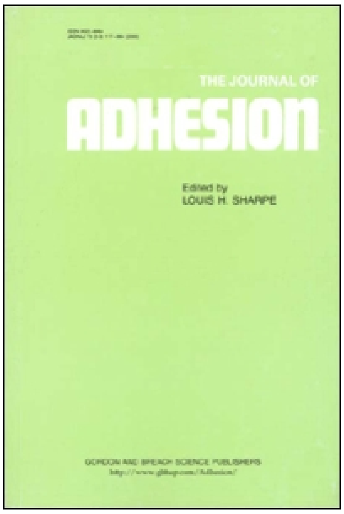

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The Effect of the Interface on the Impact Strength of Fiber-Reinforced Silicon Nitride Composites[†]

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Hot-pressed silicon nitride can offer large gains in gas turbine performance when used as a blade or vane provided the limitation of low impact strength can be overcome. Through the use of fiber reinforcement, energy absorption modes not available in monolithic materials are provided, with the result that the elevated temperature (1300°C) impact strength of an Si_3N_4 composite reinforced with 30 volume percent tungsten wires has been shown to increase ninefold over unreinforced Si_3N_4. To make use of the energy absorbing mechanisms of fiber pullout and/or interfacial splitting, one needs to retain the filament strength and have a fairly weak or low modulus interface or interfacial region between the tungsten and the silicon nitride. At elevated temperatures, this is the case; however, at room temperature the tungsten silicide layer formed at the W–Si_3N_4 interface during hot pressing has been found to render the tungsten reinforcement ineffective as a crack blunting constituent. Efforts are being made to prevent the formation of tungsten silicide through the use of interfacial barrier coatings and/or reducing the fabrication temperature.

I INTRODUCTION

Ceramic materials, by virtue of their high melting points and oxidation resistance, can offer large gains in turbine performance, provided some serious limitations can be overcome. These are poor thermal shock resistance and, more critically, low impact strength. It is the solution of the latter problem that UARL has emphasized in its efforts to employ normally

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brittle materials in aircraft gas turbines. The most promising solution, we believe, is through the use of fiber or wire reinforcements. This approach permits the strain-to-failure of the composite to be maximized by thermal prestressing and provides energy absorption modes not available in monolithic materials, provided that the interfacial bond strength between fiber and matrix can be controlled. By tailoring the properties of the composite it is possible not only to maximize energy absorption during impact but to control the type of fracture as well.

For the past two years we have been involved in a program to study the impact behavior of fiber-resin composites, primarily boron and graphite reinforced epoxy.[1,2] One of the major objectives of the program was to determine the energy absorbing mechanisms in the composites, and, using this information, improve their impact resistance by proper material selection and/or modification. Of course, the temperature capability of resin matrix composites is greatly different from that of ceramic matrix composites, but the mechanical behavior of the two systems is quite similar, i.e. relatively brittle, low failure strain. Consequently, it was believed that much of what was learned regarding impact behavior of resin composites could be applied to ceramic composites.

There are preliminary results which indicate that this transfer of technology is valid. The energy absorbing mechanisms found operative in resin composites are described in the following paragraphs along with comments on the applicability of the mechanisms in ceramic composites. Subsequent sections present the results of impact and modulus of rupture tests conducted on tungsten-silicon nitride composites and discuss techniques for further improvement of these properties.

II COMPOSITE IMPACT CONSIDERATIONS

Four mechanisms of energy absorption were considered in analyzing the impact strength of resin matrix composites: fiber pullout, fiber fracture, shear delamination, and interfacial splitting. These are discussed in turn below.

Fiber pullout

This mechanism has been described in the literature.[3,4] It has been experimentally observed that in certain composite systems, fibers fracture away from the plane of an advancing crack due to the presence of flaws, then subsequently pull out of the matrix as the crack progresses. The energy absorbed in the pullout process is:

$$W = \frac{v_f \sigma_f^2 d_f}{24\tau} \times A \tag{1}$$

where v_f = fiber volume fraction
σ_f = fiber tensile strength
d_f = fiber diameter
A = specimen cross sectional area
τ = interfacial shear strength.

Pullout was found to be an important mechanism in energy absorption for graphite-epoxy, but did not occur to a great extent in boron-epoxy. Initial experimental work at UARL indicates that pullout can occur in ceramic composites. Figure 1 shows a fractured specimen of W–SiO_2. The pullout of fibers in this composite is obvious.

The important material properties which affect the contribution of the pullout mechanism are the fiber tensile strength and the interfacial shear strength. The latter fact implies that any chemical interaction between the

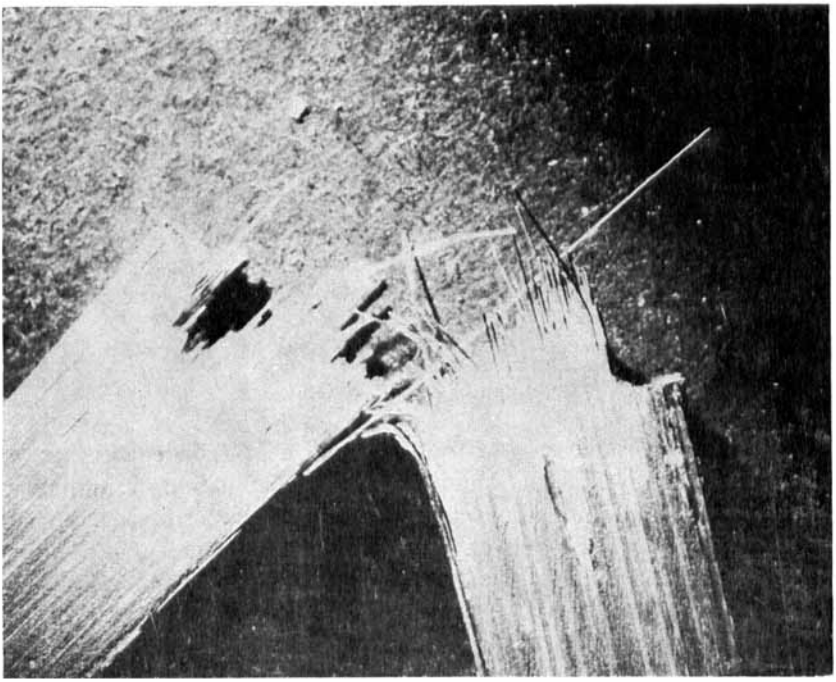


FIGURE 1 Tungsten-silica impact specimen—11 percent fiber volume.

matrix and the fibers which strengthens the interface will be detrimental to the composite impact strength. As shall be subsequently shown, this becomes a real problem in high temperature ceramic composites.

Fiber fracture

A second mechanism found operative in resin composites, termed fiber fracture, accounted for the energy required to load up and fracture the fibers. The energy required to load and fracture the matrix was neglected because of the relatively low strength of resins. The equation for fiber fracture energy in the Charpy impact specimen is:

$$W = \frac{v_f \sigma_f^2}{6E_f} \times V \tag{2}$$

where V = specimen volume
E_f = fiber modulus.

For the ceramic composites of interest, the contribution of the matrix to the fracture energy of the composite cannot be neglected, and consequently, Eq. (2) should be modified as follows:

$$W = \frac{\sigma_c^2 V}{6E_c} \tag{3}$$

where the subscript c denotes composite properties.

This equation essentially determines the average area under the composite bending stress-strain curve when the impact specimen fails at its center. It is obvious that increasing the composite strength or decreasing its modulus would increase the fracture energy contribution.

Delamination

The third and final mechanism found operative in resin composites was delamination. All specimens tested underwent delamination during impact, with the number of delaminations depending on the composite shear strength. Observation of slow bend tests of Charpy specimens revealed that delamination was the initial failure event; the specimens behaved much like short beam shear specimens with a span-to-depth ratio of approximately 4:1. Since the delamination appeared to be caused by shear stresses, an equation was developed to calculate the shear fracture energy for the composite based on the area under the composite shear stress-strain curve.

$$W = \frac{n\tau^2 V}{6G} \tag{4}$$

where n = number of delaminations
G = composite shear modulus.

Shear delamination does not occur in the SiO_2 matrix composites which have been tested in our laboratories (Figure 1). However, the possibility of delamination in ceramic composites exists, if a low enough interfacial bond strength can be achieved. It must be kept in mind, however, that too low a bond strength will result in a composite that simply falls apart on impact with no impact strength.

Interfacial splitting

The development of a low interfacial bond strength opens up the possibility of a fourth energy abosrbing mechanism which was found difficult to assess in resin composites. Interfacial splitting as described in Ref. 5 can occur in composites having two perfectly elastic phases, given a relatively weak interface (about $\frac{1}{5}$ the strength of the fiber). The occurrence of interface splitting results in the blunting of cracks which propagate in the matrix, and could lead to notch insensitivity in the material. This mechanism did not seem to occur in resin composites, possibly because the resin matrix is not perfectly elastic and therefore the stress states and the conditions under which splitting would result were different from those described in the literature. Also, in these composites fibers fail before the matrix, and this changes the situation from that described in Ref. 5.

Composite impact strength can be calculated based on the composite fracture and fiber pullout mechanisms. This has been done for W–SiO_2 composites and compared with our experimental data as shown in Figure 2. The fiber pullout contribution was calculated using an assumed shear strength of 25 ksi. Most of the data are in good agreement with the prediction, with the exception of the specimen with 30 percent fiber. A photograph of that particular specimen is presented in Figure 3, and shows that fiber fracture and pullout did not occur. Rather, there was extensive interfacial splitting, indicating that a weak interface existed in the composite. This may be a desirable mode of failure for vane materials since the composite fragmented into many small pieces rather than breaking into two large pieces. It is not surprising that the experimental impact strength did not agree with the predicted value, because the predicted mechanisms were not active. The fact that fiber fracture and pullout did not occur permits an estimation of

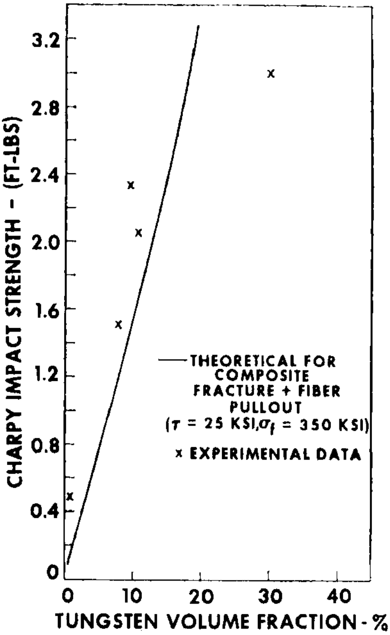


FIGURE 2 Charpy impact strength—unidirectional W–SiO_2 composites

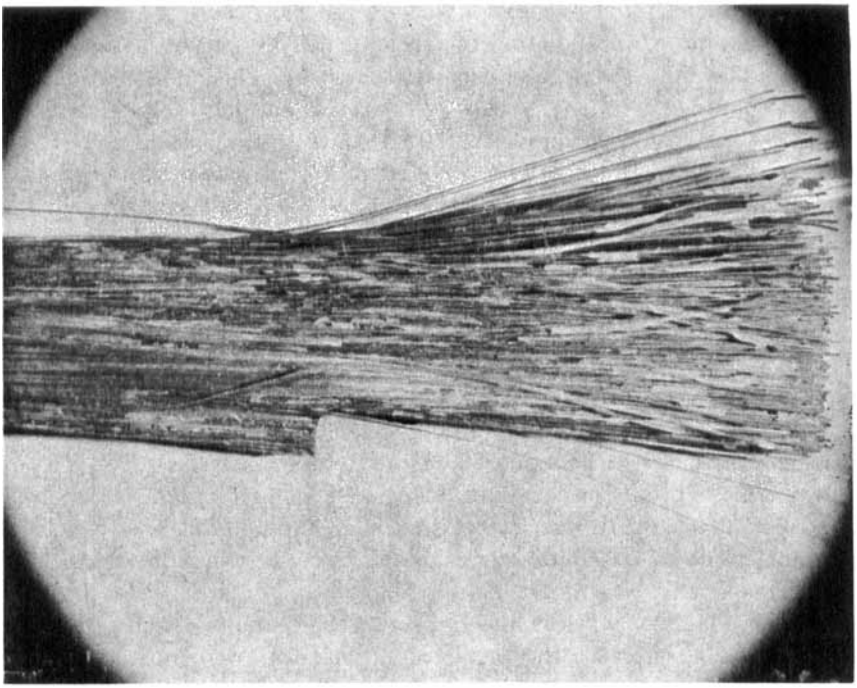


FIGURE 3 SiO_2–30 percent W, impact specimen showing interfacial splitting.

the contribution of interfacial splitting to composite impact energy. The elastic energy prior to failure for the 30 percent composite is 0.7 ft-lbs. If the remaining measured energy is assumed to be a result of interfacial splitting, that mechanism would account for 2.3 ft-lbs.

The tests of the W-SiO_2 system showed that the energy absorbing mechanisms found in resin matrix composites could also be applied to ceramic composites. This provided a good foundation for attacking the problem of impact strength of more useful ceramics such as silicon nitride. The results further indicated that the interfacial bond strength of fiber reinforced ceramics is an important factor in their impact behavior.

III EXPERIMENTAL PROCEDURE

A. Materials

The selection of a ceramic matrix material for use in high temperature fiber-reinforced composites depends upon a number of factors. Among these are: ease of fabrication, strength at both low and elevated temperatures, oxidation and corrosion resistance, thermal shock resistance, creep strength, and impact resistance. Many ceramic materials meet the criteria of strength and oxidation resistance at the temperature of interest, 2400°F. However, very few ceramic materials, capable of use at 2400°F, also meet the requirements of good thermal shock resistance and ease of fabrication. Silicon nitride (Si_3N_4) was chosen as the matrix for this study because it has excellent thermal shock and flexural strength and can be fully densified at the relatively low temperature of 1650°C by hot pressing with a few percent MgO additive.

The types of fibers available for use in a refractory ceramic matrix such as Si_3N_4 are limited. Tungsten fibers were selected as a result of their good high temperature strength, favorable coefficient of thermal expansion which results in compressive prestress on the matrix after fabrication, and their cost and availability. There are two types available which retain their strength after exposure to the temperature required to consolidate Si_3N_4: type 3D (W-3%Re) and type NF (W-1% ThO_2). Both of these fibers (5 mil diameter) were used as reinforcements for Si_3N_4.

Calculations of impact strength have been made for *W*-Si_3N_4 composites as a function of fiber volume fraction. Figure 4 presents a breakdown of the energy absorbing mechanisms for W-Si_3N_4. The lower most curve represents the amount of elastic energy absorbed in the specimen just prior to failure at the tensile surface of the impact specimen and is one-third of Eq. (2). This is obviously quite low for all fiber volume fractions. The middle curve

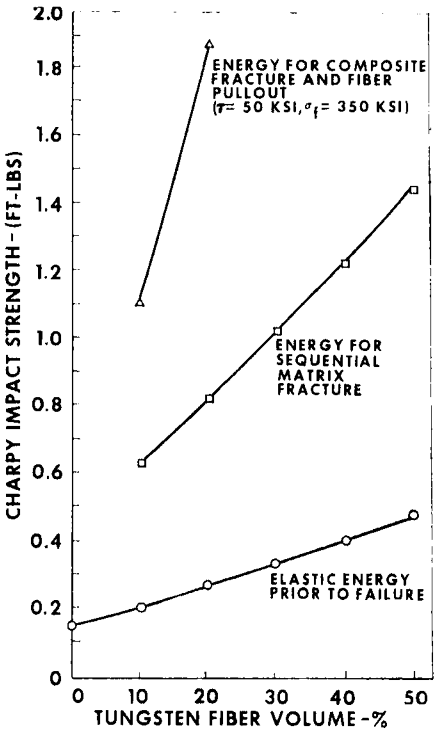


FIGURE 4 Theoretical Charpy impact energy, unidirectional W-Si_3N_4 composites.

shows the composite fracture energy absorbed as all the material in the composite is sequentially taken to its failure stress and includes the elastic energy. Finally, the uppermost curve is the total energy, composite fracture plus pullout, for the composite, assuming an interface shear strength of 50 ksi.

The calculations show that room temperature impact strengths on the order of three to four ft-lbs should be obtainable, provided the interfacial shear strength of 50 ksi can be achieved. These impact values appear to be adequate when compared with vane materials such as Mar M-200 and Mar M-302 which have impact strengths of two to six ft-lbs.

B. Specimen fabrication

The incorporation of tungsten wires in Si_3N_4 has been studied quite extensively at our laboratories and a successful composite layup procedure has been devised. Figure 5 shows a quite even array of 25 volume percent tungsten wires (NF type-1% ThO_2) fabricated by slurry coating prewound wires with

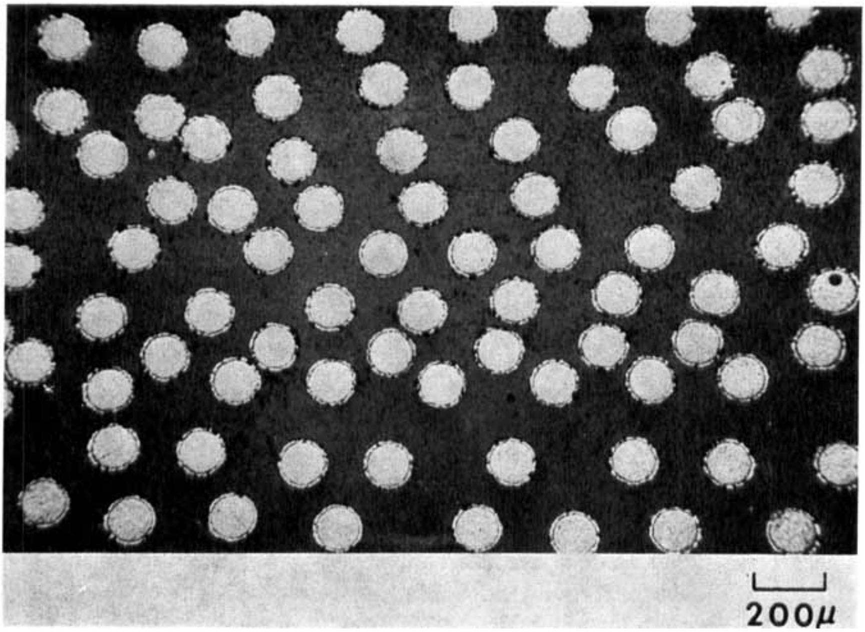


FIGURE 5 Si_3N_4-tungsten composite.

a mixture of Si_3N_4 + 5 percent MgO powder, stacking the coated sheets to a desired thickness and then hot-pressing at 1650°C, 4 ksi, for 1 hr. Sample sizes have been 2¼ in. × ⅝ in. with various thicknesses.

C. Test techniques

Impact tests were conducted using an unnotched Charpy specimen. Elevated temperature tests were carried out by heating the specimen to 100°C higher than the test temperature, then quickly transferring it to the impact machine for testing. Modulus of rupture tests were conducted at a span-to-depth ratio of 10:1. Elevated temperature tests were carried out in a heated chamber under an air environment. Specimens were isothermally maintained for 30 minutes prior to testing.

IV RESULTS AND DISCUSSION

A. Modulus of rupture and impact data

Table I shows some of the results of this study. The average room temperature modulus of rupture has been increased by over 20 ksi due to the compressive prestress put on the matrix by the wires on cooling from fabrication

TABLE I

Si_3N_4 and Si_3N_4–W composite properties

Property	β-Si_3N_4 + 5% MgO	β-Si_3N_4 + 5% MgO + 25 vol %W
Modulus of rupture (MOR)		
RT	72 ksi (3-pt)	95 ksi (3-pt)
1400°C	23 ksi (3-pt)	40 ksi (3-pt)
Charpy impact (unnotched)		
RT	0.49 ft-lbs	0.51 ft-lbs
1300°C	0.25 ft-lbs	2.25 ft-lbs

temperature. The magnitude of the prestress can be approximated from the following equation:

$$\sigma = \frac{(\alpha_m - \alpha_f)\Delta T E_m}{1 + \dfrac{V_m E_m}{V_f E_f}}$$

where α = coefficient of thermal expansion
ΔT = temperature drop from fabrication to test temperature
E = modulus of elasticity
V = volume fraction
f, m = subscripts denoting filament and matrix respectively.

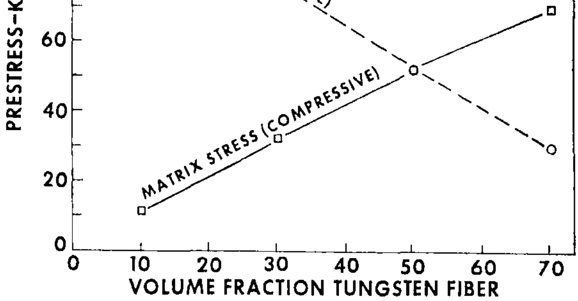


FIGURE 6 Longitudinal thermal prestress—unidirectional W fiber—Si_3N_4 matrix composite.

The calculated prestresses for the W–Si_3N_4 system are shown in Figure 6. The prestress, 25 ksi, calculated for 25 volume percent W composite is very close to the measured value of 23 ksi.

At 1400°C, the modulus of rupture of unreinforced β-Si_3N_4 + 5 percent MgO, hot pressed at 1675°C, decreases to about 20 ksi, as has been noted by previous investigators.[6] However, composites tested at 1400°C in air were found to have a modulus of rupture of 42 ksi. Prestresses at this temperature are negligible so the increase in MOR is due primarily to reinforcement by the tungsten wires. The indicated strength of the tungsten wire in the composite at 1400°C of 125,000 psi implies little tungsten strength degradation by being incorporated into the Si_3N_4.

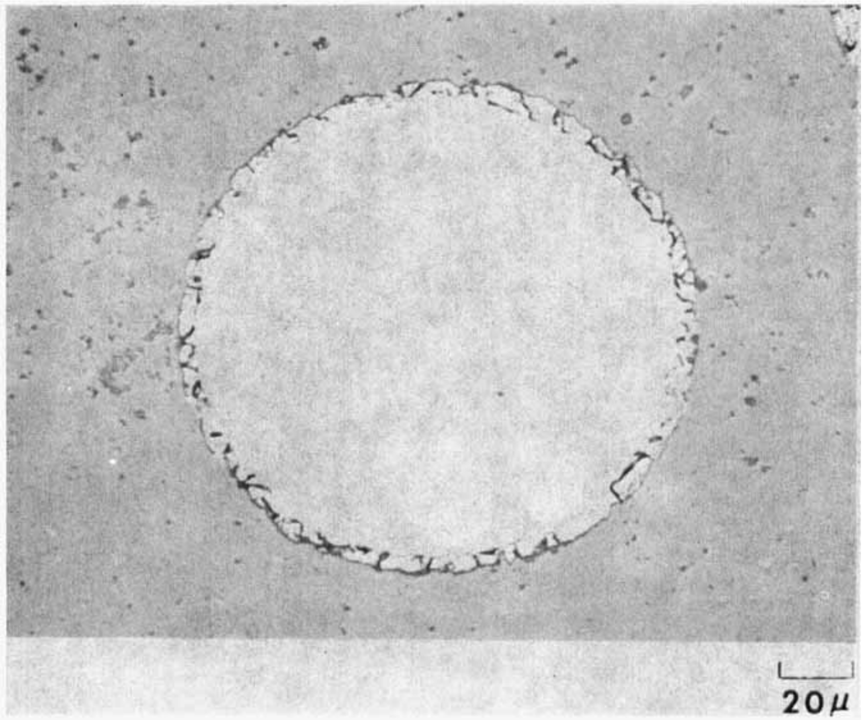


FIGURE 7 NF tungsten hot-pressed in Si_3N_4 at 1650°C.

There is, however, a reaction between the tungsten and silicon nitride during hot pressing at 1675°C. From Figure 7, one can see a reaction ring around the 5 mil tungsten fiber about 8 microns in width. From electron microprobe studies the phase formed has been identified as W_3Si_2. This silicide reaction product has a detrimental effect on composite impact strength at room temperature although not at elevated temperatures, as can be seen from Table I. The measured room temperature impact strength of both monolithic and tungsten reinforced Si_3N_4 is approximately 0.5 ft-lbs. The fact that the composite impact strength is the same as pure Si_3N_4 leads to the conclusion that either the fiber strength has been degraded through reaction or that the interfacial shear strength between the fiber and the matrix is very high, precluding any significant fiber pullout. Examination of fracture surfaces revealed very little pullout (Figure 8). We have noted, however, that at 1300°C the composite impact strength shows a ninefold increase over monolithic Si_3N_4 with fiber pullout being quite extensive (Figure 9). It appears then that the interfacial silicide reaction product has a detrimental effect on the impact strength at room temperature but not at elevated temperatures. This is due to the fact that at elevated temperatures, W_3Si_2 is relatively weak with a low modulus, allowing crack blunting to take

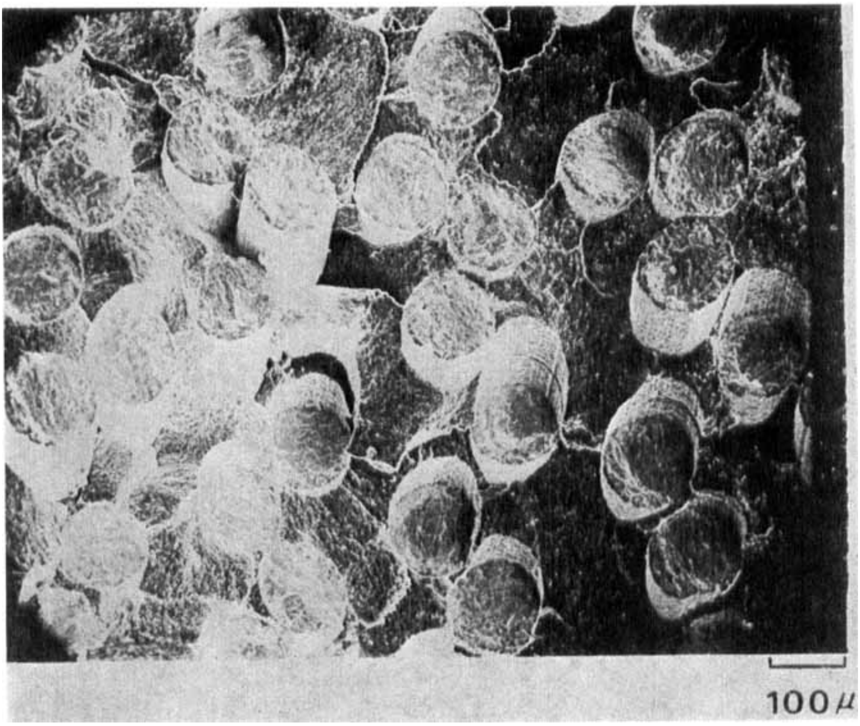


FIGURE 8 Si_3N_4-W RT impact fracture surface.

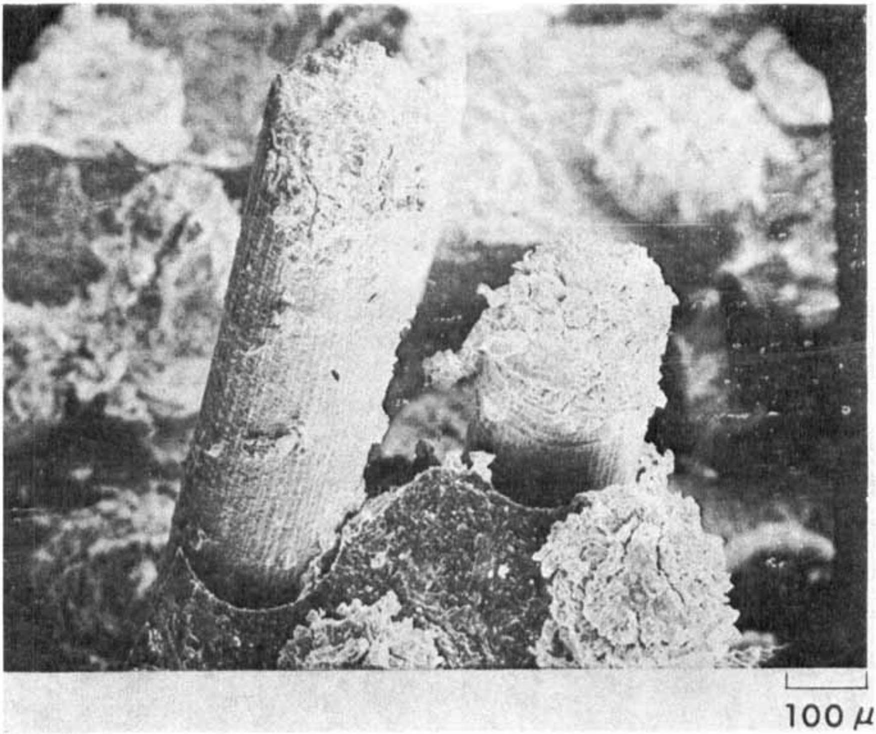


FIGURE 9 Si_3N_4–W 1300°C impact fracture surface.

place at the low shear strength interface with resultant fiber pullout, while at room temperature the W_3Si_2 is extremely brittle with a high modulus. It is also bonded very strongly to the tungsten so that when a crack moves through the Si_3N_4 matrix and encounters a fiber, it continues to propagate through the silicide and, due to the extreme notch sensitivity of tungsten, right through the wire without allowing any energy absorbing mechanisms to take place.

B. Techniques of Impact Strength Improvement

Since a reaction between the Si_3N_4 matrix and the tungsten wire reinforcement takes place during hot pressing, any possible gain in impact resistance at room temperature is negated. This is due to the extremely brittle nature and high modulus of the silicide-coated reaction product. To make use of the energy absorbing mechanism of fiber pullout and/or interfacial splitting one needs to retain the filament strength and have a fairly weak or low modulus interface or interfacial region between the tungsten and the silicon nitride. In order to do this it appears that one must stop the formation of W_3Si_2 by either lowering the fabrication temperature or by applying a reaction barrier coating on the tungsten wires.

1) *Lower temperature fabrication development* From available thermodynamic data it appears that the free energy of reaction between tungsten and Si_3N_4 is negative above about 1430°C and positive below that temperature. A tungsten silicide layer is therefore expected to form when hot pressing is

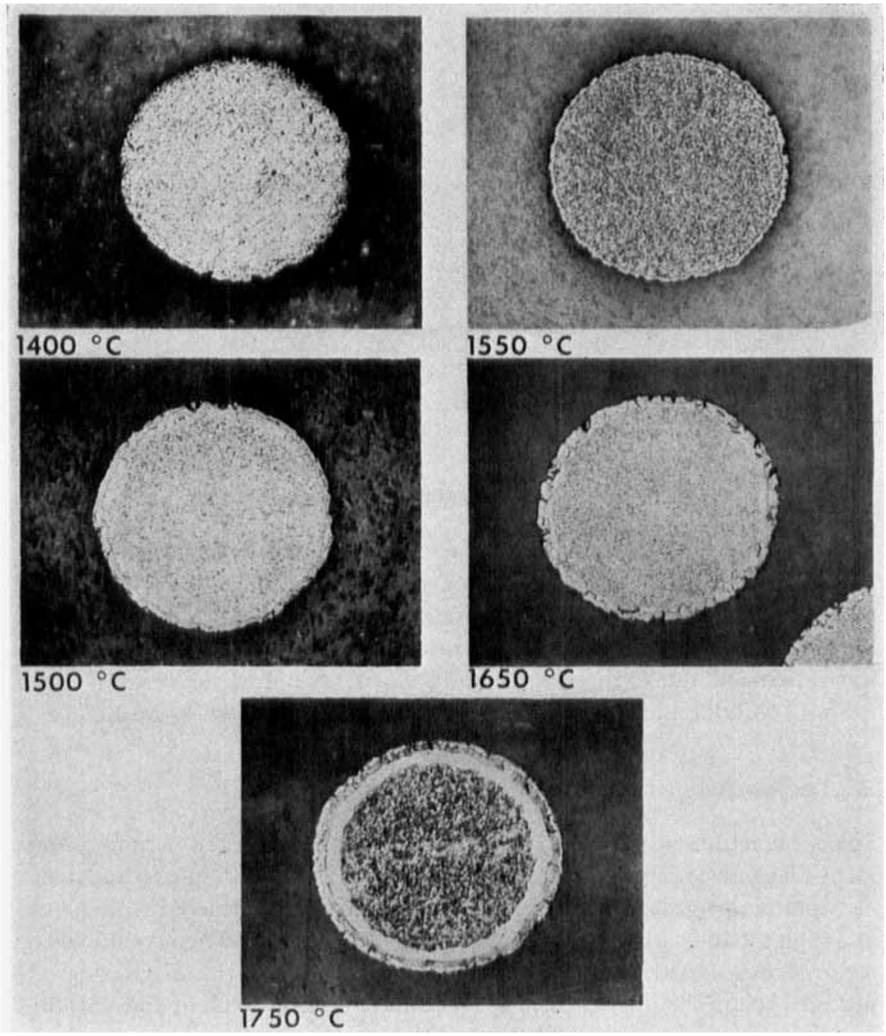


FIGURE 10 Si_3N_4-W compatibility studies.

carried out above 1430°C. The thermodynamic analysis is verified in Figure 10 which shows tungsten fibers hot pressed in β-Si_3N_4 + 5 percent MgO at temperatures ranging from 1400°C to 1750°C. It can be seen that as the temperature is increased the reaction zone becomes increasingly larger accompanied by a recrystallized tungsten zone above 1650°C. At 1400°C, no reaction zone is evident. However, with MgO as the densification aid, full densification does not occur below about 1600°C. Therefore, in order to prevent silicide formation, a densification aid is required which is effective at 1400°C or less. Some experiments were performed to explore the likelihood of accomplishing this objective.

It has been observed[7] that molten copper is one of the few metals that react with Si_3N_4. However, this reaction did not proceed in the absence of oxygen so it evidently is a combination of copper, oxygen, and Si_3N_4 that react. Accordingly, it was reasoned that CuO in small amounts might aid in the densification of Si_3N_4. However, 9 percent CuO + β-Si_3N_4 hot pressed at 1500°C did not lead to densification greater than 90 percent. The CuO appeared to remain intact and a larger reaction zone around tungsten was noted than when β-Si_3N_4 was pressed with 5 percent MgO at this temperature. Therefore, CuO was dropped as a candidate densification aid.

Work on alternate densification aids is in progress with the discovery of two proprietary aids other than MgO that allow Si_3N_4 to be hot pressed to full density. Unfortunately, neither aid allowed full densification below about 1550°C. Also, one of them proved to exhibit very poor oxidation characteristics. Work on lower temperature densification aids is continuing.

2) *Reaction barriers* In order to prevent the formation of tungsten silicide a coating is needed which will remain stable in contact with Si_3N_4 and tungsten during hot pressing at 1650°C and which will not allow appreciable diffusion of silicon. From published data on the free energy of formation of various silicides, nitrides, carbides, borides, and oxides[8], certain barrier candidates are suggested. Among these are: SiC, $TiSi_2$, TaC, TiC, MoC, TiN, TaN, BN, TiB_2, TiO_2, ZrO_2, and SiO_2. Other factors to be considered are strength at 1650°C, ease of coating a stoichiometric product, mismatch in thermal expansion, and silicon and cation diffusivities in general.

Among the barrier candidates listed above, work has been done on SiC, TiB_2, BN, and SiO_2. The SiC was deposited by the H_2 reduction of dichloromethylsilane, the process used to make BORSIC from boron fibers. It was found that on deposition of a 0.2 mil thick SiC coating on tungsten, the tungsten behaved in a brittle manner with a 60 percent loss in room temperature strength. When the SiC coating was removed from the tungsten wire in an acid bath the wire regained ductility. This same phenomenon was observed in TiB_2 and WSi_2 coated tungsten wires, as well as a wire coated with

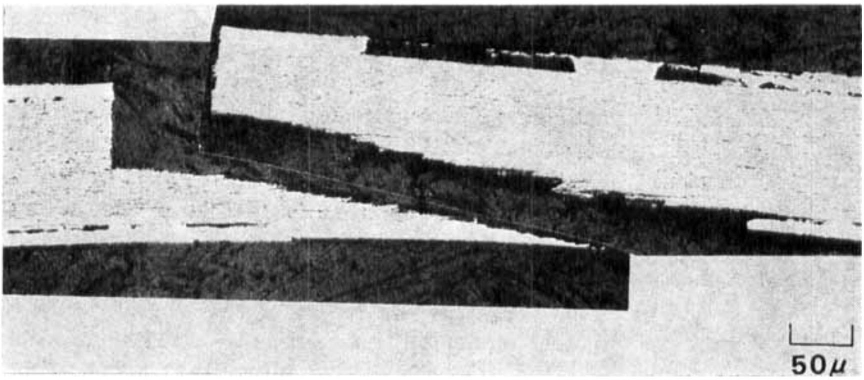


FIGURE 11 Brittle failure in Cr coated tungsten.

chromium as shown in Figure 11. However, when a ductile coating such as nickel is deposited first, followed by a brittle chromium layer (Figure 12) the tungsten behaved in a ductile manner. These observations suggest that the nature of the barrier coating is an important factor in determining its usefulness. An example of this is a TiB_2 coating on tungsten that, when hot pressed in Si_3N_4, successfully prevented the formation of silicide but fractured in a brittle manner. Tests will be conducted on other brittle coatings in order to determine if this phenomenon is consistent.

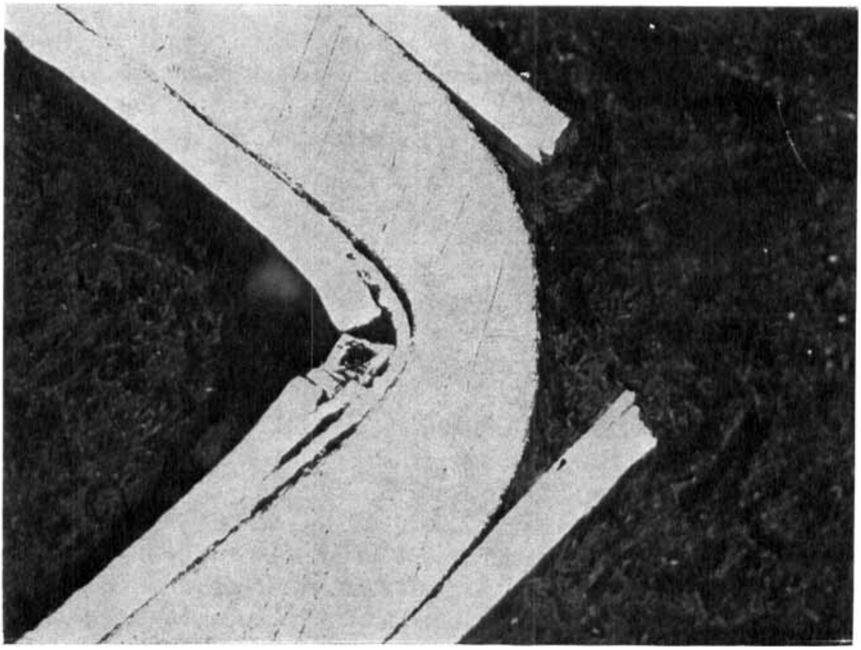


FIGURE 12 Ductile behavior in duplex Cr-Ni coated tungsten.

One coating, BN, would seem to be ideal since, along with being nonreactive with tungsten and Si_3N_4, it is quite weak and would not be expected to fracture in a manner similar to strong, brittle coatings. However, it was found that tungsten wires, when hot pressed in contact with BN powder at 1650°C, completely recrystallized with almost a total loss in strength. Evidently boron is diffusing into the tungsten causing the recrystallization to occur.

Of the barrier candidates shown to be thermodynamically stable in contact with W and Si_3N_4, only SiO_2 has proved to be successful. From Figure 13, which shows a silica-coated tungsten wire hot pressed in an α-Si_3N_4 powder + 5 percent MgO matrix at 1400°C, it can be seen that the tungsten wire is successfully protected from the Si_3N_4 by the SiO_2 and that no silicide reaction product has formed. The SiO_2 coating is applied by simply threading the 5 mil wire into a 10 mil O.D. by 6 mil I.D. silica tube. On hot pressing at 1400°C, the silica flows slightly, however, not enough to expose the tungsten to the Si_3N_4; thus, the tungsten remains strong and ductile. In fact, the room

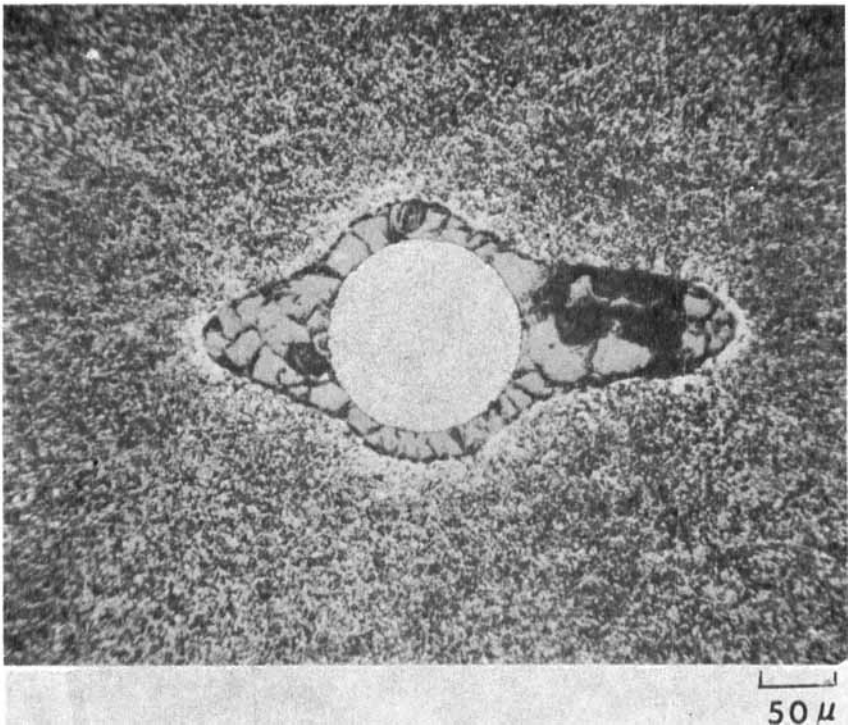


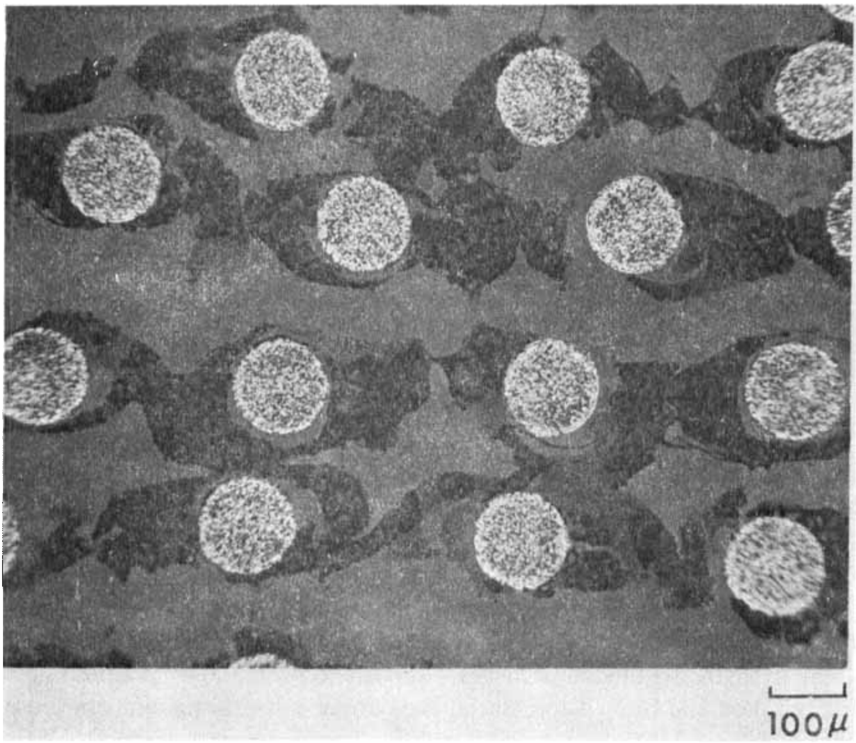
FIGURE 13 Silica coated tungsten wire hot-pressed in Si_3N_4 + 5 percent MgO at 1400°C.

temperature impact strength of a Si_3N_4–15 volume percent W composite with SiO_2 barriers hot pressed at 1375°C was 1.6 ft-lbs. From Figure 5 this can be seen to be the value predicted from theoretical calculations, which consider only elastic fracture of matrix plus reinforcement and fiber pullout contributions. Figure 14 shows this sample after impact with extensive fiber pullout and some unbroken wires visible. It is interesting to note that the sample did not break into two pieces on impact but continued to hold together.

At temperatures of 1400°C or so the Si_3N_4 + 5 percent MgO does not densify to any great extent (~70 percent) as can be seen from Figure 13.

FIGURE 14 Silicon nitride-silica coated tungsten composite RT impact specimen.

Thus, modulus of rupture values and related properties are poor. A hot press at 1500°C was done to see if greater densification of the Si_3N_4 could be obtained without disrupting the SiO_2 reaction barrier. From Figure 15, which shows a cross section of this sample, it is apparent that good densification took place but that the silica at this temperature was sufficiently fluid to allow the Si_3N_4 to contact the tungsten wires. Consequently, a silicide layer formed around many of the wires thus leading to brittle failure of the tungsten. The room temperature impact strength of this specimen was 0.4 ft-lbs which is similar to that obtained without using a silica reaction barrier. It appears then that a densification aid other than MgO to allow hot pressing at temperatures of 1400°C must be used if silica is to be used as the reaction barrier coating. It is possible that no coating will be necessary if hot pressing can be accomplished below 1400°C, since the silicide reaction is not thermodynamically favorable below this temperature, as discussed previously.



CONCLUSIONS

The main conclusions that can be reached from this study are:

1) There are certain energy absorbing mechanisms that can be active in the impact characteristics of fiber reinforced composites. These mechanisms are: fiber fracture, fiber pullout, delamination, and interfacial splitting. The contribution of each mode of energy absorption is governed in a large part by the interfacial characteristics and bond strength of the composite system.

2) The 1300°C Charpy impact strength of hot pressed Si_3N_4 has been increased from 0.25 ft-lbs to 2.25 ft-lbs by the incorporation of 30 volume percent tungsten wires. At this temperature the interfacial shear strength is sufficiently low to allow the energy absorbing mechanism of fiber pullout to be operative.

3) The RT unnotched Charpy impact strength of hot-pressed Si_3N_4 is 0.5 ft-lbs in both monolithic form and when reinforced with 30 volume percent tungsten wires. This is due to the formation during hot pressing of a very brittle, strongly bonded reaction layer of tungsten silicide which precludes fiber pullout from occurring as a result of the very high interfacial shear strength.

4) In order to obtain an increase in room temperature impact strength, one must prevent the formation of W_3Si_2 by either lowering the fabrication temperature or by applying a reaction barrier coating on the tungsten wires.

5) Various carbides, borides, nitrides, silicides, and oxides have been found to be possible reaction barrier candidates. However, it appears that some of them adversely affect the tungsten fiber strength due to their brittle nature. Notable exceptions such as BN and SiO_2, which would give a low interfacial shear strength, are limited in that BN appears to promote recrystallization in the tungsten and SiO_2 is too soft at the current hot-pressng temperature of 1650°C.

6) Through the use of a silica reaction barrier around the tungsten wires, the RT impact strength of a Si_3N_4-15 percent W composite can be increased from 0.5 ft-lbs to 1.6 ft-lbs, even though the Si_3N_4 matrix is not fully dense due to the fact that hot pressing must be done below 1400°C.

7) In order to maintain the integrity of the SiO_2 barrier and also fully densify the Si_3N_4 matrix, a densification aid must be used that will be effective at temperatures around 1400°C. Densification aids other than MgO have been found but are not effective below 1550°C.

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