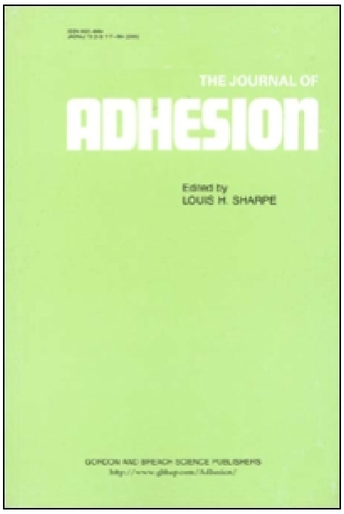

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Interfaces in Fabricated Metal Matrix Composites†

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The structure of the interfaces in metal matrix composites are reviewed and a classification of composite systems according to interface type is proposed. Changes in the interface resulting from processing or high-temperature exposure are discussed and related to corresponding changes in tensile properties. Particular interest is attached to two types of interface which are found in titanium matrix composites (reactive, Class III systems) and aluminum matrix composites (pseudo Class I system). Growth of the interface compound with titanium matrices results in three modes of control of the fracture process. Aluminum matrix composites form the initial bond through oxide films but this breaks down and results in changes in the fracture process. Models are advanced to explain these results.

1 INTRODUCTION

Interfaces in metal matrix composites are more complex than those found in plastic matrix composites and several reasons can be identified to account for this difference. Plastic matrix composites are combinations of two essentially inert constituents so that achievement of adequate bonding often requires "finishes" (exhibiting bidentate features; that is, specific portions are active with respect to matrix and filament). Metal matrix composites include examples of such inert combinations, e.g., aluminum and alumina whiskers, but the majority of the useful combinations are mutually reactive. Indeed, reaction to give strong bonds may be essential to use a metal matrix effectively. Metals are one or two orders of magnitude stronger than plastics and

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a bond is necessary to develop the cooperative properties of the composite. Therefore, interfaces in metal matrix composites are required to be much stronger than those in plastic matrix composites, and this high strength must be achieved by control of the mutual reactivity of the matrix and reinforcement.

Enough information has been gathered on metal matrix composites to propose three general classifications:

Class I. Matrix and reinforcement are not reactive and are insoluble.
Class II. Matrix and reinforcement are not reactive but are soluble.
Class III. Matrix and reinforcement are reactive.

By reactive is meant the formation of a new chemical compound (or compounds) at the interface between matrix and reinforcement. Table I gives examples of each class. Directionally solidified eutectics closely approach termal equilibrium between the phases but are included in Class II because some change of terminal solubility with temperature will occur. However, attention will be directed in this paper to the interfaces in "fabricated" metal matrix composites, that are prepared from the separate constituents by solid state diffusion, molten metal infiltration or other technique.

TABLE I
Classification of Composite Systems

Class I	Class II	Class III
Copper-tungsten	Copper (chromium)-tungsten	Copper (titanium)-tungsten
Copper-alumina	Eutectics	Aluminum-carbon (> 700°C)
Silver-alumina	Columbium-tungsten	Titanium-alumina
Aluminum-BN coated B	Nickel-carbon	Titanium-boron
Magnesium-boron	Nickel-tungsten[b]	Titanium-silicon carbide
Aluminum-boron[a]		Aluminum-silica
Aluminum-stainless steel[a]		
Aluminum-SiC[a]		

[a] Pseudo Class I system.
[b] Becomes reactive at lower temperatures with formation of Ni_4W.

There are, of course, exceptions to these classifications and one of the most important is the pseudo-class I type of composite. These are composites that appear to be non-reactive class I type when fabricated by the optimum process, usually by the solid state diffusion bonding, but the constituents are known to form stable compounds or solid solutions. The principal examples of pseudo-class I composites are: aluminum-boron; aluminum-stainless steel; and aluminum-graphite. Although filaments and matrix are initially non-reactive, the compounds AlB_2, Fe_2Al and Al_4C_3 have been identified at the interface of these composites under conditions where the equilibrium product could be formed. Examples of conditions leading to the formation of the equilibrium compound are: excess temperature, time or pressure in hot pressing (Al-B and Al-stainless steel); cold rolling and annealing (Al-stainless steel); and welding (Al-graphite). The explanation for this behavior is that bonding is initially between the omnipresent oxide film on the aluminum (Al_2O_3) and oxide or adsorbed films on the boron, steel or graphite filament. If the latter is represented by the formula, FO (e.g., B_2O_3 on boron), the initial oxide bond formed between these two oxides or films may be represented by $FO.Al_2O_3$. Subsequent reaction will occur between the oxide bond and the matrix,

$$FO.Al_2O_3 + Al = Al_2O_3 + F$$

to generate almost pure alumina at the interface. This alumina film is in pseudo-equilibrium with both matrix and reinforcement, but becomes unstable under the conditions cited above. Breakdown of the film results in local contact and local reaction to form the equilibrium compounds at the interface.

Rule-of-mixtures (ROM) strengths are found in composites from each of the three main classifications. Class I composites are inherently stable and give ROM strengths in the longitudinal direction under all conditions, although the lack of strong bonding may adversely affect the transverse properties. The pseudo-class I composites show ROM properties before breakdown of the interface film takes place, and begin to lose strength only when a minimum amount of reaction has occurred. Class II composites are least affected by reaction and may increase in off axis strength as a result of interdiffusion of the two mutually soluble constituents (e.g., Cb-W composites). Class III composites are the most difficult to control to produce ROM properties, because there is no period of pseudo stability such as occurs in pseudo-class I composites, but it has been shown that the predicted properties can be attained up to a well defined thickness of the reaction product.

Optimization of properties other than strength may require other conditions at the interface. Hence, a detailed knowledge of the structure of the interface, the relation of the interface to mechanical properties, and factors such as reaction kinetics that control changes at the interface must be known if metal-matrix composites are to be produced with desirable properties. This type of information is being collected for the important composite systems and will be reviewed briefly in this paper beginning with pseudo-class I composites.

II PSEUDO-CLASS I SYSTEMS

The principal systems of this class are: aluminum-boron; aluminum-stainless steel; and aluminum-graphite. Other systems may belong to this category, but insufficient work has been done to define the interface for other cases.

Bonding in these systems is initially between the omnipresent aluminum oxide films and the surface films on the reinforcement. The latter may be an oxide such as a boron oxide on boron, or a spinel on stainless steel. The less stable oxides from the reinforcement will be reduced by reaction with aluminum to give almost pure alumina and deposition of the reduced elements on to the filament. Figure 1 shows a thin section from an aluminum-boron composite where the alumina film has been identified by electron diffraction[1]. These composites are fabricated by solid state diffusion under carefully controlled time, temperature and pressure so that the oxide films are essentially preserved. Parameters above the optimum values result in disruption of the film and the degradation of properties as a consequence of interaction. Incomplete densification and bonding result in lower strengths associated with the porosity and inconsistent bonding. Accordingly there is a processing "window" for these types of composites.[1] This behavior is illustrated for one series of Al(6061)/48B panels bonded at different temperatures. As shown in Figure 2, for constant time and pressure, the window is approximately 50°F in width for optimum tensile strength of this material.

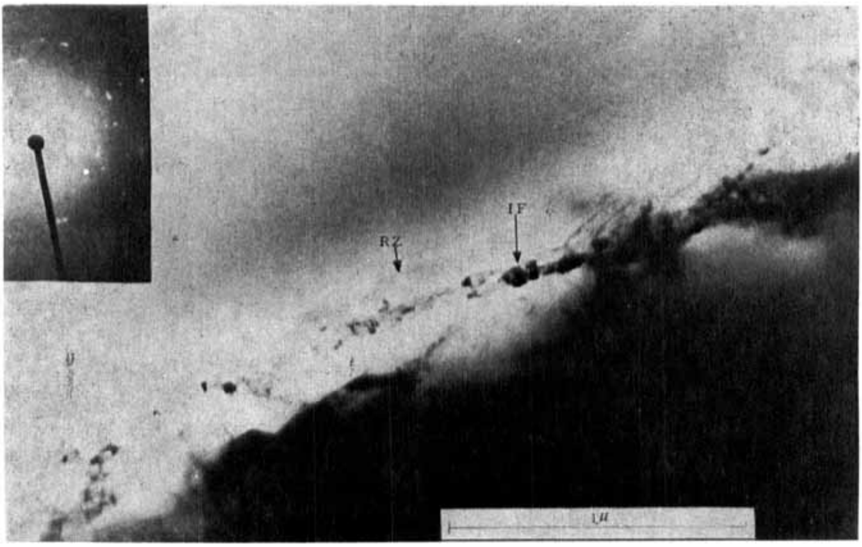


FIGURE 1 Thin Section of Aluminum-Boron Composite Showing the AlB_2 Reaction Zone (RZ) and the Original Al_2O_3 Interface (IF).

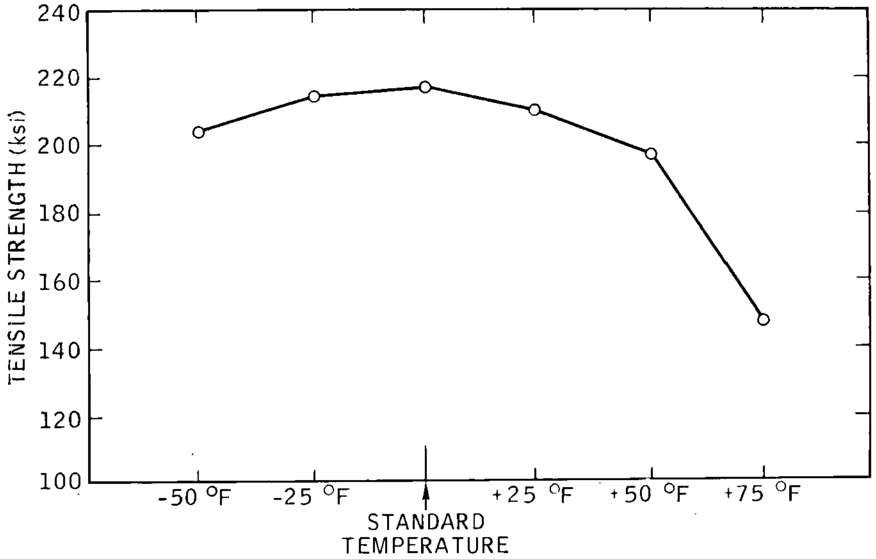
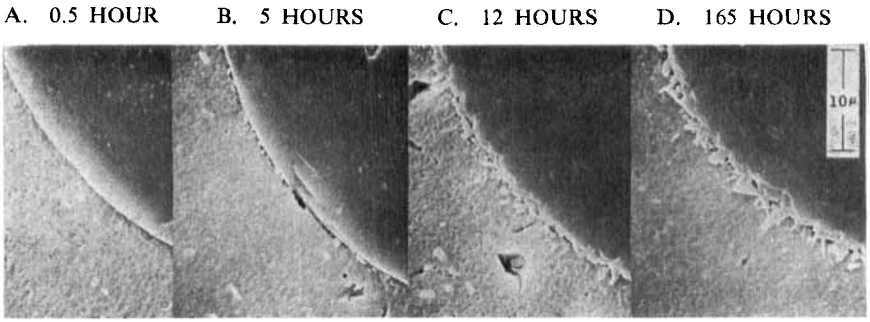


FIGURE 2 Effect of Press Bonding Temperature on the Tensile Strength of Al(6061)/48 per cent B Panels. The temperature is shown in increments of 25°F variations from the standard bonding temperature used.

However, this window is not necessarily at the same temperature for other mechanical properties. For example, recent work suggests that the optimum fracture toughness may be found where the bonding is less strong, i.e., on the low temperature side of the bonding window. Also, work in England on aluminum-stainless steel and on aluminum-silica,[2,3] suggests that the optimum processing for longitudinal fatigue strength may not be the same as that for optimum tensile strength.

The effect of processing on the structure of the bond in aluminum (6061)-boron can be followed readily by subsequent heat treatment of specimens cut from a composite panel bonded under uniform standard conditions. Figure 3 shows the effect of heat treatments at 940°F for times up to 165 hours on the condition of the interface. Isolated needles of aluminum-boride grow laterally as well as into the matrix. These needles, which have been identified as AlB_2 by electron diffraction,[1] grow through the oxide film shown in Figure 1. Eventually the reaction product appears to grow together to form an irregular reaction zone that completely surrounds the filament. The interface reaction product has been extracted from composites by etching away the aluminum matrix with alkali and preferentially etching the boron with Murikami's reagent. In this way, the interfacial film has been extracted and examined in the electron microscope to confirm the general interpretation



A. 0.5 HOUR B. 5 HOURS C. 12 HOURS D. 165 HOURS

FIGURE 3. Interface in Al(6061)/B Composite Heat Treated at 940°F for the Indicated Times.

given above. Similar growth products have been observed in the case of aluminum-stainless steel and described as "corn-cob" in appearance.

The effect of reaction at the interface on the longitudinal and transverse strengths of aluminum (6061)-boron has been investigated in some detail, but observations on the effect of such reaction on other properties has not been studied. Figure 4 compares the effect of annealing time on the longitudinal strength of one of these composites.[1] Three regions of behavior can be identified. The first shows no change in strength, or may show a small increase. The structure of the interface at the end of this first region corresponds to that shown in Figure 3 after 0.5 hours at 940°F. There are two

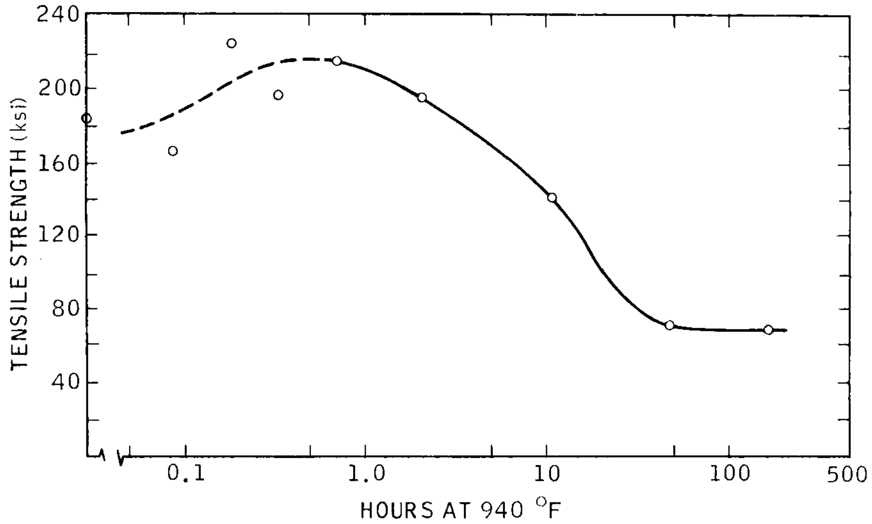


FIGURE 4 Variations in Tensile Strength of Al(6061)/45B with Pre-Test Heat Treating Time at 940°F.

reasonable explanations for the apparent peaks in strength. The first is that the material was processed to less than the optimum condition shown in Figure 2, and this was attained in the subsequent annealing. The second explanation is that the growth of aluminum diboride across the interface increases the efficiency of load transfer by interfacial shear. Both of these explanations may be equivalent because there is evidence that optimally processed aluminum-boron shows signs of breakdown of the oxide film with a few aluminum diboride crystals penetrating the film to provide a keying action between matrix and reinforcement. Loss of tensile strength begins when progressive interface reaction extends over most of the surface of the filaments. This degradation reaches a limit where the strength reaches a constant minimum value at the beginning of the third region in Figure 4.

The elastic modulus does not vary as a result of these amounts of interface reaction. This is to be expected because the modulus is an additive property, and the amounts of matrix and filament are almost unchanged by the reaction at the interface. On the other hand, the strain-to-fracture shows a marked effect of the reaction. Figure 5 shows that although it parallels the strength curves, the strain-to-fracture is not affected by the volume percentage of boron, by the heat treatment given the matrix, or by the size of the boron. Although the third region in Figure 4 corresponded to different strengths depending on the composite studied, the strain-to-fracture has a constant value of 3200 ± 200 microinches per inch. By analogy with reaction theory

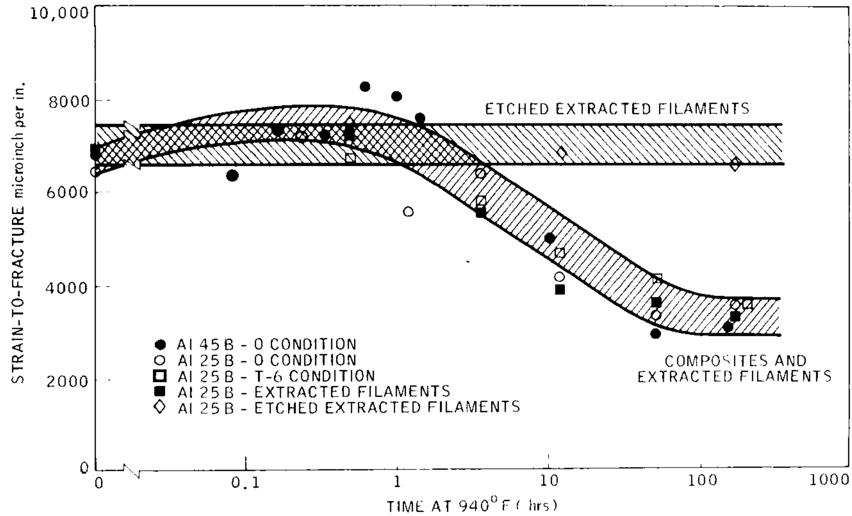


FIGURE 5 Variation in Strain-to-Fracture of Al(6061)/B Composites and Extracted Filaments with Pre-Test Heat Treating Time at 940°F.

first worked out for systems such as titanium-boron and titanium-silicon carbide,[4] it was tentatively concluded that this constant value could be attributed to the presence of aluminum diboride. This viewpoint was substantiated by extracting filaments from composites. The strength of these filaments was measured and converted into fracture strain by means of the known elastic modulus.[5] It can be seen in Figure 5 that the fracture strain of the extracted filaments matches the fracture strain of the composites. Removal of the attached aluminum diboride by means of fuming nitric acid restored the strength of the degraded filaments, demonstrating that the loss of strength was caused by the attached compound and did not result from intrinsic degradation of the boron.

The transverse strength of these composites follows a similar relationship to that found for the longitudinal strength. However, the influence of the interface on the strength is less clear because the modes of failure include transverse filament rupture, interface failure and matrix failure. Similarly, notch tensile strength and fracture toughness are affected by the weakening of the filaments as well as by the narrowing of the distribution of strengths.[1] Further discussion of the relation of interface to mechanical properties is not warranted for these cases.

III CLASS II COMPOSITES

Class II composites are formed from two constituents that are soluble in each other to some degree but do not form a reaction product at the interface. An interesting example of this type of system is a columbium alloy matrix strengthened by tungsten wire reinforcement.[6] Complete miscibility of columbium and tungsten allows extensive solid solutions to form.

The as-bonded interface in these composites consists of trapped contaminants from the original constituents as well as trapped gases. Some upgrading of the original interface condition is expected because columbium is able to dissolve large quantities of the typical elements present in such contaminants. Interdiffusion of the tungsten and columbium will occur at the same time as the contaminants are dissolved, and appreciable quantities of tungsten are found throughout the matrix after exposures of many hours at temperatures above 2000°F.

Figure 6 shows the effect of annealing at 2200°F on the strength of a composite of Cb-42Ti-9Cr-4Al with 24 percent of 0.010 inch diameter W-3Re reinforcement.[7] The decrease in longitudinal strength is less than predicted from the effect of annealing on the strength of the W-3Re wire alone. The wide diffusion zone surrounding the tungsten wire consists of highly alloyed

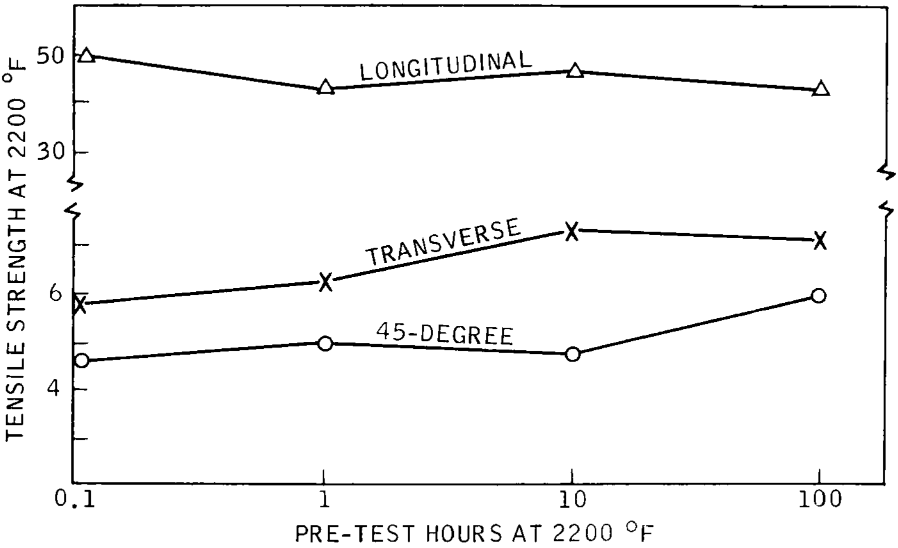


FIGURE 6 Effect of Pre-Test Heat Treatments at 2200°F on the Strength of Cb (Alloy)/ 24W at 2200°F for Different Filament Orientations.

columbium with compositions rich in tungsten. Such compositions are of high strength and are desired in conventional columbium alloy development, but cannot be made because these high tungsten alloys are not amenable to conventional processing. The increase in strength derived from the wide diffusion zone tends to compensate for the strength lost through annealing of the tungsten alloy wire and loss of wire section due to dissolution. The effect of alloying the matrix is particularly beneficial to the strength of the matrix, and this is shown by the marked increase of strength in the off-axis directions. Further indication of the value of this wide inter-diffusion zone is provided by a stress rupture test of a composite that lasted for 1140 hours at 2000°F with an equivalent stress of 115,000 psi in the tungsten wires.[8] The rupture strength of the wires before incorporation in the composite was only 60,000 psi for this duration.

Class II interfaces (and, to a lesser extent, Class III interfaces) continue to extend in width as a result of interdiffusion. In most cases, the difference in intrinsic diffusion coefficients will cause Kirkendall voids to nucleate at some position in the concentration gradient. These rings of voids around the filaments result in uncoupling and may be particularly deleterious to the off-axis strength. Although Figure 6 did not show any decrease in off-axis strengths for up to 100 hours at 2200°F, Figure 7 shows the marked Kirkendall porosity found in a longitudinal tensile specimen after this treatment.

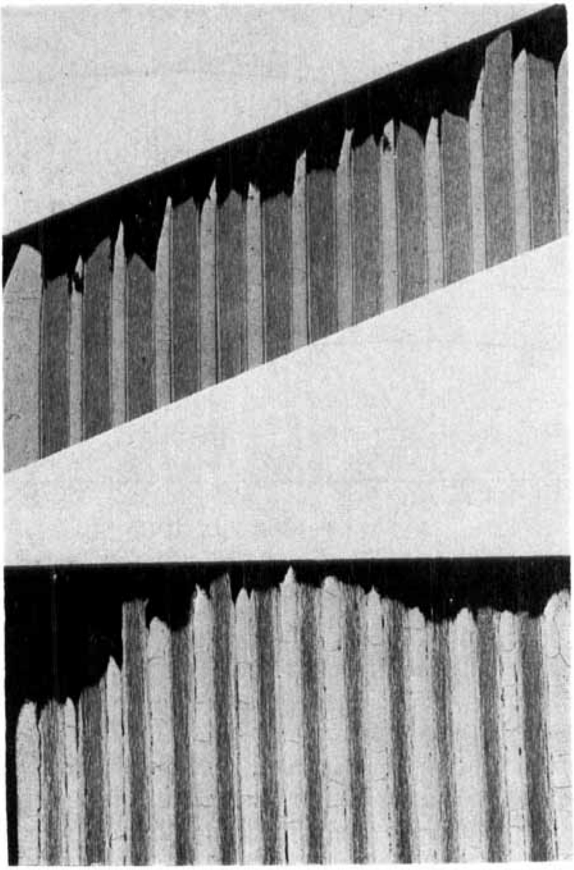


FIGURE 7 Structure of Cb(Alloy)/24W Composite after 100 Hours at 2200°F.

IV CLASS III COMPOSITES

Class III composites are those where a reaction compound forms in a uniform band around the filaments by reaction with the matrix. Examples of systems in Class III are titanium-boron and titanium-silicon carbide (including silicon carbide coated boron).

It has been shown that solid state bonding in these systems occurs without an incubation period. The reaction appears to start instantaneously because titanium and its alloys dissolve surface films such as oxides at the temperature used in bonding to present film-free metal that can react without hindrance. The reactions have been studied for a number of Class III systems and appear to be diffusion controlled with the increase in reaction zone thickness following a parabolic law. In the case of titanium-boron, the principal reaction product is the diboride, TiB_2, with a small amount of monoboride formed between the diboride and the matrix.[9] Growth occurs by transport of boron from the filament across the original interface to cause outward growth of the diboride. Such inbalance of atom transport leads to condensation of vacancies within the boron. However, these tend to condense at the core in the case of the smaller, 4 mil filaments and within the filament in the case of the larger 5.7 mil filament, and do not directly affect the interface. The volume of the principal product, TiB_2, is 20 percent less than that of the elements from which it forms and voids form at the interface principally from this effect.

The titanium-silicon carbide interface has several reaction products and is considerably more complex. Again, overall growth follows the parabolic relationship. However, the differences in intrinsic diffusion coefficients are not as great as for the titanium-boron system, and the volume difference between reactants and product is small. Therefore, detectable voids do not form in this system.

The reaction kinetics have been studied by several workers, and a good summary will be presented in a forthcoming book.[10] In general, reaction rates between boron, silicon carbide or alumina filaments and titanium do not differ greatly. However, significant changes in the reaction rate can be derived by alloying the titanium matrix.

The effect of interaction on the tensile properties of unalloyed titanium matrix composites has been studied by testing specimens heat treated for various times at selected temperatures. The composites were made by a high speed continuous method in the form of tape.[11] The thickness of TiB_2 reaction in the tape before heat treatment was between 500 and 1000 Angstroms. This is below the limit predicted for the onset of loss of tensile strength.[4] The results follow the general trends shown in Figure 8 where the normalized failure stress is plotted against the thickness of interaction zone. Similar profiles are obtained for plots of strain-to-failure versus thickness, but the strain profile is independent of volume percentage of filaments. The curve falls into three regions. The first plateau in strength and strain-to-fracture extends for several thousand Angstroms of interaction; the second region covers the rapid degradation; and the third is a lower plateau at a strain-to-fracture characteristic of the interface product.

The first plateau was predicted to extend up to 1000 Angstrom of TiB_2 by a fracture mechanics type of approach[4] but this ignored the effect of the matrix. Experimental results[12] showed that the extent of this plateau varied with the strength of the matrix. This plateau extended to 4000 Angstroms

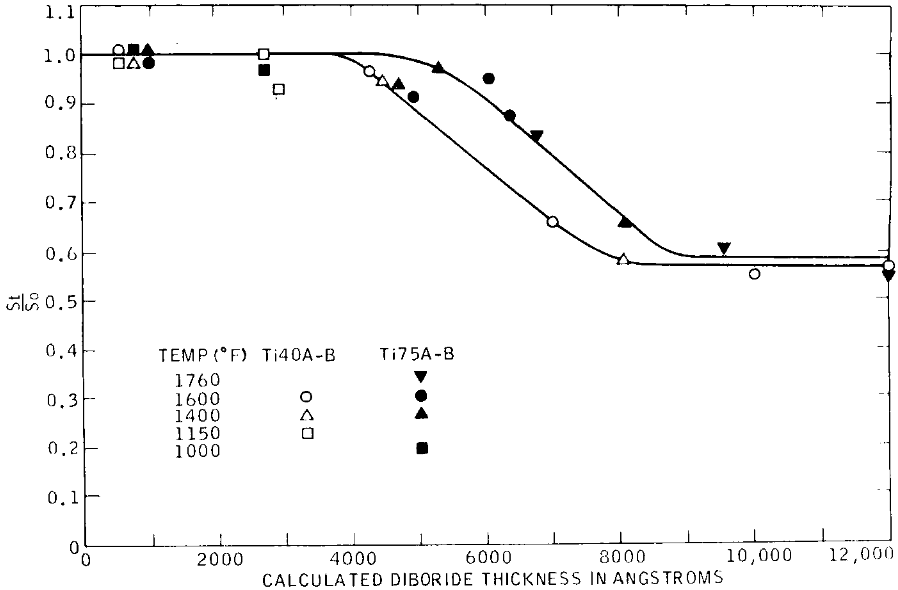


FIGURE 8 Variations in Normalized Tensile Strength, St/So, with Diboride Thickness at the Interface in Ti/25 per cent B Composites.

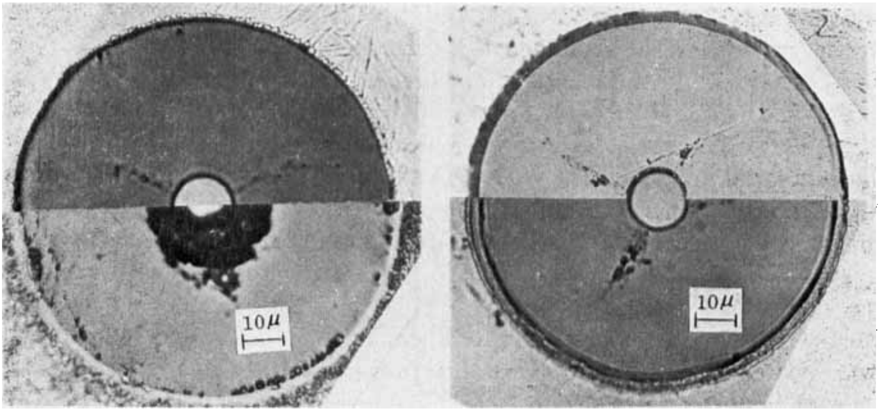
with a weak, unalloyed Ti40A matrix and 5500 Angstroms with the stronger unalloyed Ti75A matrix. These results indicate that the limit may reach 8000 Angstroms before degradation of composite strength begins with higher strength alloy matrices.

The decrease of strength in the second region occurred more rapidly than predicted in the original model so that full degradation in the third region was reached after 8000–10,000 Angstroms of interaction with the unalloyed titanium matrices. The strain-to-fracture in the third region after full degradation was equal to the predicted values of 2500 microinch per inch for the titanium-boron interface and 4500 microinch per inch for the titanium-silicon carbide interface. The latter value has been found also in the systems Ti-6Al-4V/silicon carbide and Ti-6Al-4V/silicon carbide coated boron with agreement within a few hundred microinches per inch. Figure 9 is a comparison of the typical interfaces in both systems before and after a 90-minute heat treatment at 1600°F.

The effect of interface on other mechanical properties has not been studied to the same extent as longitudinal strength. However, some work on transverse and off-axis strength shows that the effect is small, unless voids are formed at the interface. As in the case of the pseudo class I composites, several competing effects may be present. For example, the following may

AS-BONDED

BORON FILAMENT IN TITANIUM BORSIC FILAMENT IN TITANIUM



90 MINUTES AT 1600°F

FIGURE 9 Comparison of As-Fabricated and Heat-Treated Interfaces in Titanium/Boron and Titanium/Silicon-Carbide Coated Boron Composites.

affect fracture propagation: decrease in matrix ductility through solution of interstitials, such as carbon from silicon carbide; weakening of interface by voids; change in strength of filament as a result of interaction layer; change in distribution of strength of the filaments; and change in filament-matrix bond strength by increased keying (for example, growth of needles of titanium monoboride into matrix similar to keying by aluminum diboride needles). However, no detailed study has been made to assess the effect of each of these variables on the various mechanical properties.

V RELATION OF INTERFACE TO FRACTURE

Mechanical analyses of composites have been limited to those where the interface has either zero strength or adequate strength so that failure of the interface does not affect load distribution or failure of the composite. The latter may be termed "Strong Interface Theories." However, high strength metal matrices in combination with interfaces weakened by interaction require that attention be directed to the neglected intermediate area. Some simple analyses for fracture under these conditions in pseudo class I and class III systems have been made and are termed "Weak Interface Theories." A full review of these theories will be made in a forthcoming book.[10]

The more brittle interfaces formed by pseudo class I and class III composites are most likely to exhibit the behavior described by the Weak Interface Theories. Two principal failure modes under longitudinal loading can be considered. In the first, interface failure can occur without effect on composite failure, and, in the second, interface failure may trigger composite failure. In addition, the principal theory of weak interfaces considers a transition between the two modes. This theory was derived from application of a simple fracture mechanics approach to a class III system. Briefly, it was proposed that the interaction zone would be weak because it would contain typical growth defects and would fail at a strain determined by its strength and its elastic modulus. The latter was expected to have more influence on the fracture strain of the reaction zone, so that titanium diboride (elastic modulus 77 × 10^6 psi) would fail at 2500 microinch per inch, whereas titanium trisilicide (elastic modulus 37.5 × 10^6 psi) would fail at 4500 microinch per inch.

A filament with an annular ring of interaction compound will form cracks in the compound under longitudinal straining, and the network of cracks in the reaction zone surrounding the filament (as shown in Figure 10 for Ti-B) will create stress concentrations proportional to the square root of their length. Therefore, the length of the crack will be proportional to the thickness of the reaction zone. However, final failure will remain under the control of the intrinsic defects within the filament so long as the cracks in the reaction zone remain small. The first region in Figure 8 corresponds to the limit where the reaction zone defects do not affect failure. Fracture control is gradually assumed by the reaction zone defects as the thickness of the reaction zone increases across the second region. In the third region of Figure 8, failure of the composite under longitudinal loading. The excellent quantitative agreement

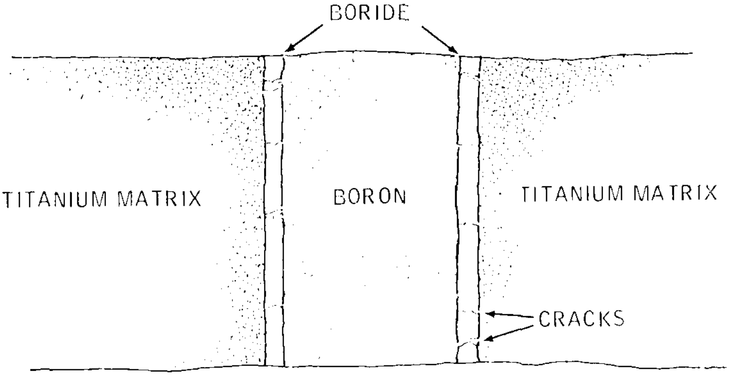


FIGURE 10. Schematic Illustration Showing Cracks at the Boride Interface Induced by Tensile Loading.

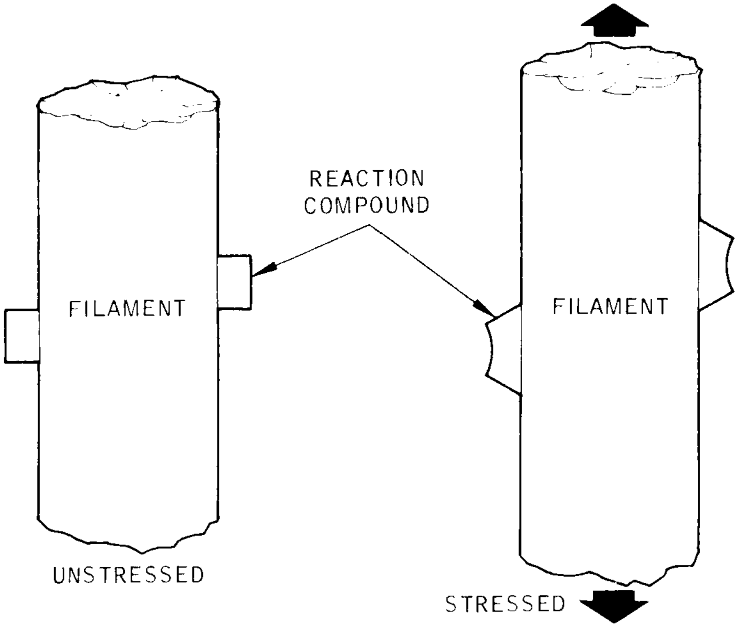


FIGURE 11 Model Showing Effect of Contact Area of Reaction Grain on Strain at Interface.

of predicted and observed failure strains for several systems for such conditions lends good support to this portion of the theory.

The effect of matrix support on the stress concentration (noted) in the filament can be appreciated by reference to Figure 10. The cracks in the reaction zone will be less deleterious on the filament when a strong and stiff matrix reduces the extent of crack opening at the matrix-end of the crack. This effect provides qualitative agreement with the observed effect of matrix strength on the tolerance for reaction.

The theory for Weak Interfaces in Class III systems cannot be applied directly to pseudo class I systems where the reaction occurs at random sites. It is proposed that the area on the filament at the base of each reaction site is the criterion for transition between regions such as those shown in Figure 4. The basis for this reasoning is shown in Figure 11. When the base of the reaction growth is small, the longitudinal strain equals that of the filament at the base of the reaction zone, but is reduced rapidly away from the filament. For this condition peeling may occur between reaction compound and filament to relieve stress, so that cracks are not likely to form at right angles to the longitudinal direction. However, when the compound has a large area in contact with the filament, relaxation by this mechanism is not possible at all points, and cracking can occur under the same conditions governing the behavior of Class III systems. Figure 11 shows a schematic example where cracking has become possible under tensile loading.

VI STATUS OF RELATIONSHIP OF INTERFACE AND MECHANICAL PROPERTIES

The relationship of longitudinal strength to the condition of the interface has been studied in some detail for pseudo class I and class III composite systems. Good agreement of experimental observation with previously developed theory has been obtained for class III systems, and the results for pseudo class I systems are in qualitative agreement with theory when appropriate modifications are made in the basic model. Some additional work is needed to examine details of the models to provide additional verification, particularly on the properties of aluminum diboride, and the influence of this compound on fracture of aluminum-boron composites. Also, work is needed to characterize other fabricated composite systems.

The major thrust of new work must be to develop further theory for Weak Interface Composites and to extend the analyses to conditions arising from other than longitudinal loading.

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