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### Journal

Ceramic Transactions, 46

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### Publication Date

1995-03-01



# Lawrence Berkeley Laboratory

UNIVERSITY OF CALIFORNIA

## Materials Sciences Division

Presented at the American Ceramic Society  
Annual Meeting, Indianapolis, IN, April 24-28, 1994,  
and to be published in Ceramic Transactions

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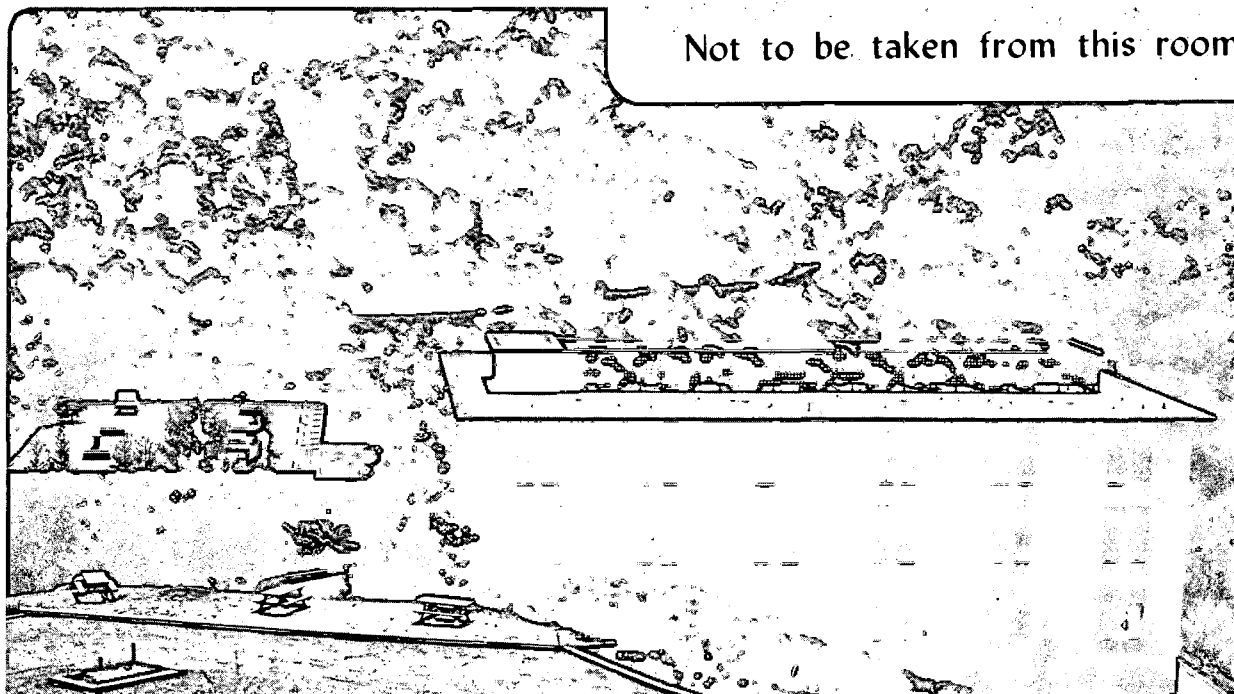
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March 1995

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COMPOSITES

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This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division, of the U. S. Department of Energy under Contract No. DE-AC03-76SF00098.

# PROCESSING AND CHARACTERIZATION OF SiC PLATELET / SiC COMPOSITES

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Hot pressed  $\beta$ -SiC and SiC matrix composites containing encapsulated  $\alpha$ -SiC platelets were prepared and investigated. The microstructures were characterized using electron microscopy, Auger electron spectroscopy, and x-ray diffraction. Prior to hot pressing, the platelets were either encapsulated with hydrated aluminum sulfate or yttrium hydroxycarbonate (later calcined to form alumina or yttria) from aqueous solutions, or oxidized to form a silica layer. The effect of these interfacial layers on toughness was described.

## 1. INTRODUCTION

SiC is an attractive material for high temperature structural applications due to its high strength, low creep rate, good thermal conductivity, and high oxidation resistance. A major drawback of SiC as a structural material resides in its modest fracture toughness. The incorporation of reinforcement phases to enhance toughness may be effective, but must be evaluated in conjunction with the role of sintering additives to ease potential densification difficulties.

Pressureless sintering of SiC to high density at temperatures upwards of 2000°C, using small amounts of boron and carbon, was first reported in 1975 [1]. Later, it was found that the addition of aluminum and carbon could also assist in densification [2]. The combination of Al, B, and C has been extremely effective as sintering additives [3, 4], providing enhanced densification particularly at lower sintering temperatures. Lin et al. [5] hot pressed silicon carbide to 96% theoretical density at 1650°C by adding 6wt% Al metal, 1wt% B metal, and 1wt% C-black. The possible role of these additives was not fully understood, and was only briefly discussed based on limited optical and x-ray analysis. The low sintering temperature, however, allows consideration of a range of reinforcement phases that would not be compatible at higher sintering temperatures either for chemical or

microstructural reasons. The low sintering temperature also significantly reduces manufacturing cost.

The dispersion of second-phase particles can improve the fracture toughness of ceramics. However, the choice of toughening phase(s) in a SiC matrix composite is restricted. High temperature stability and chemical compatibility of the second phases are necessary requirements during densification. It has been demonstrated that  $\text{TiB}_2$  [6, 7] and TiC [8, 9] particles can be effective in raising the fracture toughness of SiC. Both composites exploit the thermal expansion coefficient mismatch between the particulate and the matrix phases to generate a compressive hoop stress and a tensile radial stress around the particles, causing crack deflection and an increment in fracture toughness. However, at elevated temperatures, the internal stresses are relieved by creep, with a corresponding degradation of the mechanical properties [10].

SiC platelets have been shown to be effective reinforcements in alumina [11, 12], silicon nitride [13], and mullite [14]. The apparent chemical compatibility of  $\alpha$ -SiC platelets make them an ideal reinforcing phase in a  $\beta$ -SiC matrix. A high aspect ratio of the platelets could potentially provide a significant toughening [15]. However, without a protective barrier, the reinforcement of a  $\beta$ -SiC matrix with  $\alpha$ -SiC platelets is likely to be negated by the  $\beta \rightarrow \alpha$  phase transformation and strong bonding between the matrix and the platelets. In such a case, the resulting mechanical properties would be expected to be similar to those of monolithic silicon carbide. Therefore, fabrication of useful SiC platelet/SiC composites requires a means of protecting the platelets. Production of an interfacial phase can protect the integrity of the platelets during sintering and hopefully also achieve a weak bonding between the platelets and the matrix, thereby promoting crack deflection and/or debonding and pullout.

The isolation of the platelets from the matrix can be accomplished by the encapsulation of the platelets with an appropriate oxide prior to incorporation in the (matrix)  $\beta$ -SiC powder. A layer of silica, for example, is readily introduced by oxidation of the platelets in air. Ytria, alumina, or titania coatings from aqueous solutions onto a number of ceramic substrates have been demonstrated by De Jonghe and co-workers [11, 16-19]. A preliminary use of alumina-coated SiC platelets in a SiC matrix has shown promising mechanical properties, yielding in some instances a fracture toughness of over  $7 \text{ MPam}^{1/2}$  [20].

The present research studies hot pressed SiC composites reinforced with encapsulated SiC platelets using sintering additives similar to Lin's [5]. Transmission electron microscopy, scanning electron microscopy, auger electron spectroscopy, and x-ray diffraction are used to study the microstructures. The relative effects of the processing and the nature of interfacial layers of alumina, ytria, and silica in  $\alpha$ -SiC platelet /  $\beta$ -SiC composites are presented.

## 2. EXPERIMENTAL

### 2.1 Encapsulation of SiC Platelets in Oxides

The SiC platelets used in this study were provided by Third Millennium Technologies, Inc. The crystal structure was determined to be predominantly 4H  $\alpha$ -phase. The platelets were a -400 mesh grade, with diameters ranging from 3 to 30 microns (average 10 microns), and thickness ranging from 0.5 to 3 microns.

#### 2.1.1 Production of a silica layer on SiC platelets

SiC platelets were oxidized in air at 1300°C for 3 hours to form a layer of silica. The oxidation kinetics are known to follow parabolic kinetics [21-23], from which the thickness of the silica layer was estimated to be about 100 nm.

#### 2.1.2 Encapsulation with alumina

Kapolnek and De Jonghe[17] and Mitchell and De Jonghe[11] obtained heterogeneous precipitation of hydrated basic aluminum sulfate on powder surfaces using urea decomposition in aqueous solutions of salts. The SiC platelets used in this study were coated with this alumina precursor extending the procedures described in [11, 20] as follows.

The concentrations of the aqueous suspension were: 0.1 M/L hydrated aluminum sulfate (crystals, Fisher Scientific), 0.2M/L urea (Mallinckrodt), 10g/L platelets, and 0.75g/L PVP K30 (a polymeric dispersant, GAF Chemical Corporation). The suspension was vigorously stirred and maintained at 88°C for 24 hours. The coated platelets were annealed in inert gas at 880°C for 6 hours to convert the coating to crystalline  $\text{Al}_2\text{O}_3$ .

#### 2.1.3 Encapsulation with yttria

The hydrolysis of yttrium salts by urea decomposition was first used to synthesize monodispersed particles of yttrium hydroxycarbonate [24]. Garg and De Jonghe[16] extended this method to encapsulate powders and whiskers of silicon nitride with a yttria precursor. This method had also been used to coat alumina fibers [18]. In the present work, SiC platelets were coated in the same manner with the following modifications of the processing parameters.

A small amount of polymeric dispersant (PVP K30) was dissolved at room temperature in distilled water prior to the addition of SiC platelets. This suspension was thoroughly dispersed with magnetic stirring. An aqueous solution of yttrium nitrate (NOAH Teck. Co., TA) and urea was mixed with the suspended platelets in a flat bottom distilling flask. The final suspension contained 0.1 M/L yttrium nitrate, 0.75 M/L urea, 10

g/L SiC platelets, and 0.75 g/L PVP K30. The mixture was slowly heated on a heating mantel, capped with a water-cooled reflux condenser, to a final temperature of 65°C and held for 20 hours. Magnetic stirring was maintained to prevent the platelets from settling or agglomerating. After the reaction run, the suspension was allowed to settle, and the supernatant was discarded. The coated platelets were re-sedimented in water to remove any free precipitates, rinsed twice in acetone, and air dried at room temperature. The coated platelets were then calcined at 650°C for 12 hours to convert the coating to crystalline yttria.

## 2.2 Processing of Composites

Submicron size  $\beta$ -SiC powder (BSC-21, Ferro) was mixed in toluene with 6wt% metallic aluminum powder (H-10, Valimet, CA), 0.6wt% boron (high purity, sub-micron particle size, Callery Chemical Co., PA), and 4wt% apiezon (which was found to yield 50% carbon upon pyrolysis). The slurry was ultrasonically agitated for 5 minutes, followed by wet screening through a 325 mesh sieve. After drying, the powder mixture was screened through a 200 mesh sieve. Appropriate quantities of  $\alpha$ -SiC platelets with the various types of encapsulations (see Table 1) were then dry-mixed with the  $\beta$ -SiC powder, and cold compacted into 38 mm diameter discs at a uniaxial pressure of 35 MPa. The composites were hot pressed in graphite dies lined with graphite foil, at 1690°C, for 45 minutes, at 50 MPa, and under flowing argon gas.

Table 1. Processing, densities, and Fracture Toughness of silicon carbide and SiC Platelet/SiC Composites

Sample designation	SiC platelet (vol%)	Platelet surface treatment	Density (g/cm <sup>3</sup> )	Fracture toughness (relative value)
#1	0	/	3.17	1
#N	20	none	3.11	1.2
#5	10	oxidized	3.18	1.1
#Y	14	yttria coated	3.18	1.2
#2	8	alumina coated	3.20	1.2
#A	20	alumina coated	3.23	1.6
#HFA	20	etched and alumina coated	3.23	1.5
#A2	28	alumina coated	3.27	not measured

## 2.3 Mechanical Testing

A four-point bend testing was used to measure fracture toughness. Beams of 2.5 by 2.8 by 28 mm were cut from the hot pressed billets. The tensile surfaces were polished to a 3  $\mu$  finish, and the tensile edges were beveled to eliminate edge flaws. An elliptical



flaw was then introduced on the tensile surface with a Knoop indentation. The flaw was made parallel to the hot pressing direction and perpendicular to the long axis of the beam. The beams were tested at an outer span of 25.4 mm (1 in.) and an inner span of 9.5 mm (3/8 in.). The crosshead speed was of 0.05 mm/min. The fracture toughness was determined from the relation [25]:

$$K_{Ic} = \frac{2}{\sqrt{\pi}} \sigma_f \sqrt{a}$$

where  $\sigma_f$  is the fracture stress and  $a$  is the initial flaw radius of the indentation measured on the fracture surface using a scanning electron microscope (SEM).

### 3. RESULTS AND DISCUSSION

#### 3.1 Encapsulation of SiC platelets

The microstructural development of the coating of alumina precursor has been described elsewhere [11]. Figure 1 shows an SEM micrograph of alumina coated platelets after calcination. The coating presents a good coverage on the platelets and has a thickness of 1 to 2  $\mu\text{m}$ . Experiments also show that etching of the platelets with HF, prior to the application of alumina coating, does not significantly affect the morphology nor the thickness of the alumina coating under identical coating conditions.

Figure 2 exhibits an SEM image of the yttria coating on SiC platelets. This coating also completely covers the surface. X-ray diffraction determines that the coating has been converted to crystalline yttria after calcination. The heating rate and the soaking temperature of the calcination process have to be carefully controlled to avoid extensive cracking of the deposit. Annealing at excessive temperatures (e.g., 900°C) induces film cracking. The insert in Figure 2 shows a crack, the edge of which indicates the yttria film is approximately 0.25 micron thick.

Urea decomposes upon heating in aqueous solutions, thereby causing the pH of the system to progressively increase [26]. Yttrium hydroxycarbonate (YBC) forms by the hydrolysis of yttrium nitrate in a urea-containing aqueous solution [24]. The pH decreases upon formation of yttrium hydroxycarbonate. Measurements indicate that the pH of the suspension drops initially due to the ionization of yttrium nitrate. Subsequently, the pH rises as a result of continued urea decomposition. After two hours, the pH stabilizes at approximately 5.5, due to the rate balance between urea decomposition and precipitation of the yttria precursor, until the coating run is terminated at 20 hours. The constant pH should correspond to a yttrium hydroxycarbonate concentration between the homogeneous nucleation and the heterogeneous nucleation limits, as is traditionally shown in a LaMer [27] diagram. Therefore, nuclei could form heterogeneously either on the surface of the platelets or on existing precipitates. Simultaneously, the already existing precipitates will grow. Since the concentration never reaches the homogeneous nucleation limit, free

precipitation in the solution is minimized. The morphology of the obtained coating is consistent with such a mechanism. Experimental observation shows that the coating initially appears as discrete particles on the surface of the SiC platelets. These precipitates grow, meanwhile the population of the precipitates increases with time. Eventually the coating covers the entire surface.

### 3.2 Sintering and Characterization

SiC matrix composites with up to 35wt% of alumina-coated SiC platelets (approximately 28vol% of SiC platelets) are hot pressed to nearly full density at 1690°C and 50 MPa. Densities are measured using the Archimedes principle in distilled water, and the results have been listed in Table 1. As the exact quantities of the encapsulation phases are difficult to measure and the quantities of sintering additives are relatively large, the theoretical densities of the composites are not accurately known. However, the relative densities of the SiC composites containing encapsulated platelets are estimated to be 98% and up. The sintered densities are also examined by optical and scanning electron microscopy of polished surfaces. Figure 3 shows a polished surface of the composite with 35wt% alumina coated platelets. The bright areas are the alumina coatings. Very few, small isolated pores are observed in the matrix.

Figure 4 shows the typical grain sizes of silicon carbide, ranging from submicron to 2 microns. Most of the grains are equiaxed. Less than 10% of the grains are elongated along the planes of most stacking defects. Electron diffraction indicates that all these grains have a cubic crystal structure ( $\beta$ -phase). The low processing temperature apparently suppresses the  $\beta \rightarrow \alpha$  phase transformation. Figure 5 depicts a typical grain boundary of  $\beta$ -SiC grains. Lattice fringes from both grains extend into the interface because this grain boundary is tilted. However, it appears that the grain boundaries are free of amorphous phase.

The sintering additives, especially Al, are present in concentrations well above their solubilities in SiC. Secondary phases are therefore expected to form. Figure 6 is a fractograph of a "monolithic" silicon carbide (without the addition of SiC platelets). Secondary phases with dimensions of about 10  $\mu\text{m}$  are apparent. It can be also noted that the secondary phases are preferentially oriented with the long axis perpendicular to the hot pressing direction. Moreover, TEM determines that these "pockets" are comprised of many grains approximately 1-2  $\mu\text{m}$  in size. This suggests that these regions exist as liquid during hot pressing. With the presence of aluminum, carbon, and boron, (and probable oxygen from the surfaces of Al and SiC powders), two liquid phases are plausible at the sintering temperature. One is associated with the eutectic reaction between  $\text{Al}_2\text{O}_3$  and  $\text{Al}_4\text{O}_4\text{C}$  [28]; the other one is  $\text{Al}_3\text{B}_4\text{C}_7$  [29].

Minor peaks in bulk X-ray diffraction data confirms the presence of  $\text{Al}_4\text{O}_4\text{C}$  and  $\text{Al}_3\text{B}_4\text{C}_7$ . Electron diffraction determines both ternary phases form within the "pockets" of

secondary phases. XRD also indicates the existence of  $\text{Al}_2\text{O}_3$  and  $\text{Al}_4\text{C}_3$ . EDS analysis shows that the secondary phase regions contain Al, but are absent of Si. (The EDS spectrometer used is not sensitive to B, C, and O.) The average atomic ratio of Al vs Si over a large area is found to be 8:100, which is close to the atomic ratio of the raw materials (10:100). Figure 7 shows Auger electron spectra acquired of various regions of the fracture surface. Figure 7a is acquired of the matrix. Figures 7b and 7c analyze two neighboring grains inside a single secondary phase region (Figure 6). Figure 7b indicates mainly Al and O, with minor amounts of C and B; Figure 7c indicates mainly Al and C, with minor quantities of B and O. Therefore, the microstructural observations and the spectra results conclude that the secondary phase regions are composed of not only multiple grains, but also multiple phases.

Sintering of a composite with a large volume fraction of reinforcement phase, such as platelets, can be hindered by the percolation of the second phase [30]. Making composites with encapsulated reinforcement phases have been shown to ease densification [11, 17]. Typically, the coated phase would have a large volume fraction and becomes the matrix of the composite. However, a similar principle applies in the densification of coated SiC platelet / SiC composites. The oxide coating prevents the platelets from physically contacting each other. In addition, creep of the oxide coating during hot pressing reduces stress shielding and eases sliding of the platelets relative to each other. All these factors facilitate densification. A SiC composite containing 35wt% alumina-coated platelets (28vol% SiC platelet) was successfully hot pressed at 50 MPa to nearly full density at a temperature below 1700°C (Figure 3).

The coatings are also essential to protect the integrity of the platelets (i.e. to prevent the  $\beta \rightarrow \alpha$  phase transformation, or the sintering together of the platelets and the matrix). Experimental observation shows that a composite made with the uncoated platelets suffers from sintering reaction between the platelets and the matrix grains. In this case, the composite acts similar to monolithic SiC because of lack of a phase barrier, as will be shown in mechanical testing. The coating of alumina separates the  $\alpha$ -SiC platelets from the  $\beta$ -phase matrix (Figure 3). However, the coated yttria film (Figure 2) has formed clumps after sintering, and the yttria coating is no longer continuous (Figure 8). A reaction may also have occurred at the platelet/yttria interface. The silica layer formed by oxidation of the platelets was not resolved by SEM and may have been reduced during processing. (TEM analysis on these concerns are currently underway.)

### 3.3 Mechanical Testing

Fracture toughness is measured by a four-point bend test, with a well defined elliptical flaw made by Knoop indentation on the tensile surface (Figure 9). The measured fracture toughness results have been summarized in Table 1. Fracture toughness of the alumina-coated platelets reinforced SiC composites are significantly increased over that of monolithic SiC. Addition of 25wt% alumina-coated SiC platelets (approximately 20vol%

platelets) achieves a 60% increase in the fracture toughness. Etching SiC platelets prior to the coating process, shows no effect in mechanical behavior of the composite. Figure 10 displays the fracture surface of the composite. Crack path deflection is present at the platelet surface. This crack deflection alters the crack tip stress intensity, resulting in an improvement in toughness [15].

Platelet encapsulation proves to be essential in this composite system. As has been shown in the previous section, the addition of uncoated SiC platelets leads to sintering together of the platelet and the matrix. It is observed that the crack breaks through most of the uncoated platelets, resulting in a minimal increase in toughness over that of monolithic SiC (Table 1). A thin layer of silica, introduced by oxidation, also does not produce effective separation between the matrix and platelets, with only a limited increase in toughness. Two possible reasons can be cited. First, the oxidation product has been considered as a mechanically strong protective layer [21-23], thereby suggesting the bonding between silica and silicon carbide is very strong. Strong interfacial bonding is well known to be disadvantageous in ceramic composites. Second, there is a possibility that the silica layer is completely reduced by sintering additives during hot pressing.

The composite with yttria-coated platelets shows very limited increase in toughness over that of monolithic SiC (see Table 1). The bonding strength between SiC and yttria is not well known. Moreover, the morphology of the yttria coating has changed during sintering, and it does not fully separate the platelets from contacting the matrix. Therefore, the platelets may have been partially sintered together with the matrix.

## **SUMMARY**

Silicon carbide and SiC matrix composites with up to 35% of coated SiC platelets were hot pressed to near full density at 1690°C and 50 MPa. Liquid phase sintering was believed to play a role in densification. Encapsulation of SiC platelets with oxide coatings prevented direct contact of the platelets (platelet network), and eased densification of the composites. An alumina coating proved to be most effective to protect the integrity of SiC platelets, and to promote crack deflection, thereby resulting in increased fracture toughness.

## **Acknowledgment**

The authors gratefully acknowledge the help of J. P. Daniel and T. D. Mitchell, Jr. with developing the coating processes. This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division, the U. S. Department of Energy under Contract No. DE-AC03-76SF00098.

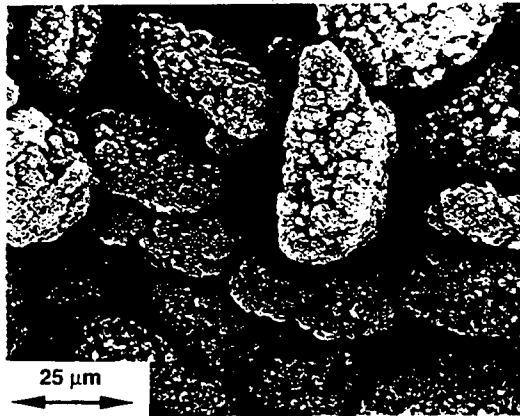


Figure 1

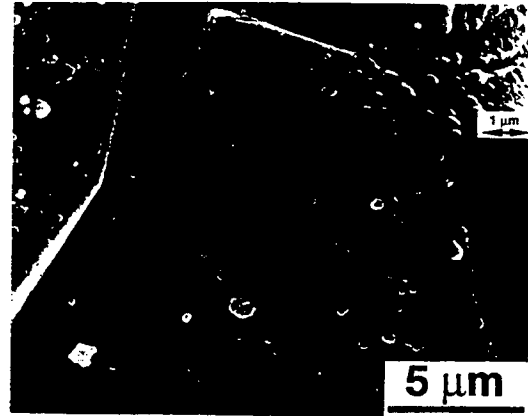


Figure 2

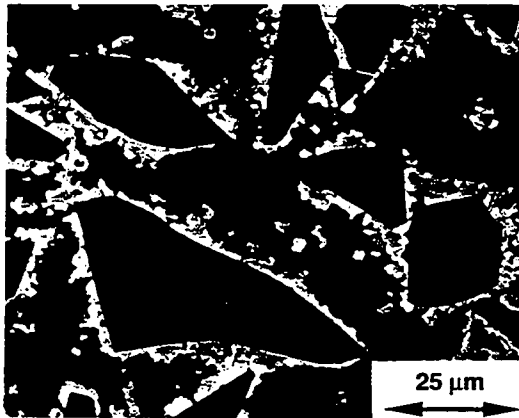


Figure 3



Figure 4

Figure 1 Scanning electron micrograph (SEM) of alumina coated platelets.

Figure 2 SEM of yttria-coated SiC platelets. The yttria film completely covers the platelets. The inserted picture shows a crack (induced by calcination at 900°C) through the coating. From the side view of the crack, the coated film is determined to be approximately 1/4 micron thick.

Figure 3 SEM micrograph of a polished surface of the SiC composite with 35wt% of alumina-coated SiC platelets in a direction perpendicular to the hot pressing direction. The composite is nearly full dense. The coating acts as a barrier to protect the integrity of the platelets.

Figure 4 Transmission electron micrograph (TEM) of SiC matrix grains. The grain size ranges from sub-micron to two microns.

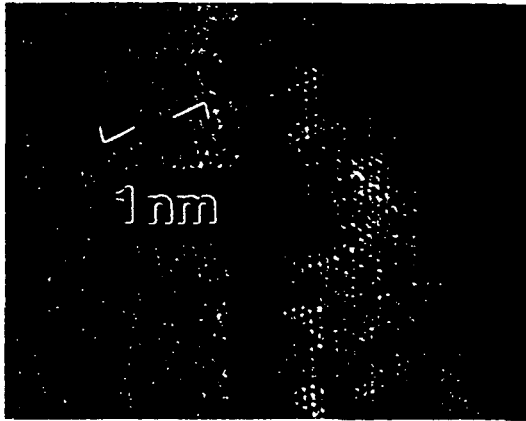


Figure 5

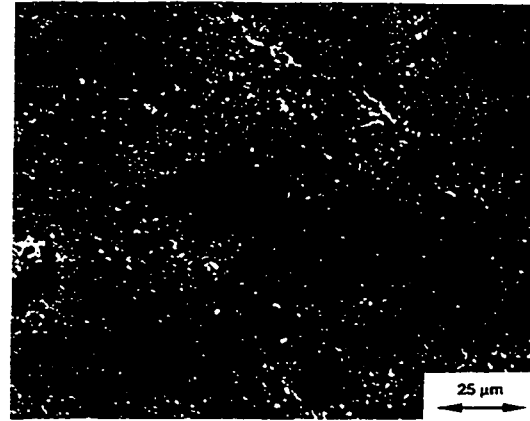


Figure 6

Figure 5 High resolution electron micrograph (HREM) of a typical SiC-SiC grain boundary. No amorphous interphase was observed.

Figure 6 SEM micrograph of the fracture surface of "monolithic" silicon carbide. Regions of secondary phase (approximately 10 microns long) are evident, which are determined to be composed of many 1-2 microns size grains.

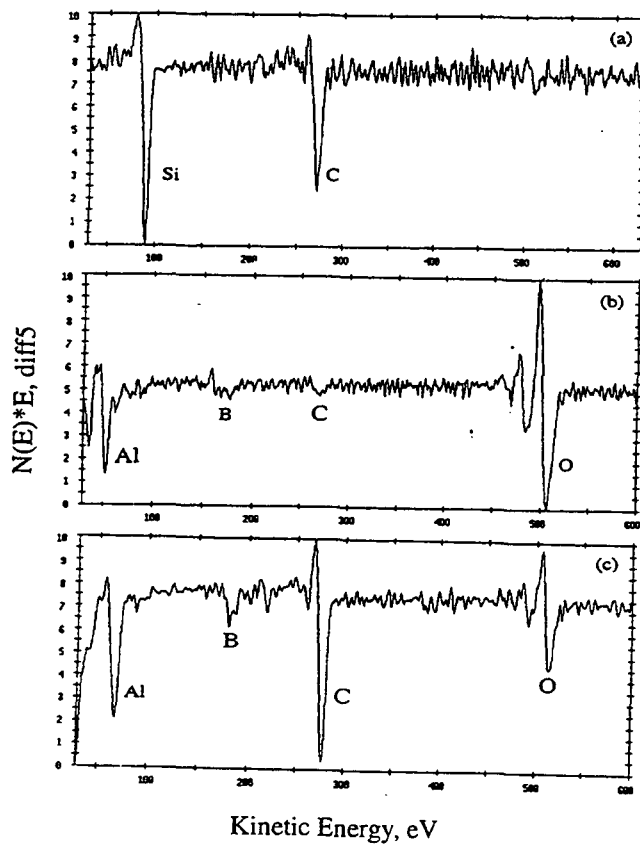


Figure 7 Auger electron spectra of the fracture surfaces of the "monolithic SiC" specimen are acquired (a) in the matrix (away from the secondary phases), and of two grains, (b) and (c), inside a single region of the secondary phase.

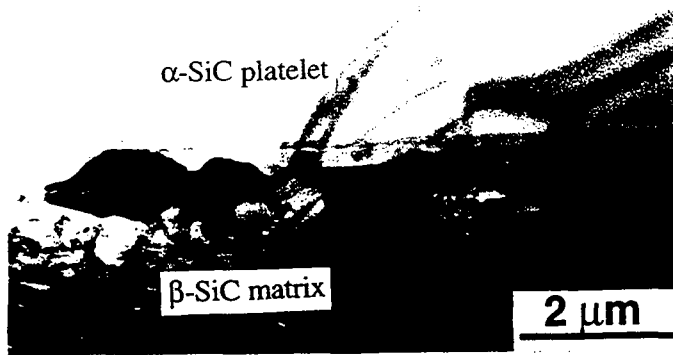


Figure 8

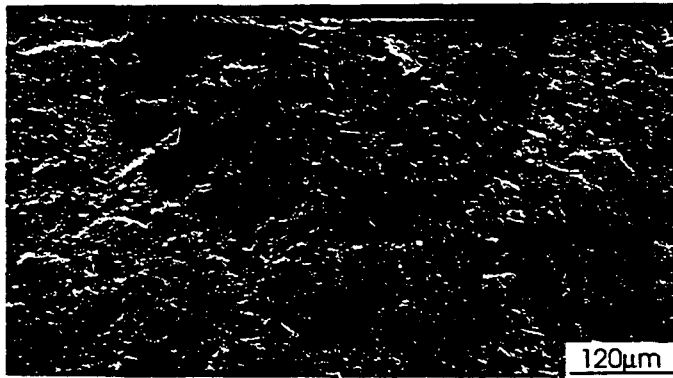


Figure 9

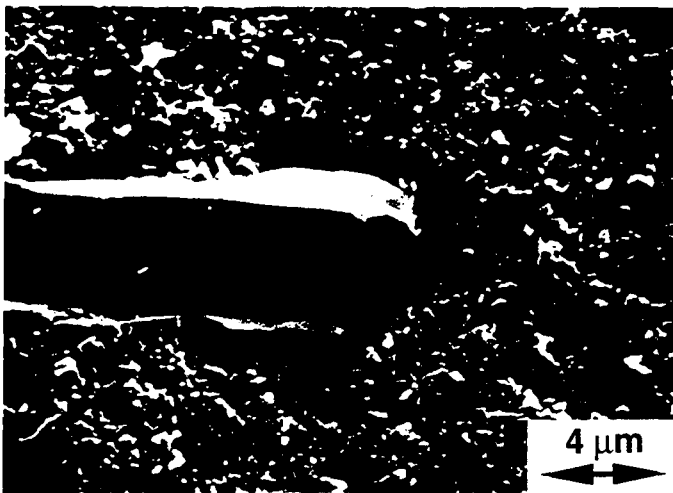


Figure 10

Figure 8 TEM micrograph of SiC composite with ytria-coated SiC platelets.

Figure 9 SEM micrograph of an elliptical flaw induced by Knoop indentation.

Figure 10 Fractograph of alumina-coated SiC platelet / SiC composite. Some crack deflection has occurred at the platelets.

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