewed include tensile, compression, shear, flexure, x-ray radiography, neutron radiography, ultrasonics, and fluorescent penetrant.

Carbon-carbon composite material consists of graphite fibers which are held together by a matrix of carbon char. The physical and mechanical properties of C-C can vary greatly and depends upon the precursor materials and the processing steps. These materials have the general characteristic of increases in strength with increases in temperature, but need a oxidation resistant coating or inhibitor for applications in oxidizing environments above 425^{o}C.

A readily available low modulus C-C material, K-Karb 'C' produced by Kaiser Aerotech, was examined for physical and mechanical properties to gain testing, characterization, and evaluation experience. X-ray radiography revealed thickness variations and the thinner areas were scrapped. Ultrasonic C-scan showed attenuation differences which did not correlate with any of the physical or mechanical properties measured. The density, 1.55 g/cc, was measured using a wax immersion technique, ASTM C 914. The hardness, 74 15W, was measured with a Rockwell Superficial Hardness tester on the 15W scale. Flexure tests were conducted at different support spans to obtain flexural and shear failures. The warp shear, warp flexural, and fill flexural strengths were 14.5 MPa(2100 psi), 137 MPa(19,800 psi), and 95.1 MPa (13,800 psi) respectively.

The stress oxidation test plan consists of initially characterizing coated and uncoated C-C materials with x-ray radiographs, metallography, and oxidation tests, and also for the density, flexural strength, and shear strength. Four point flexure tests will be conducted at the examination temperatures, 540^{o}, 820^{o}, 1090^{o}, and

1370°C. Next specimens will be stressed in the four point bending mode at the test temperatures in an oxidizing environment until failure to determine the static fatigue life. Specimens will then be exposed for a fraction of their static fatigue life and removed for microstructural examination and residual strength measurements. Finally high temperature-inert atmosphere four piont bend tests will be conducted to examine the influence of the atmosphere in a short term bend test.

MECHANICAL BEHAVIOR OF CARBON-CARBON COMPOSITE

Gary A. Rozak

ABSTRACT

A general background, test plan, and some results of preliminary examinations of a carbon-carbon composite material are presented with emphasis on mechanical testing and inspection techniques. Experience with testing and evaluation has been gained through tests of a low modulus carbon-carbon material, K-Karb 'C'*. The properties examined are the density - 1.55 g/cc; four point flexure strength in the warp - 137 MPa (19,800 psi) and the fill - 95.1 MPa (13,800 psi,) directions; and the warp interlaminar shear strength - 14.5 MPa (2100 psi). Radiographic evaluation revealed thickness variations and the thinner areas of the composite were scrapped. The ultrasonic C-scan showed attenuation variations, but these did not correspond to any of the physical and mechanical properties measured. Based on these initial tests and a survey of the literature, a plan has been devised to examine the effect of stress on the oxidation behavior, and the strength degradation of coated carbon-carbon composites. This plan will focus on static fatigue tests in the four point flexure mode in an elevated temperature, oxidizing environment.

INTRODUCTION

The use of composites in structural applications has increased markedly in recent years. Composites provide stable structural materials with high strength to weight ratios. Special purpose composites are used at high temperature. Carbon-carbon composites (C-C) are among these special purpose composites and

*K-Karb 'C' is a material produced by Kaiser Aerotech, San Leandro, California.

have the highest strength to weight ratio above 900°C. This material increases in strength with increasing temperature up to 2200°C[1] because of the improved coupling between the fiber and matrix. These properties make C-C attractive to the aerospace industry for use as a structural material for the hot section and exhausts of jet engines. The C-C materials would improve the efficiency by allowing operating temperature increases and decreases in the overall weight.

The major drawback of C-C is the lack of oxidation resistance; this requires some protection for use in oxidizing environments. The C-C begins to oxidize above 425°C without any protective coating or inhibitor. Many types of coating systems consisting of various carbides, nitrides, silicides, and metals have been applied by various methods. These methods include pack cementation, chemical vapor deposition (CVD), plasma spray, hot pressing, slurry coating following by sintering, and electrodeposition.[2] The mismatch of thermal expansion between a brittle ceramic coating and C-C causes coating cracks which may lead to oxidation of the substrate. Ductile metal coatings, e.g. platinum, have been explored to overcome this failure mode, but the underlying C-C substrate is susceptible to pinhole oxidation.

The coating system which has attained the most success is the pack cementation conversion of the composites surfaces to silicon carbide followed by overlay of glass sealers. These sealers are activated at about 760°C and flow into the cracks to block oxidation. Below this temperature the sealers are too viscous to flow and an oxidation window exists between 425° and 760°C. The composition of the glass sealer could be adjusted to allow sealing in this window, but at the expense of higher temperature protection.

The application of C-C materials has been successful in nose cones and exhaust nozzles of missiles, jet aircraft brakes, and in coated form, for the nose cone and leading edge on the Space Shuttle. Applications of coated C-C materials in the exhaust nozzles of jet engines are being pursued. Such

applications require a greater understanding of the coating-substrate mechanical and chemical behavior.

The objectives of this research effort are to increase knowledge of how applied stress influences the oxidation behavior, and the determination of mechanical degradation mechanisms of coated C-C at elevated temperatures in an oxidizing environment. A test plan has been developed to guide this effort and achieve these objectives. Initially coated C-C specimens will be stressed until failure in the four point flexure mode at elevated temperatures in an oxidizing environment. Next, coated specimens will be exposed to this stress and environment and removed prior to failure for microstructural examination and measurement of residual flexure strength. Initial four point bend tests have been conducted to obtain testing and evaluation experience. This knowledge and background will assist in the conduct and evaluation of engine performance and burner rig tests of coated C-C at NASA Lewis Research Center.

BACKGROUND

Carbon-carbon can be fabricated by a number of different processes which yield different composite properties. Carbon-carbon evolved from polymer matrix-graphite fiber composites.[2] The matrix of these materials carbonizes at elevated temperatures to a carbon char. Subsequent reimpregnations with the polymer precurser; followed by a pyrolization step produced C-C.

The graphite fibers in composite materials are fabricated from organic precursors, e.g., rayon, or polyacrylonitrile (PAN) in the form of yarns.[4,5] These yarns can contain up to 6000 filaments with individual diameters of between four and twelve microns.[5] Processing these bundles into graphitic fibers occurs with heat treating steps of carbonization and graphitization. Variations in these steps can yield a modulus of elasticity between 2.76 and 517 GPa (4 to 75 x 10^6 psi).[5] The fiber modulus in C-C may decrease because of subsequent higher composite processing temperatures and residual stress.[4]

The lay up of the yarns can be one, two, or three dimensional. Unidirectional tapes or plies, and two-dimensional woven fabric laminates provide no interlocking between layers and consequently the interlaminar shear strengths are very low. Achieving this desirable multilayer interlocking is possible through complex multi-directional weaving patterns as employed in 3-D composites.

For the 2-D woven fabric laminate the types of commonly available weaves are the plain, 5-harness and 8-harness. The harness variety refers to the crossover spacing as shown for the 5-harness weave in Figure 1. Two directions are relevant in a woven laminate, the warp and fill. Figures 1 and 2 illustrate these directions. The composite is stronger in the warp direction because the yarns are straighter and generally have a greater volume fraction.

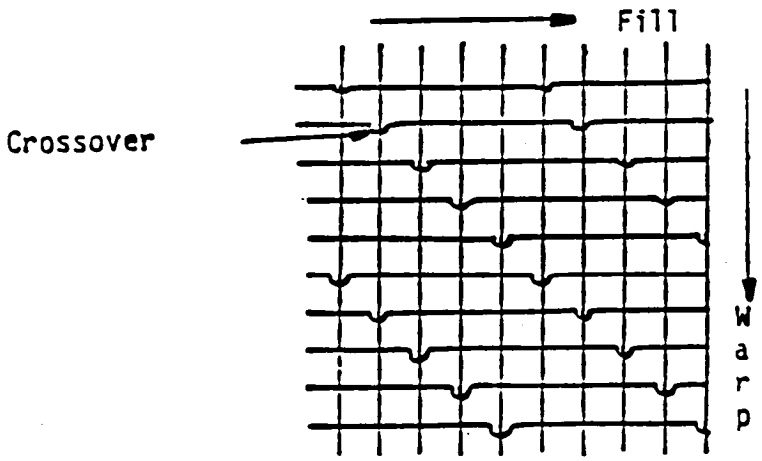


Figure 1 - Five Harness Weave

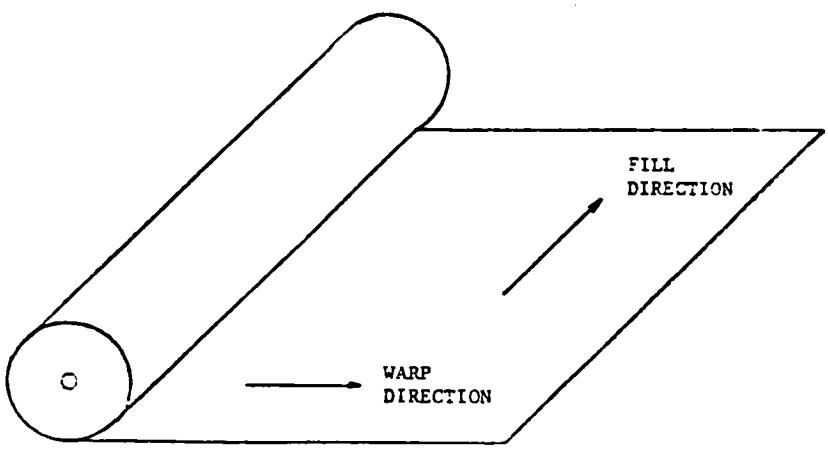


Figure 2 - Direction Explanation

The organic materials such as phenolics and epoxy resins, furfuryl alcohol and coal-tar pitch (CTP) are the common matrix precursor because they have a high char yield.[4] Pyrolytic carbon deposited by CVD is also used to form or densify the matrix.[3,4] The strength of C-C, especially in shear, increases with the number of impregnations, but the processing time and cost also increase. The matrix initially forms as amorphous carbon during the carbonization and pyrolization step. A further heat treatment converts this carbon into graphite for a CTP precursor, but the phenolic, epoxy-novalac, and furane resins remain amorphous after this treatment. The furfuryl alcohol matrix precursor graphitizes only locally.[4] The amorphous matrix generally offers a higher strength than the graphitic matrix.

TESTING C-C

A relatively complete evaluation of C-C properties are required for their optimal utilization. Confirmed mechanical properties are necessary for the design of structural members while quality control, environment simulation, and performance testing are important for C-C implementation. The oxidation resistance of coated C-C must be included in all investigations of these materials because of their use at elevated temperatures. The critical mechanical properties for design are the tensile, compression, shear, bearing, and transverse tensile strengths. The flexural properties of C-C are required to determine the deformation of this material under stress. The limiting design property is often the interlaminar shear strength because this value is low and interlaminar failure must be avoided.

Tensile Test

The tensile test is used to obtain the engineering properties of a material, e.g., ultimate strength, ultimate strain, elastic modulus, and Poisson's ratio. No standard tensile test exists for C-C materials but a few ASTM standards such as C297,[2] C749,[6] and D638[7] are guides to conducting these tests. The

basic specimen configuration is the flat dogbone shape. The tensile test may present problems because of shear failures at the shoulder or grips[1,7,8] of the specimen instead of within the gage length. Increases in the shear strength and the fillet radius at the shoulder,[8] and decreasing the thickness and width in the gage section may overcome these problems. The elastic strain is measured with clip-on extensometers[1,2,6] or electrical resistance strain gages.[2,8,]

The gripping configuration for loading the specimen is critical in the conduct of a successful tensile test. The commonly used gripping systems include: friction grips,[2,8,] pin loading, doublers,[8] and collet grips.[6,9] Pin loading is the only system that does not transfer the load to the surface of the specimen as shear. Doublers are sheet metal tabs which are attached to the specimens with glue or mechanical devices at the opposite ends of the gage length. The load is applied through these sheet metal tabs. Collet gripping is a type of friction grip similar to the jaws on a lathe. Friction grips clamp onto the specimen and transfer the load by friction. When any of these types of grips are employed, the crushing strength of the material must not be exceeded.

Elevated temperature tensile tests of C-C have been conducted by several investigators.[1,2,6] Such tests require long specimens or a high temperature load train. For short term tests these configurations are adequate, but for extended duration tests, such as creep or fatigue, the environmental resistance of the C-C may breakdown, and failures may occur outside the gage section.

Starret, et. al.[6] examined the tensile properties of 3-D C-C at 1090°C and determined they were similar to those at room temperature. The Aerojet Solid Propulsion Company[1] tested many different C-C; composites their maximum testing temperature was limited to 1650°C because of failures outside the gage area.

Schmid[2] conducted tensile tests on coated and uncoated C-C up to 1650°C. The two types of material he tested were the Hitco (CVD matrix) and Vought's Advanced Carbon-Carbon with four imprenations (ACC-4). The Hitco materials were

uncoated, and the tensile strengths and moduli at 1090°C were somewhat lower than ambient values. The decrease in strength is inconsistent with the knowledge about C-C increasing in strength with temperature. This deviation was explained as statistical scatter. The ACC-4 material was also tested in the uncoated condition, and the strength increased with temperature up to 1650°C in both fill and warp directions. The ACC-4 material was coated with a conversion coat of silicon carbide plus a slurry overlay in the gage area, but not in the grip areas. When the dimensions of these tensile specimen were considered, 40% of the load bearing area was coating and 60% was C-C substrate. Since the coating weakens a the C-C, there were observed descrese in the apparent strength of 63% and the apparent modulus by 30%.[2]

Compression Tests

Guides for the compression testing of C-C materials are the ASTM Standards D695,[6,7,8] C365,[1,2,7,9] C365,[2] and C364.[2] Specimen geometries are either flat rectangular[8] or dogbone[1,2,7,9] shaped, with lateral support of the gage section. Gripping is accomplished by wedge grips,[1] or adhesive bonding.[7] The compressive strength of C-C increases with temperature up to 2200°C.[1] The coating does not change the compressive strength and the across-ply compressive strength is similar to that in the in-plane directions.[2]

Flexure Tests

The flexure test, using three or four point bending, is a very common mechanical test performed on C-C; ASTM standards C651[6] and D790[7,11] are procedural guides. Determination of the failure mode in four point flexure prior to testing is necessary to conduct this test properly and expounded in Appendix A. This test is popular because:[1] 1) specimens are easily machined; 2) no fixture or gripping complexities occur; 3) the resultant properties are reproducible between laboratories; 4) many tests can be run quickly and inexpensively; and, 5) the loading simulates actual loads experienced in many C-C applications.

Some problems arise from the test results for the following reasons: 1) nonhomegeneous cross section may invalidate the linear beam theory for calculation purposes; 2) a high ratio of tensile to shear strength and moduli may lead to shear failures and lower apparent moduli; 3) the calculated tensile strength obtained may be significantly higher than that for the tensile test; and, 4) the coated specimen may not have equal moduli in tension and compression, thus cause a shift of the neutral axis from the center line. Proper interpretation and reporting of the types of failure modes obtained is needed to avoid improper analysis of the test.

Aerojet[1] tested a number of C-C in flexure. Generally the flexure strengths are greater than those in either tension or compression. Three explanations of this phenomena are considered: 1) the statistical strength theory predicts lower strengths for a larger volume of a material at maximum stress; 2) the friction at the loading point generates a bending moment which opposes the applied load; and, 3) the stress/strain curve may not be linear. Corrections are necessary in the modulus calculation to account for shear deformation,[2] and for strains greater than 5%.[12]

The high modulus materials failed in flexure by compressive buckling between the load points for uncoated specimens,[2] and for coated specimen by tensile cracking[2] or fibrous failures.[13] When span-to-thickness ratios were too small, shear failures were experienced.[2] A thick silicon carbide coating enhanced the flexure properties, but a thin coating had no effect.[13] When data were reduced using the external coated specimen dimensions, the strength at ambient temperature decreased because of the coating; no effect was observed at elevated temperatures.[2]

Shear Tests

Guides for conducting shear tests of C-C materials are the ASTM standards C-273,[2] D2344,[2] D3039,[1] and D3518.[1] The types of shear strengths measured are

the across-ply and interlaminar shear strengths with the across-ply being five times greater than the interlaminar.[1] Short beam shear, SBS, or three point bending with shear failure is a popular test for determining the interlaminar shear strength.[2,7,11] The double shear test,[8] as shown in Figure 3, is similar to SBS, but contact loading is employed instead of point loading.

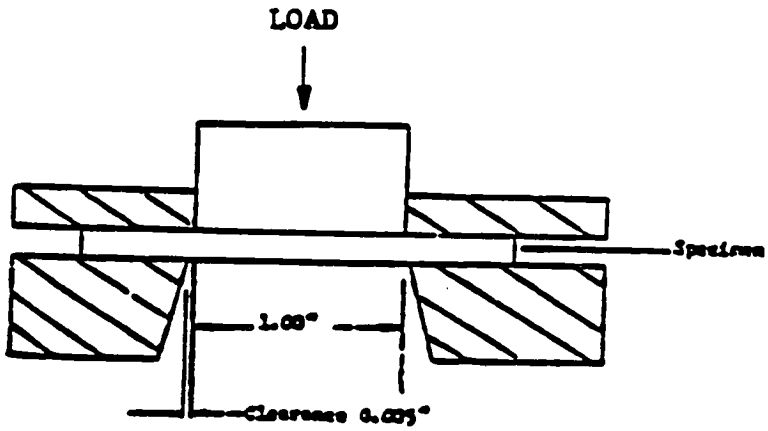


Figure 3 - Double Shear Test (Ref. 8)

A double notched shear specimen,[1,10,11] as shown in Figure 4, is another configuration for the measurement of interlaminar shear properties. An analysis of this test indicated that the apparent shear strength depended on the notch spacing.[9]

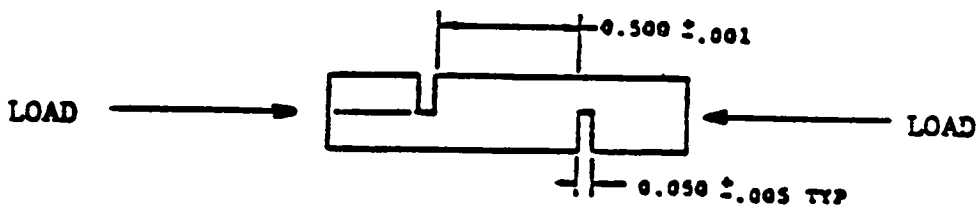


Figure 4 - Double Notch Shear Specimen (Ref. 8)

The button torsion test[2] measures the shear strength from the amount of twist and the applied load at failure. The across-ply shear strength test uses a pin shear specimen and is loaded as shown in Figure 5.

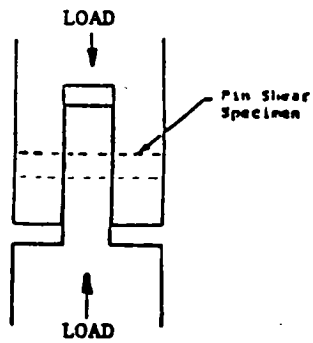


Figure 5 - Across-Ply Shear Test (Ref. 8)

No data reduction equations are available for the double notch shear nor the across ply shear tests.

Bearing Test

The bearing test is used to define the strength that could be obtained with a simple mechanical attachment, such as a rivet or bolt. The ASTM Standard D953[1] and the double lap shear test[2] (Fig. 6) guide the conduction of these tests.

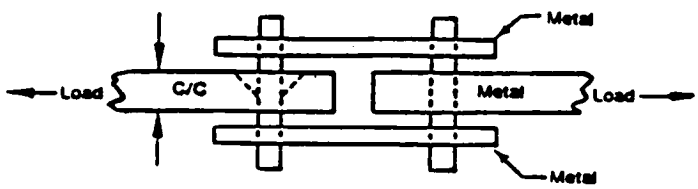


Figure 6 - Double Lap Shear Test (Ref. 2)

Modulus Test

The modulus is usually measured in the same test as strength, with the strain increments being measured with strain gages or clip-on extensometers. With C-C materials, it may be necessary to measure the strength and moduli in a different test because the high strength and directionality require a small gage size, but the high modulus requires a larger gage size to measure small increments of strain. The flexure moduli can be measured with a sonic-resonance technique[13] (ASTM C-747). A rectangular specimen is sonically excited in the

flexural mode and the resultant vibrational frequency is proportional to the flexural modulus[13] in this latter test.

Dynamic Tests

Dynamic response of C-C are necessary for the final design properties. These tests include fatigue, creep, impact, fracture toughness, stress rupture, and stress oxidation. Impact testing has been conducted as a Charpy type test for 3-D C-C,[6] but no data have been located for 2-D material. Stress oxidation and flexural fatigue tests have also been conducted.[10,11] Fullerton, et al.[11] conducted stress oxidation tests in the four point bending mode either at a constant stress or a constant strain. Specimens of Vought's low modulus reinforced C-C with 3 impregnations (RCC-3) were permanently deformed after five hour tests at 870° and 1090°C. The strain level for these tests was set at the proportional limit. No detectable loss of substrate was observed after this test by either weight change or x-ray radiographic measurements.

In the flexural fatigue tests,[11] RCC-3 specimens were cyclically loaded until a failure in the coating occurred. This failure would lead to substrate oxidation at 870°C. Detection of the critical failure occurred when the mid-span deflection increased. Additional dynamic test data and other dynamic tests are necessary for future applications of C-C.

Inspection Techniques

The types of non-destructive test techniques used to evaluate C-C materials are x-ray radiography including computer aided tomography, neutron radiography, ultrasonic, fluorescent penetrant, and visual inspections. For neutron radiography the composite must be infiltrated with a neutron absorber and thus this technique is not as sensitive as x-ray radiography for detecting flaws.[8] X-Rays are used to detect substrate defects and to assess density uniformity; they also find use for inspection of the coating thickness, uniformity, and oxidation

modes.[2]

The types of ultrasonic techniques include C-scan, pulse-echo, and velocity measurements. The C-scan is a through-transmission technique which measures the attenuation or absorption of the ultrasonic wave. The pulse echo technique records all the back reflected waves and reveals the internal interfaces. This technique does not produce any readily useful information for C-C because of reflections from every ply. Results[8] of ultrasonic measurements on unidirectional C-C showed trends of lower attentuation and higher velocities which corresponded to higher shear and compressive strengths. Higher attenuation and lower velocity tends indicated higher tensile strengths. Fullerton, et al.[11] found C-scan evaluations with 2-D C-C yielded inconsistent results.

Fluorescent penetrant inspection (FPI) is used to define the larger cracks in the coating which may lead to spalling or oxidation. It is difficult to find smaller cracks and pinholes. Visual inspection of the coating is used to detect major defects. The severity of a coating defect on the oxidation resistance cannot be ascertained through FPI or visual inspection.[11]

PLANS AND STATUS

When designing the hot section of gas turbine engines with coated C-C, the oxidation response of the material to stress must be addressed. Our research will examine this effect using the four point flexural mode for static fatigue and stress oxidation tests. High and low modulus C-C (ACC-4 and RCC-3 or equivalent) materials in the coated and uncoated condition are on order for these experiments. Upon arrival of the specimens, characterization tests will be performed for the shear and flexure strengths, density, and oxidation resistance. The materials will be further characterized with x-ray radiography and micrographic examination.

The examination temperatures will be 540°, 820°, 1090°, and 1370°C since this is the range of interest for oxidation effects in gas turbine exhaust

nozzles. Four point bend tests will be conducted at each of these temperatures determine the material strength at temperature.

Static fatigue tests of the C-C materials at each test temperature will be conducted in the four point flexure mode with dead weight loading. The test is called static fatigue because this is a delayed-failure phenomenon in a constant environment. The initial stress level in the specimen will be one half the flexural strength determined at each test temperature. This level was chosen because C-C is often times designed with a safety factor of two. The first test will be conducted until failure or for a maximum time of 240 hours. The stress level in subsequent tests will then be adjusted to obtain failure in approximately 150 hours.

In the stress oxidation segment, specimens will be loaded in four point flexure as in the static fatigue test for half of the previously determined static fatigue life for each test temperature. The stress level will also be established in the previous testing segment. Specimens will be examined for weight loss, microstructural degradation, and residual flexural strength at room and the test temperature. This section will comprise the bulk of the research.

Four point bend tests at each temperature will be run in an inert atmosphere for the final testing segment. Both coated and uncoated specimens will be tested to ascertain if there is an influence of the atmosphere on the failure mode and strength level. Twenty-five percent additional specimens are ordered, and if specimens remain after the initial test sequence is completed, static fatigue exposures will be run while cycling the temperature to simulate the temperature profile and expected loading in gas turbine exhaust systems.

Current Status

All necessary materials, fixturing, and equipment are at least in the procurement stage at the present time. The specimens will be ACC-4 and RCC-3 or similar materials, with dimensions of 19.7 cm long by 1.5 cm wide by 0.64 cm

thick (7.75 x 0.60 x .25 in.). This size is necessary to obtain flexural failures. A total 165 specimens of each material (28 noncoated and 137 coated) will be included. A silicon carbide conversion coat plus a slurry overlay of glass sealers will be the coating employed. Compression creep test frames with lever arm loading and a furnace will be used for static fatigue tests and exposures. An existing high temperature box furnace will be retrofitted for use in the elevated temperature four point bend test in a universal test machine. Four silicon carbide high temperature four point bend fixtures will be employed. Deflections in the static fatigue test will be monitored with a linear variable differential transformer (LVDT) and recorded on a hard disk by an IBM data acquisition system.

Six-Month Plan

During the time before arrival and installation of equipment and materials, it will be necessary to accomplish one task in order to execute the experimental plan. This is the procurement of coated flexural test specimens. These specimens will be used to develop the experimental procedures and eliminate initial system errors prior to focusing on the primary materials. Other tasks which will be performed are fractography of K-Karb 'C' flexural specimens, characterization and inspection of C-C divergent seals which are being tested at NASA Lewis, and mechanical testing and characterization of high modulus C-CAT C-C materials.

RESULTS AND DISCUSSION OF K-KARB 'C' TESTS

To aid the development of the above test plan, K-Karb 'C', a readily available low modulus carbon-carbon material, was characterized. The C-C panels were inspected with nondestructive techniques and examined for density and the mechanical properties of hardness, shear strength and flexure strength. The two non-destructive techniques were x-ray radiography and ultrasonic C-scan. The radiograph revealed the thinner or less dense areas. The C-scan, through-

thickness transmission technique, showed areas of greater attenuation on Panel 2 from 5 to 10 db (see Fig. 7), but these could not be correlated with any of the measured physical or mechanical properties.

The bulk density measurements were guided by ASTM C-914 wax immersion technique. The exact procedure are listed in Appendix B. The average density of the two panels was 1.55 g/cc. The density was measured near the center and at one corner and the values were 1.550 and 1.556 g/cc respectively, for Panel 1, and 1.550 and 1.553 g/cc respectively, for Panel 2. The center specimens in Panel 2 were removed above the precise center to obtain measurements in the less attenuated area revealed as light areas in the C-scan (Fig. 7).

The mechanical tests performed on K-Karb 'C' material were hardness and four point bending. The hardness test was conducted on a Rockwell Superficial Hardness Tester on the 15W scale with a 15 kilogram major load and a 1/8 inch ball. The procedural details are listed in Appendix B. The hardness values obtained ranged from 67.2 to 78.0 with the average near 74 15W. The hardness standard deviations from average value varied from 0.7 to 8.0 with most of the scatter about 2.5. All of the densities and hardness readings are listed in Table I of Appendix C.

Hardness readings were taken on each specimen side, perpendicular to the plies. An accounting of the specimen side with respect to the panel side was inadvertently not kept, and therefore, a comparison of hardness variation from side to side could not be made. Only the center specimens from Panel 1 indicated a possible side to side variation.

Four point flexure tests were conducted at three different support spans 4.76, 7.94, and 12.7 cm (1.88, 3.12, and 5.00 in.) and a constant load span of 2.54 cm. The short span was chosen to obtain shear failure and the longer spans to obtain flexural failures. The type of failure was predicted through the

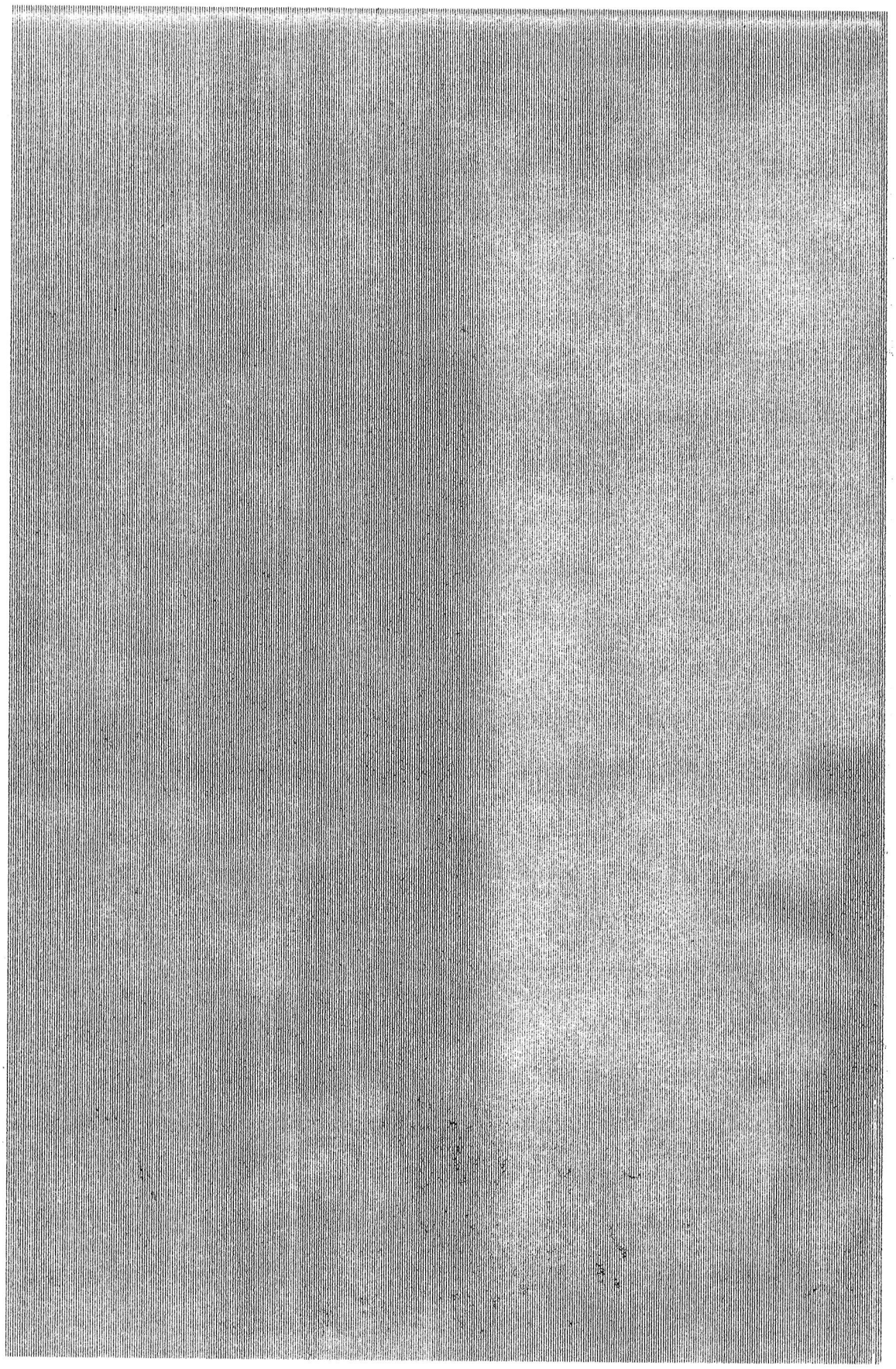


Figure 7 - C-scan of Panel 2

equations developed in Appendix A. Specimens were machined from Panel 2 according to Figure 8 and tested, following the procedures listed in Appendix B. The shortest span tests resulted in three types of shear failures: along the center line, between load and support points, and at the centerline only detectable with microstructural examination. At the longer spans lengths, all failures were flexural in tension between the load points. All stress values were calculated with linear beam theory. The shear strength was 14.7 MPa (2140 psi) in the warp directions; no shear strength in the fill direction was measured. At the 7.94 cm span, the strengths were 137 MPa (19,800 psi) for the warp direction and 95.1 MPa (13,800 psi) for the fill direction. At the 12.7 cm support span, only warp aligned specimens were tested and the strength was 130 MPa (18,800 psi). The test data from the individual tests are listed in Table II of Appendix C.

The failure mode of the K-Karb 'C' material, flexural or shear, is predicted from the beam theory equations developed in Appendix A. The maximum shear stress which occurs in a flexure test of K-Karb 'C' material is normalized against the shear strength and is plotted as a function of the effective span to thickness ratio in Figure 9. The test results of this material agree with the prediction:

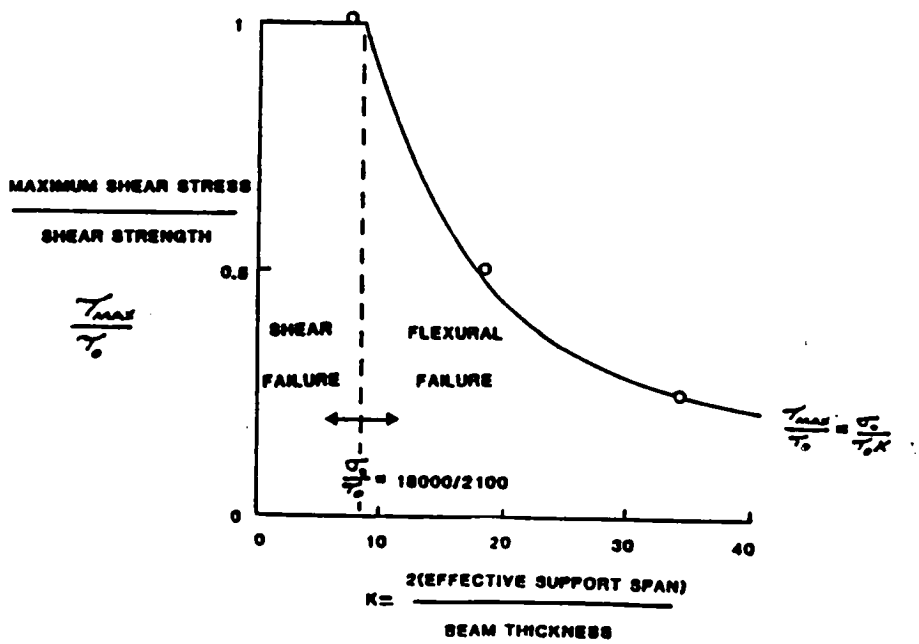


Fig. 9 - Maximum Shear Stress in a Flexure Test of K-Karb 'C' Material

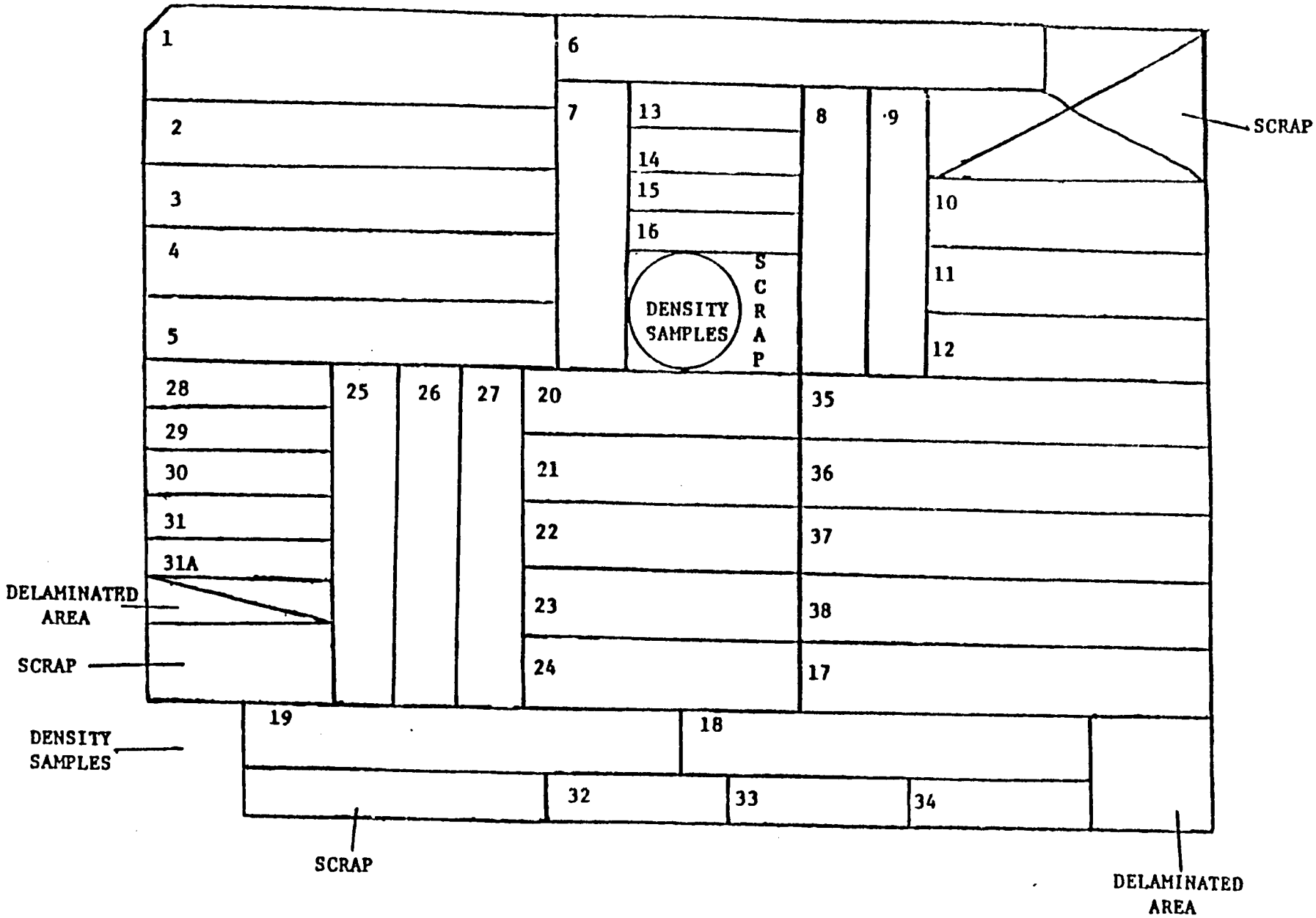


Figure 8 - Specimen lay-out, panel 2

Only Panel 2 of the K-Karb 'C' material was tested and the results from a C-Scan (Fig. 7) showed areas with a difference in attenuation. The C-scan guided the cutting of specimens (Fig. 8) from this panel. The flexural results obtained for the specimens in each of the two regions are compared in Table III of Appendix C, but no correlation existed between the strength and attenuation differences. The conclusions from the tests of the K-Karb 'C' material were that specimens from the panel edge had lower flexural strengths but not lower shear strengths. The shear failure mode for the edge specimens was different, because the shear damage at the centerline was only detectable with micrographic examination.

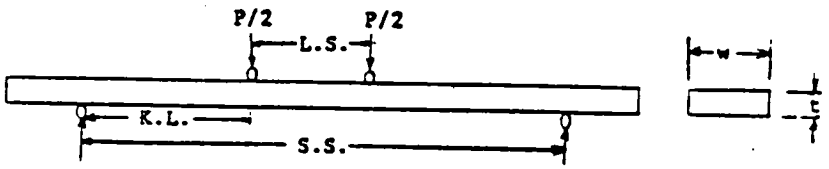
CONCLUDING REMARKS

The preliminary four point bend test results on low modulus carbon-carbon composite materials (C-C) agreed with predictions from linear beam theory. After this testing and a survey of C-C mechanical test methods, a plan has been developed to examine the effects of a constant stress at elevated temperatures in an oxidizing environment on coated C-C. This research effort will increase the understanding of coating/substrate behavior and yield degradation mechanisms of C-C in this type of environment.

APPENDIX A

Determination of Failure Mode in Four Point Bend Test

The ability to predict the failure mode in a four point flexure test is needed for the proper conduct of this test. The two significant types of failures are flexural and shear. Flexural failures occur in the outer layers where the maximum tensile and compressive stresses develop. Shear failures generally occur at the specimen center line where the shear stress is maximum. Linear beam theory allows the prediction of the failure mode to be made from the knowledge of a materials tensile and shear strengths, and the specimen and loading geometry as defined in Figure A-1.



where: P = Load
L.S.= Load Span
S.S.= Support Span
w = Width
t = Thickness
K.L.= $\frac{S.S.-L.S.}{2}$

Figure A-1 - Load and Specimen Geometry Nomenclature

The following beam theory equations assume that the neutral axis is the geometric center line. The maximum shear stress (max) in a beam under four point loading is given by equation (1).[14]

(equ. 1) $$\tau_{max} = \frac{Vt^2}{I8} = \frac{P/2\ t^2}{8\ \frac{wt^3}{12}} = \frac{3\ P}{4\ wt}$$

where: V = Shear Force
I = Moment of Inertia

The maximum flexural strength (σ max) is given by equation (2).[14]

(equ. 2) $$\sigma_{max} = \frac{Mc}{I} = \frac{P/2\ (K.L.)\ t/2}{\frac{wt^3}{12}} = \frac{3\ P\ (K.L.)}{wt^2}$$

where: M = Moment
c = Maximum Distance from Neutral Axis

Dividing equation (2) by (1) yields:

(equ. 3) $$\sigma_{max} = \frac{4\ K.L.}{t}\tau_{max} = \frac{2\ (ESS)}{t}\tau_{max}$$

ESS = Effective Support Span
ESS = SS - LS

Normalizing equation (3) against the shear strength of the material, and setting σ max equal to the flexural strength, gives:

(equ. 4) $$\frac{\tau_{max}}{\tau_o} = \frac{\sigma o}{\tau_o}\frac{t}{2\ ESS}$$

Where σo, and o, are the flexural and shear strength respectively. The plot of this equation shows the two failure regimes (Fig. A-2).

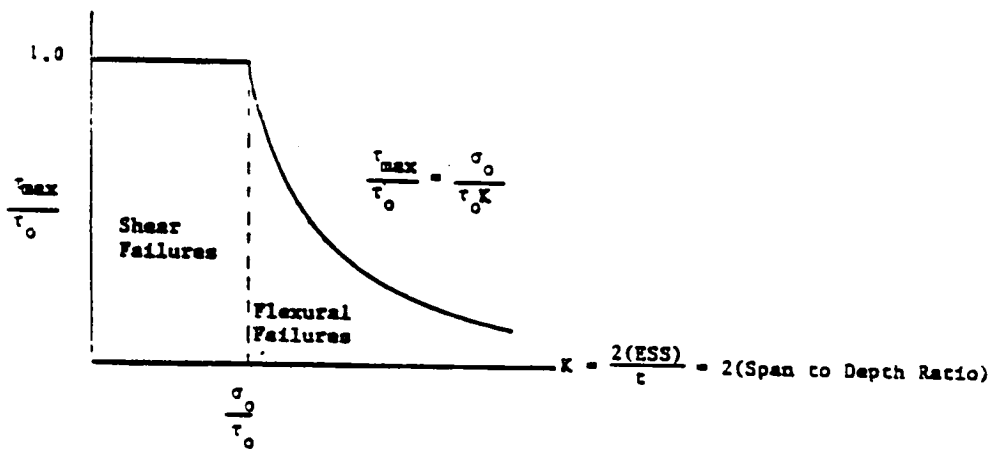


Figure A-2 - Failure Mode Prediction

Therefore, once the effect support span and thickness are set, and the strengths known, the failure mode can be predicted.

APPENDIX B

Procedure for Apparent Density Measurements

The density of K-Karb 'C' material was measured at the corner and near the center. A center coupon was cut with a hole saw and a corner with a silicon carbide saw. One quarter inch from the panel edge was trimmed off the corner coupon to eliminate any edge effect. The coupons were then cut into four approximately equivalent sizes. These specimens were then dried in an oven to a constant mass. Next, two-thirds of a specimen was coated with wax by dipping it into liquid paraffin, and then the remaining third of a specimen was wax coated with some overlapping. Then the mass of the coated specimen was measured. The coated specimen was totally immersed in ethanol and the suspended mass was measured. The ethanol temperature was recorded at each measurement. The liquid density was then determined by interpolating from a temperature-density chart in the CRC Handbook.* The following calculation was used to determine the apparent density.

$$\text{Specimen Density} = \frac{SM}{\frac{CSM-CIM}{L} - \frac{CSM-SM}{W}}$$

SM = Uncoated suspended mass

CSM= Coated suspended mass

CIM= Coated immersed mass

L = Density of liquid

W = Density of wax

The nomenclature for the specimens listed in Table I, Appendix C are the following: the first number identifies the panel, the first letter distinguishes specimens in the same group, and in the final position a blank indicates the corner and a 'C' indicates the center.

Procedures for Hardness Measurements

The wax on the density coupons was removed by wrapping coupons in a paper towel and placing it in a oven at 80°C for 16 hours. Both ply surfaces were

*CRC - Handbook of Chemistry and Physics, R. C. Weast, ed., 60th edition, 1979-80, pg. F-6.

ground smooth with 320, then 600 grit silicon carbide paper. A Rockwell Superficial Hardness Tester with a 15 kilogram major load and 1/8 inch ball indentor (15W scale) was calibrated to a 30T hardness scale calibration block with a hardness of 44.2 30T measured 84.7 on the 15W scale. The minor load (3kg) was applied, then the major load and held for ten seconds. The coupon surface was carefully watched for loose fiber motion leading to a invalid reading. The dial was also watched for too deep of penetration which invalidates a reading.

Procedure for the Flexure Test

The C-C panel was cut into smaller, more manageable sizes with a silicon carbide saw. These were then cut into specimens with a diamond saw. Water was used as a lubricant and to minimize dust. The specimens were dried in an oven at 100°C for twenty hours. The dimensions of width and thickness were measured with a vernier caliper at a few spots in the midspan to within ± 0.003 cm. The average dimension was recorded. The specimens were kept in envelopes and equilibrated in the test room overnight.

A universal test machine with a one thousand pound load cell under the testing platforms was used for the flexure test. The output of this load cell was connected to a servo-driven pen and was recorded versus crosshead displacement on a strip chart recorder. The load cell was calibrated at twenty pounds full scale deflection with twenty pounds of dead weight.

The specimen and test fixture were aligned with the following procedure: the specimen was set on the support points of the fixture, and an aluminum alignment block was set squarely along the fixture length. Two 1/4 inch bolts were positioned in the block so that the loading fixture fit between the bolts and was precisely centered between the two support points. The specimen was optically aligned parallel with the fixture.

The fixture and the alignment device were moved to the testing platform on a universal testing machine, and this device was carefully removed. The mass of

the fixture and specimen was zeroed on the strip chart recorder on the most sensitive scale, so that only the applied load was recorded. The upper ram of the load train was lowered manually until a preload of two pounds was read on the chart paper, and then raised to removed the preload. The load train speed was set at 0.13 centimeters/minute (.05 inches/mintue).

The load train was set into motion and stopped after a catastrophic failure, and the type of failure was noted. The data was reduced with linear beam theory equations:

$$\text{Flexure Stress} = 3 \cdot \text{Load} \cdot \text{ESS}/(2\ wt^2)$$

$$\text{Shear Stress} = 3 \cdot \text{Load}/(4\ wt)$$

Where ESS, w, and t were the effective support span, specimen width and specimen thickness respectively. The effective support span is the support span length less the load span length.

APPENDIX C – TABLES

TABLE I

BULK DENSITY AND HARDNESS VALUES FOR K-KARB 'C' C-C

Specimen	Density g/cc	Average Hardness 15W	HARDNESS READINGS 1	2	3	4	5
1A	1.572						
1B	1.567						
1C	1.560	78.0	74.4	77.1	77.8	79.6	81.0
1D	1.563	74.4 75.3 76.9	69.8 78.7 76.1	74.1 72.8 79.3	75.1 76.0 75.1	74.2 73.0 77.0	75 76.2 77.2
MEAN	1.566	76.2					
1AC	1.545						
1BC	1.552						
1CC	1.554	73.3	75.0	68.4	77.0	74.1	71.8
1DC	1.549	67.7 71.3 67.2	74.0 72.8 55.7	67.0 72.2 76.0	59.1 75.0 73.1	65.0 71.3 67.1	73.4 67.3 64.0
MEAN	1.550	72.6/67.4					
2A	1.551						
2B	1.554						
2C	1.550	72.0	71.7	67.3	74.7	72.8	73.6
2D	1.555	76.7 73.5 72.5	76.0 70.9 74.1	77.1 75.1 70.2	76.5 72.8 72.0	76.2 77.7 72.0	77.6 71.0 74.1
MEAN	1.553	73.7					
2AC	1.548						
2BC	1.546						
2CC	1.556	74.6	75.6	69.4	77.7	72.3	78.2
2DC	1.551	74.4 73.6 77.8	84.1 71.9 82.1	70.9 74.0 78.0	74.4 74.5 85.2	72.0 73.9 72.1	70.8 59.2* 71.7
MEAN	1.550	75.1					
MEAN	1.555	75.0					

*Not included in average.

TABLE I:

FOUR POINT FLEXURE TEST DATA FOR PANEL 2

12.7 Centimeter Support Span, Warp Orientation, Flexural Failure

Sample Number	Flexure Strength (MPa)	Shear Stress at Failure (MPa)	Flexural Modulus (GPa)	Deflection at Failure (cm)	Load at Failure (N)	Thickness (cm)	Width (cm)	Comment on C-Scan
1*	113	3.16	13.4	.472	49.9	.566	2.05	Greater Attentuation
2	122	3.43	13.0	.528	54.0	.569	2.03	
3	124	3.50	13.4	.513	55.4	.572	2.04	
4	130	3.66	13.5	.536	58.1	.572	2.04	
5	126	3.58	13.6	.508	57.2	.577	2.04	
6*	120	3.52	13.6	.467	58.6	.597	2.05	Less Attentuation
Average	126	3.53				.572	2.04	
18	144	4.13	12.5	.620	68.1	.589	2.03	Less Attentuation
19	130	3.79	13.0	.521	59.9	.589	2.05	
36	125	3.58	13.4	.509	61.3	.584	2.15	
37	130	3.72	13.1	.541	62.7	.579	2.15	
38	136	3.86	13.5	.551	64.9	.577	2.15	
Average	133	3.82	13.1			.584	2.11	
Total Average (For 12.7 cm Support Span)	130	3.69	13.3			.579	2.08	

7.94 cm Support Span, Warp Orientation, Flexural Failure

11	138	7.58	15.2	.226	111	.597	1.79	Greater Attentuation
12	142	7.85	14.2	.234	114	.597	1.78	
Average	140		14.7			.597	1.79	
21	128	6.89	14.9	.206	117	.582	2.15	Less Attentuation
22	132	7.17	14.7	.214	121	.582	2.15	
23	133	7.30	14.6	.211	126	.592	2.14	
24	145	7.99	14.3	.231	140	.599	2.14	
Average	134		14.6			.589	2.14	
Total Average	136	7.44	1.46			.592		

*Edge Specimen, not included in the average.

TABLE II

FOUR POINT FLEXURE TEST DATA FOR PANEL 2 K-KARB 'C'
(Continued)

7.94 cm Support Span, Fill Orientation, Flexural Failures

Sample Number	Flexure Strength (MPa)	Shear Stress at Failure (MPa)	Flexural Modulus (GPa)	Deflection at Failure (cm)	Load at Failure (N)	Thickness (cm)	Width (cm)	Comment on C-Scan
7	101	5.37	13.0	.218	74.5	.571	1.78	Greater Attentuation
8	95.8	5.44	11.9	.198	75.5	.592	1.78	
9	96.5	5.37	12.3	.193	78.1	.599	1.78	
Average	97.8		12.4			.587	1.78	
25	92.5	4.82	12.1	.203	66.3	.592	1.78	Less Attentuation
26	93.0	4.96	11.4	.208	69.0	.574	1.78	
27	92.3	5.03	11.5	.203	70.8	.584	1.78	
Average	92.5		11.6			.574	1.78	
Total Average (For Fill, 7.94 cm Span)	95.1		12.0			.584	1.78	
4.76 cm Support Span, Warp Orientation, Shear Failures								
13	108	13.9	14.5	.0864	144	.572	1.33	Greater Attentuation
14	111	14.3	14.7	.0914	147	.572	1.33	
15	116	15.0	14.5	.0914	155	.574	1.33	
16	115	14.9	14.6	.0889	154	.574	1.33	
Average		14.5	14.5			.574	1.33	
28	120	15.2	15.0	.0864	153	.561	1.33	Less Attentuation
29	123	15.5	15.3	.0889	157	.561	1.33	
30	116	14.7	15.2	.0914	148	.559	1.33	
31	117	14.7	14.9	.0889	149	.559	1.33	
31A	118	14.8	14.7	.0889	149	.559	1.33	
Average		14.5	14.5			.574	1.33	
32*	116	15.4	14.5	.0864	197	.589	1.59	Edge Specimens
33*	116	15.5	13.0	.0864	201	.594	1.60	
34*	113	14.7	11.6	.0889	178	.577	1.54	
Average		15.2	13.0			.597	1.58	
Average (For Warp, 4.76 cm Span)		14.7	14.8			.566	1.33	

*Edge Specimens not included in the average

TABLE III

STRENGTH COMPARISON OF K-KARB 'C' FOR
AREAS WITH C-SCAN ATTENTUATION DIFFERENCES

			Strength			Cross Sectional		
Span cm	Support Orientation	Failure Type	5 db Attn. MPa	10 db Attn. MPa	Percent Difference	5 db Attn. cm	10 db Attn. cm	Percent Difference
4.76	warp	shear	15.0	14.5	-3.2%	0.742	0.761	+ 2.6%
7.94	fill	tensile	92.6	97.9	+5.7%	1.02	1.05	+ 2.5%
7.94	warp	tensile	134.0	140.0	+4.1%	1.26	1.06	-15.8%
12.7	warp	tensile	133.0	126.0	-5.4%	1.23	1.16	- 5.8%

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16. Abstract

A general background, test plan, and some results of preliminary examinations of a carbon-carbon composite material are presented with emphasis on mechanical testing and inspection techniques. Experience with testing and evaluation has been gained through tests of a low modulus carbon-carbon material, K-Karb 'C'*. The properties examined are the density - 1.55 g/cc; four point flexure strength in the warp - 137 MPa (19,800 psi) and the fill - 95.1 MPa (13,800 psi,) directions; and the warp interlaminar shear strength - 14.5 MPa (2100 psi). Radiographic evaluation revealed thickness variations and the thinner areas of the composite were scrapped. The ultrasonic C-scan showed attenuation variations, but these did not correspond to any of the physical and mechanical properties measured. Based on these initial tests and a survey of the literature, a plan has been devised to examine the effect of stress on the oxidation behavior, and the strength degradation of coated carbon-carbon composites. This plan will focus on static fatigue tests in the four point flexure mode in an elevated temperature, oxidizing environment.

*K-Karb 'C' is a material produced by Kaiser Aerotech, San Leandro, California.

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