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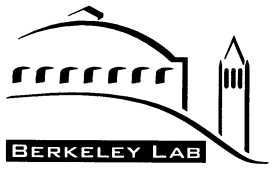

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FATIGUE OF CERAMICS AT ELEVATED TEMPERATURES: MICROSTRUCTURAL DESIGN FOR OPTIMAL PERFORMANCE

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Abstract: The high-temperature mechanical properties of an *in situ* toughened silicon carbide have been examined at temperatures from ambient to 1300°C with the objective of optimizing structural performance. It was found that elevated temperatures up to 1300°C do not severely compromise the strength, toughness and fatigue resistance of ABC-SiC, compared to properties at room temperature. Mechanistically, the damage and shielding mechanisms governing cyclic fatigue-crack advance are essentially unchanged between ~25° and 1300°C, involving a mutual competition between intergranular cracking ahead of the crack tip and interlocking grain bridging in the crack wake. The unusually good high-temperature properties of ABC-SiC are attributed to *in situ* crystallization of grain-boundary amorphous phase, which on subsequent cooling also enhances the ambient-temperature mechanical properties.

Keywords: Silicon carbide, toughness, fatigue, grain-boundary phase.

1 Introduction

As a high-temperature structural material, silicon carbide (SiC) ceramics offer many advantages, including a high melting temperature, low density, high elastic modulus and strength, and good resistance to creep, oxidation and wear. This combination of properties makes it a promising candidate for use in such applications as gas turbines, piston engines and heat exchangers, although its use to date has been severely limited by its poor toughness properties.

The low inherent fracture toughness of conventional SiC ceramics (K_c is typically ~2-3 MPa$\sqrt{m}$) can be improved, however, by several processing and reinforcement routes. One approach is to produce a composite, which is typically accomplished by incorporating continuous fibers, whiskers, platelets, or second phase particles [1]. For monolithic ceramics, *in situ* toughening can also be effective with microstructures consisting of elongated grains encased with a residual glassy film. Such microstructures induce intergranular fracture and are thus effective in promoting toughening from the consequent crack bridging, as has been well demonstrated in silicon nitride (Si_3N_4) ceramics [2]. The problem in monolithic ceramics is that although the amorphous grain-boundary film is critical for good low-temperature toughness, its

presence at high temperatures provides a preferred site for softening and creep cavitation, which typically limits the high-temperature strength, creep and oxidation resistance.
Recently, in an attempt to avoid such tradeoffs between low-temperature toughness and high-temperature strength, a monolithic SiC with additions of Al metal as well as B and C (termed ABC-SiC) has been developed. At ambient temperatures, ABC-SiC exhibits fracture toughnesses as high as 9 MPa√m with strengths of ~650 MPa [3], mechanical properties that are among the highest reported for SiC. The high toughness has been attributed to various crack-bridging processes in crack wake resulting from the intergranular crack path [4]; specifically, crack-tip shielding from both elastic bridging and frictional pullout of the grains provide the major contributions, with the frictional pullout component being the more potent. At elevated temperatures, however, a critical factor governing properties is the viscosity of the grain-boundary phase, which results from the presence of sintering additives that are present as densification aids [5]. The softening of this phase can severely degrade properties [6]; however, *in situ* crystallization can provide a potent means to increase its viscosity at high temperatures.

In the present work, we examine how the high temperature mechanical properties of ABC-SiC are affected by the nature of the grain-boundary phase, and investigate whether its superior room-temperature strength and toughness properties [3,4] can be retained at high temperatures. In addition, we specifically investigate the high-temperature cyclic fatigue properties, in part due to the contradictory nature [7-14] and paucity of published results on this topic to date.

2 Experimental Procedures

ABC-SiC was processed with submicron β-SiC starting powders, which were mixed with additions of 3 wt.% Al powder (nominal particle diameter ~5 μm), 0.6 wt.% B powder, and 2 wt.% C (as Apiezon wax). The Apiezon wax, which also served as a binder, was dissolved in toluene, and the other powders added; the resulting suspension was agitated ultrasonically for 5-10 min to minimize agglomerate formation, and then stir-dried. The dried material was then ground in a mortar and pestle prior to sieving through a 200 mesh screen. Hot-pressing was conducted for 1 h at 1900°C, at 50 MPa pressure in a flowing argon atmosphere, in a graphite die using green compacts that were previously formed by uniaxial compression at 35 MPa in a metal die. The resulting hot-pressed disks were surface-ground, and polished to a 1 μm diamond powder finish, prior to the machining of samples.

Fracture toughness (K_c) and cyclic fatigue-crack growth tests were performed on disk-shaped compact-tension DC(T) specimens (28 mm wide, 3 mm thick), containing "large" (> 3 mm) through-thickness cracks. Toughness testing was performed on fatigue pre-cracked specimens at temperatures between 25 and 1300°C; in addition, the toughness of as-processed samples was compared with those following a prior thermal exposure for 85 hr at 1300°C. Strength tests were performed in four-point bend on 3 mm × 3 mm × 30 mm beam specimens.

Cyclic fatigue-crack growth testing was performed in general accordance with ASTM Standard E-647. Specifically, DC(T) specimens were cycled at 550, 850, 1200 and 1300°C under automated stress-intensity stress intensity (K) control while maintaining a constant load ratio (ratio of minimum to maximum applied loads) of R = 0.1 at frequencies of ν = 3 and 25 Hz (sinusoidal waveform); corresponding tests at 25°C were carried out under the same conditions only at frequencies of 25 and 1000 Hz.

Toughness and crack growth experiments were conducted on computer-controlled servo-hydraulic mechanical testing machines. Elevated temperature tests were performed in an environmental furnace with graphite elements (temperatures to within ±1°C). The environment at elevated temperatures was flowing gaseous argon at atmospheric pressure; corresponding tests at 25°C were conducted in room air. Crack lengths were continuously monitored *in situ* at elevated temperatures by a direct-current electrical-potential drop technique [15,16].

Fracture surfaces and crack profiles were imaged in a field-emission scanning electron microscope (FESEM). The atomic and structural nature of the grain boundaries was also examined using high-resolution transmission electron microscopy (HRTEM). Chemical compositions of any grain-boundary phases were analyzed using X-ray energy-dispersive spectrometer (XEDS) with a 8 nm probe. In addition, the microstructure and damage in regions directly ahead of the crack tip were examined in the transmission electron microscope (TEM). Specifically, 3-mm diameter TEM foils were taken from the crack-tip region of fracture and fatigue test specimens such that the crack line was parallel to the axis of the foil with the crack tip located ~500 μm away from the foil center. The foils, which were ground down to 20 μm using a precision dimpling machine and further thinned by argon ion milling, were examined on a Philips CM200 microscope, operating at 200 kV.

3 Results and Discussion

As-processed microstructure

The microstructure of as-processed ABC-SiC consisted of a network of interlocking plate-like grains of 5 vol.% β-phase (cubic polytype 3C) and 95 vol.% α-phase (49 vol.% 4H and 46 vol.% 6H hexagonal polytypes), with a maximum grain aspect ratio of ~4 to 5. Between the grains, an amorphous grain-boundary film, typically ~1 nm thick and rich in Al, O and C, can be seen in the hot-pressed material (Fig. 1). The remaining sintering additives were found to form bulk secondary phases at triple-junctions and multigrain-junctions [3]. In addition, in specific α-SiC grains, the presence of isolated dislocations could be seen; these dislocations, which were always in very low densities, are likely to be partial dislocations bounding the stacking faults.

Strength and fracture toughness

The fracture toughness of the as-processed ABC-SiC was measured as K_c = 6.2 MPa√m at 25°C and 4.3 MPa√m at 1300°C. However, annealing for 85 h at 1300°C led to a ~20% increase in these values (Table I). Moreover, such thermal annealing can also increase the high-temperature strength; specifically, annealing for 7 days at 1500°C led to a four-fold increase in the bend strength in this ceramic at 1300°C [17]. It should be noted that the corresponding strength and toughness properties of commercial SiC (Hexoloy) are a factor of ~2 to 3 times lower.

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	Fracture toughness (MPa√m)		Strength (MPa)
	25°C	1300°C	25°C
as-processed	6.2	4.3	620
pre-exposed	7.2	5.2	604
SiC (Hexoloy)	2.5	1.9	380

Table I Fracture toughness and strength in as-processed (amorphous grain boundaries) and 1300°C pre-exposed (crystallized grain boundaries) ABC-SiC tested at 25 and 1300°C. Data for Hexoloy are listed for comparison.

Following such prolonged high-temperature annealing, HRTEM imaging indicated that a large majority of the grain-boundary glassy films had become fully crystallized in both toughness and strength testing specimens (Fig. 2); this was found to occur at all temperatures above ~1100°C for times in excess of ~5 h. Moreover, the average thickness of grain-boundary film decreased to less than ~1 nm, with a corresponding increase in the Al concentration revealed by the XEDS spectra. Such crystallization of the grain-boundary phase clearly would minimize softening and grain-boundary sliding and possibly induced flaw healing within this region, which would account for the four-fold increase in strength.

In contrast, although such crystallization is less common in Si_3N_4, when it does occur it has been found to *degrade* the subsequent low temperature strength and toughness [18]. Remarkably, in ABC-SiC, after crystallization the strength at 25°C was increased by ~16%, and the toughness increased by some 16 to 21% at both 25 and 1300°C (Table I). Since microstructural changes often have opposing effects in ceramics, e.g., coarsening grain sizes in Al_2O_3 and Si_3N_4 SiC can promote toughness yet decrease strength, the process of *in situ* (or thermally-induced) crystallization of the grain-boundary phase in ABC-SiC is particularly effective as it acts to increase both strength and toughness, with only a small reduction in properties at elevated temperatures (up to 1300°C).

It is uncertain why the crystallization of the grain-boundary films should lead to a slight improvement in the subsequent toughness and fatigue resistance at room temperature. However, as stated above, the mechanisms of both fracture and fatigue are associated with grain bridging, resulting from the frictional tractions generated via contact of opposing crack faces [19]. The pullout resistance, represented by bridging stress, acts to reduce the near-tip crack-driving force for crack extension; its magnitude is linearly proportional to the frictional coefficient between sliding grain faces [20]. Crystallization of the grain-boundary phase is expected to increase the frictional coefficient, and the resulting increase in the bridging stress may well provide the origin of the enhanced grain bridging.

Fatigue-crack growth behavior

Fig. 3 illustrates the variation in fatigue-crack growth rates, da/dN, with applied stress-intensity range, ΔK, for ABC-SiC at a load ratio of 0.1; shown are the effects of temperature and loading frequency. It can be seen that at both ambient and elevated temperatures, crack-growth rates display a marked sensitivity to the stress intensity regardless the loading frequency; this is a

common characteristic of monolithic ceramics at low homologous temperatures [21]. In terms of a simple Paris power-law formation: $da/dN = C\,\Delta K^{m}$, (where C and m are scaling constants), these data show a Paris exponent m between 35 and 68.

Although changing the frequency over the range 3 to 1000 Hz has little effect on fatigue-crack growth behavior, growth rates are accelerated with increasing temperature. Indeed, ΔK_{th} fatigue thresholds decrease from just over 5 MPa√m at 25°C to between 3.3 and 3.4 MPa√m at 1200°C; interestingly, there is no further decline at 1300°C. Mechanistically, the lack of a frequency effect in ABC-SiC at ambient temperatures is expected as crack advance occurs via predominantly intergranular cracking ahead of the tip, balanced by shielding by grain bridging in the wake, both essentially rate-insensitive processes. However, the absence of a frequency effect at elevated temperatures is much more surprising, particularly since comparable materials, such as Si_3N_4, Al_2O_3 and silicide-matrix ceramics, show a marked sensitivity to frequency at temperatures due to the onset of creep damage above ~1000°C [7,10-12,22]. As described below, fatigue fracture mechanisms at 850 to 1300°C in the present material are effectively identical to those at room temperature, which is consistent with the absence of significant creep phenomena at, and below, 1300°C.

Similar to the toughness properties, the fatigue-crack growth resistance of ABC-SiC was also found to be far superior to commercial SiC (Hexoloy) at both room and elevated temperatures. Results at 25 to 1300°C, which are compared to ABC-SiC in Fig. 3, indicate that Hexoloy fails catastrophically, with no cycle-dependent cracking, at stress intensities some 40-50% lower than the fatigue thresholds in ABC-SiC. Such extremely brittle behavior is attributed to the absence of toughening from crack bridging behind the crack tip, resulting from its fully transgranular mode of fracture (Hexoloy shows little to no evidence of an amorphous grain-boundary film).

A comparison of the crack-growth velocities under cyclic and static (sustained) loading indicated that at both ambient and elevated temperatures, growth rates in ABC-SiC are significantly faster under cyclic loads than under sustained loading (static fatigue) at equivalent stress-intensity levels. Such behavior has been observed previously in Mg-PSZ [23], Al_2O_3 [24] and Si_3N_4 [25], and is consistent with the fact that at low temperatures, crack-advance (damage) mechanisms are identical under both types of loading; specifically, it is the cyclic-loading induced degradation in wake shielding that accelerates growth rates under cyclic loads. In contrast, the sustained-load mechanisms at elevated temperatures are generally far more damaging in such ceramics as Al_2O_3 and Si_3N_4 [7,11-13] because of the onset of creep damage, which can cause softening and cavitation in the grain-boundary films [8]. Such behavior was not seen in ABC-SiC at temperatures up to 1300°C.

SEM of fracture surfaces and crack profiles in ABC-SiC at 1300°C revealed a predominantly intergranular fracture under both static and cyclic loads (Fig. 4a,c), with extensive interlocking grain bridging behind the crack tip (Fig. 4b,d). Also noticeable on the cyclic fatigue surfaces was the presence of debris, formed by wear and abrasion of the bridging crack faces during cycling. Fracture surfaces at 25°C were essentially identical [4], implying that a similar sequence of mechanisms, namely intergranular cracking coupled with degradation of the resulting wake zone of bridging grains, is active at both temperatures. TEM studies of regions in the immediate vicinity of the crack tip provided direct confirmation of these observations. Akin to behavior at

room temperature, crack extension at 1300°C under both sustained and cyclic loading was predominantly along the grain boundaries with no indication of cavitation damage ahead of the tip or viscous-phase bridging in the wake (Fig. 5). HRTEM observations also revealed that the grain-boundary films had fully crystallized (e.g., Fig. 2) *in situ* during the high-temperature fatigue tests, which typically lasted ~3 to 10 days at 1200 or 1300°C.

The absence of creep damage and/or viscous softening of the grain-boundary phase at 1300°C in ABC-SiC is quite startling. In Si_3N_4 [9,10], Al_2O_3 [11] and silicide-matrix composites [22] at these temperatures, grain boundary cavitation, microcracking zones, and viscous-phase bridging are commonly observed. It is apparent that the unique high-temperature characteristics of ABC-SiC appear to be a result of the *in situ* crystallization of grain-boundary glassy phase.

Even though the primary mechanisms of damage (intergranular cracking) and shielding (grain bridging) are apparently unchanged between 25 and 1300°C in ABC-SiC, there is a small change in the fatigue-crack growth resistance in that fatigue thresholds ΔK_{th} are approximately 30% lower at the higher temperature. This may be rationalized by considering the nature of grain bridging and its degradation under cyclic loading due to frictional wear [26,27]. The pullout resistance from frictional tractions generated via sliding contact of opposing crack faces is proportional to the normal stress acting on the interface, which in turn is a function of the residual stress resulting from thermal expansion anisotropy during cooling from the processing temperature. As the residual stresses will "anneal out" with increasing temperature, the normal stress will decrease. However, once crystallization of grain-boundary phase occurs above ~1100°C, as stated above, the frictional coefficient may be expected to be higher. Thus, the minimal change in fatigue properties between 25 and 1300°C can be related to (i) the lack of any apparent change in mechanisms, (ii) the fact that the decrease in residual stress with temperature is compensated by an increase in the frictional coefficient, and (iii) the absence of significant creep damage, the latter effects being associated with the *in situ* crystallization of the grain-boundary phase. Such a result is consistent with studies in other ceramics that show a beneficial effect of crystallization on oxidation and mechanical properties at high temperatures [28,29].

The beneficial effect of the crystallization of glassy phase is also clearly demonstrated by the creep behavior. Following an initial primary creep stage with primary strain $\varepsilon_p \approx 0.001$, steady-state creep rates, $\dot{\varepsilon}$, in ABC-SiC are relatively low, i.e., $\dot{\varepsilon} < 2\times10^{-10}$ /s at 1200°C and $\dot{\varepsilon} = 6\times10^{-10}$ /s at 1300°C under the load of σ = 100 MPa [17]. In comparison, Hexoloy exhibits far higher creep rates, i.e., $\varepsilon_p \approx 0.018$ and $\dot{\varepsilon} \approx 8.8\times10^{-9}$ /s at 1200°C (σ = 139 MPa) [17]. Similarly, in Si_3N_4-GN-10, creep rates are an order of magnitude faster, i.e., $\varepsilon_p \approx 0.009$, $\dot{\varepsilon} = 1.78\times10^{-9}$ /s at 1300°C (σ = 100 MPa) [30]. ABC-SiC also exhibited low creep rates at stresses that were a much higher percentage of its fracture strength than the silicon nitrides; for example, the Si_3N_4/SiC nano-composite had a similar creep rate in bending at 1400°C, but with a bend strength 895 MPa [31], compared to ~ 300 MPa for ABC-SiC.

4 Concluding Remarks

Structural ceramics have often been regarded as exhibiting a conflict between toughness, strength, and fatigue resistance. Indeed, this conflict is not unlike the competition between brittleness and strength in metallic systems. A summary of many results on creep and toughness at 1300°C for SiC and Si_3N_4, extracted from the literature, is shown in Fig. 7, to illustrate this paradigm, and to show that ABC-SiC can be processed to negate it. Essential in the success of

retaining simultaneously strength, toughness, fatigue and creep resistance, is the creation of a crystalline grain-boundary film that retains a stable structure and composition up to high temperatures. In ABC-SiC, this can be achieved *in situ* at elevated temperatures, or by a high temperature annealing process. Where high temperature post-processing treatments of this kind are relatively uncommon for enhancing the properties of ceramics, their value is evident here. It should be added that the beneficial effects of the Al, B, and C additives and the subsequent heat treatments could, at this time, not have been predicted from either first principle computations or existing lore, but rather resulted from extensive experimentation and application of general materials science principles. It is hoped that, in the future, computational efforts may assists in further developing the necessary nature and role of the grain-boundary films, to improve further the mechanical behavior of these high temperature structural ceramics.

5 Conclusions

The high-temperature mechanical properties, including strength, fracture toughness and cyclic fatigue properties, of an *in situ* toughened silicon carbide sintered with Al, B and C (ABC-SiC) have been studied, and related to the corresponding microstructural and mechanistic characteristics. Based on this work, the following conclusions can be made:

(1) High-temperature annealing at 1100°C and above was found to lead to a remarkable improvement in strength and toughness properties. For example, the ~30% decrease in fracture toughness between 25 to 1300°C was partially offset by prior annealing for 85 h at 1300°C, which produced a ~20% increase of toughness at both 25 and 1300°C. Such annealing treatments were found to result in full crystallization of the glassy grain-boundary phase.

(2) Cyclic fatigue-crack growth rates in ABC-SiC were only minimally increased, and ΔK_{th} thresholds decreased by ~30%, with increase in temperature from 25 to 1300°C; behavior, however, was independent of frequency. At equivalent stress-intensity levels, crack-growth velocities under cyclic loads were significantly faster than those under static loads at both 25 and 1300°C. The fatigue-crack growth resistance of ABC-SiC was found to be superior to that of commercial SiC (Hexoloy) at all temperatures tested.

(3) Crack profile and fractographic studies showed a predominantly intergranular cracking mode at both low and high temperatures, with crack-tip shielding by grain bridging in the crack wake. Such bridging was degraded under cyclic loads, as evidenced by the extensive wear debris on fatigue fracture surfaces. No evidence of creep cavitation or any form of viscous-ligament bridging by the grain-boundary glassy phase was seen at all temperatures up to 1300°C. The mechanistic sequence of intergranular damage ahead of the crack tip, and the cyclic-loading induced degradation of grain bridging behind the tip, was considered to be essentially unchanged between 25 and 1300°C.

(4) HRTEM observations revealed that the grain boundary film/phase in specimens which underwent high-temperature (≥ 1100°C) fatigue/creep tests or prior annealing treatments were all fully crystallized. Such crystallized grain boundaries are considered to be the primary reason for the impressive mechanical properties of ABC-SiC at elevated temperatures, involving simultaneous enhancements in high-temperature strength, toughness, fatigue and creep resistance.

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