Toughening Behavior in Ceramics and Cermets*

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Abstract

The development of high strength (≥ 1 GPa), high toughness (≥ 10 MPa√m) ceramic systems is being examined using two approaches. In silicon nitride, toughening is achieved by the introduction of large prismatic shaped grains dispersed in a fine grain matrix. For the system examined herein, both the microstructure and the composition must be controlled. A distinctly bimodal distribution of grain diameters combined with controlled yttria to alumina ratio in additives to promote interfacial debonding is required. Using a cermet approach, ductile Ni_3Al-bonded TiC exhibited toughening due to plastic deformation within the Ni_3Al binder phase assisted by interfacial debonding and cleavage of TiC grains. The TiC-Ni_3Al cermets have toughness values equal to those of the WC-Co cermets. Furthermore, the TiC-Ni_3Al cermets exhibit high strengths that are retained in air to temperatures of ~ 1000°C.

I. Introduction

In brittle systems, crack wake bridging processes are an important approach to enhance the fracture resistance. Some successful examples include whisker-reinforced ceramics,[1] self-reinforced silicon nitrides,[2,3] fiber-reinforces ceramics,[4,5] and cermets that combine a ceramic matrix with a ductile metallic or intermetallic phase.[6,7] In many of these, crack front mechanisms (e.g., crack tip deflection,[8] microcracking, deformation of a ductile phase[5,6]) also contribute to the toughening response. In many cases, it is necessary to design ceramics to have both high toughness and high fracture strength which is the case in the present study. Two systems are considered: the first based on the use of microscopic brittle reinforcements–the self-reinforced silicon nitrides,[9] and the second based on an intermetallic bonding phase in carbides (e.g., Ni_3Al-bonded TiC[10]).

II. Experimental

1. Processing

The processing of the self-reinforced silicon nitride ceramics is based on the seeding method to develop a distinct bimodal distribution of grain sizes consisting of large elongated grains in a fine grained matrix.[11] Details of the processing can be found in refs. 9 and 11. The Ni_3Al-bonded TiC cermets were fabricated both by pressureless sintering of mixtures of TiC and Ni_3Al powders and by melt-infiltration sintering as described in refs. 12a and b.

2. Characterization

The microstructures of dense materials were assessed by optical and scanning electron microscopy. R-curve responses were determined using a precracked applied moment double cantilever beam (AMDCB)

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specimen and a test stage that can be mounted on the stage of either an optical microscope or a scanning electron microscope.[9a, 10a]

Fracture strengths were measured in air at 22°C in four point flexure (loading rate of ~ 300 MPa/s), using inner/outer spans of either 20/40 mm or 6.35/19.05 mm. In the case of the cermets, samples were tested over the temperature range of 22°C to 1100°C. Test samples were cut from the as-sintered pieces using electrical discharge machining and then surface finished with diamond abrasive wheels to remove the surface layers. Flexure test bars had 3 x 4 mm cross-sections with diamond (400 grit) ground (grinding direction parallel to tensile axis) surfaces.

III. Results and Discussion

1. Self-Reinforced Silicon Nitride Ceramics

The development of larger elongated grains in the silicon nitride matrix to gain increases in toughness is well known. However, it has now been shown that simply generating larger elongated grains is not a sufficient condition; in fact, the uncontrolled development of large elongated grains can be quite detrimental. As seen in Figure 1, the plateau value of the fracture resistance (or steady-state toughness) can be raised over that of a fine grained equiaxed material with the formation of elongated reinforcing grains. However, only when a distinct bimodal distribution of grain diameters is developed does the toughening effect begin to be optimized, Figure 2.[9] Of equal importance is the fact that the fracture strengths of these same silicon nitrides exhibit a similar trend with the highest strength again achieved in the material with the distinct fraction of larger elongated grains in a very fine grained matrix, Table I.

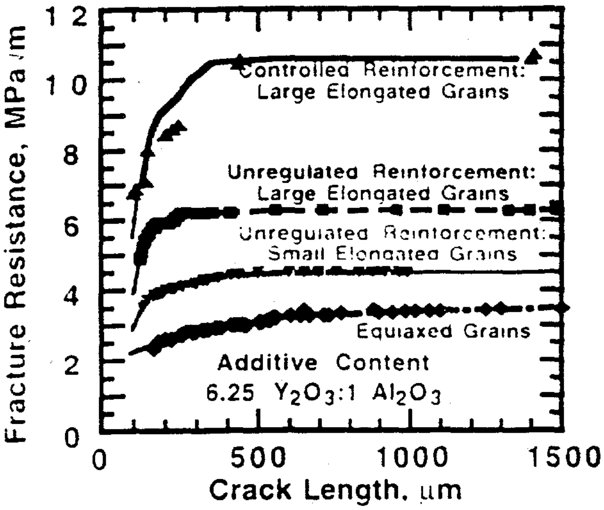

Figure 1. The formation of a controlled microstructure with larger elongated grains dispersed in a fine grained matrix is used in the development of self-reinforced silicon nitrides with high fracture toughness and strength.

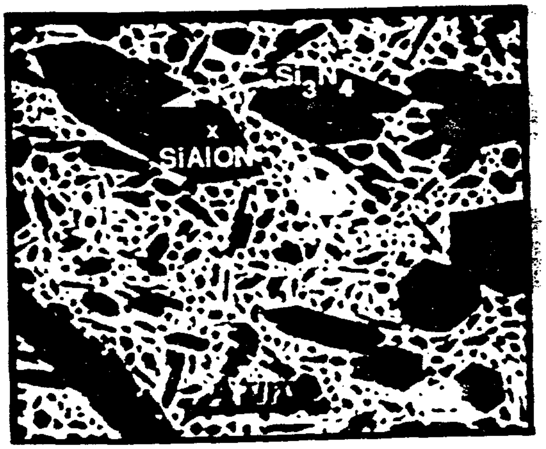

Figure 2. Controlled use of larger β-seeds in a fine matrix powder was used to generate a distinct bimodal microstructure. The growth of an epitaxial SiAlON layer on the larger β-seeds is noted (plasma etched).

The toughening involving crack wake bridging by the elongated grains relies on debonding of the interface between the grains and the amorphous intergranular phase. Earlier studies of whisker-glass model systems with aluminum as one of the glass constituents revealed that such interfacial debonding is influenced by the structure and composition of the interface region.[13] More recent findings indicate that the interfacial debond strength in both the glass-whisker model systems and the self-reinforced silicon nitrides decreases as the Al (and O) content of the epitaxial SiAlON growth layer on the large elongated grains decreases. The Al (and O) contents of both the SiAlON growth layer and the intergranular glassy phase can be controlled by regulating the Y_2O_3:Al_2O_3 ratio in the sintering additives. As a result, both the steady-state toughness values, Figure 3, and the steepness of the initial rising portion of the R-curves increased as the Y:Al additive ratio increased in self-reinforced samples having comparable microstructures. In-situ observations of crack interactions with microstructures revealed increasing amounts of interfacial debonding and intergranular failure associated with the large elongated Si_3N_4 grains as the Y:Al ratio in the additives increased. In the material with the lowest Y:Al additive ratio, interfacial bonding between the large elongated β-Si_3N_4 grains and the intergranular phase was strong, and most of the larger elongated grains failed transgranularly.

Table I. Fracture strength, as well as toughness, is dependent upon control of the microstructure.

Material: Silicon Nitride Using 6.25 wt.% Y_2O_3 - 1 wt.% Al_2O_3 As Sintering Additives	Toughness, MPa√m	Flexure Strength, MPa
Controlled Reinforcement Large reinforcing grains Distinct Bimodal Grain Size Distribution	10.5	1140
Unregulated Reinforcement Larger reinforcing grains Broad Monomodal Grain Size Distribution	6.5	850
Unregulated Reinforcement small reinforcing grains Broad Monomodal Grain Size Distribution	4.5	925
No Reinforcement small equiaxed matrix grains	3.5	660

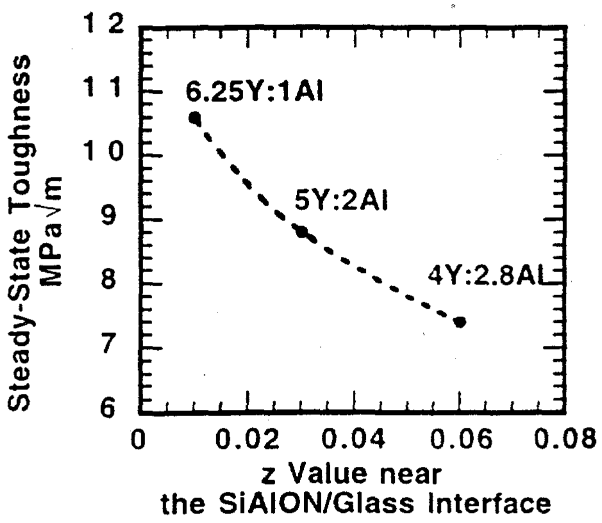

Figure 3. The steady-state (plateau) toughness of self-reinforced silicon nitrides with the same controlled microstructure increases with decrease in z-value (Al and O content) of the epitaxial β-$Si_{6-z}Al_zO_zN_{8-z}$ layer in contact with the amorphous intergranular phase.

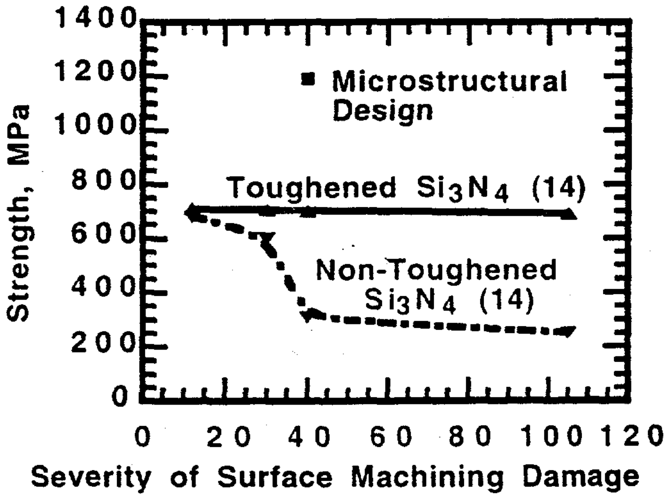

Figure 4. The strengths of silicon nitrides with low toughness degrade with increase in the size of the diamond particles in the surface finishing wheel. On the other hand, toughened materials are damage resistant. Controlled self-reinforced with controlled microstructures can yield high toughness and strength with damage resistance.

What other benefits might be derived by designing self-reinforced silicon nitrides with controlled microstructures? Earlier studies showed that toughened silicon nitride ceramics can exhibit much greater resistance to strength losses induced by increasingly severe surface machining.[14] A comparison of the response of the present high toughness, high strength silicon nitride with the controlled microstructure to the results of the studies by Tajima and Urashima is shown in Figure 4. Clearly, the use of microstructural design that results in the generation of larger elongated grains that are well dispersed in a fine grain matrix offers exciting opportunities for developing damage tolerant ceramics.

2. Ni_3Al–Bonded TiC Cermets

The Ni_3Al-bonded TiC cermets are part of a family of intermetallic-bonded cermets developed recently.[10,12,15] The Ni_3Al binder phase has certain advantages in that it exhibits a reasonably high yield stress that is retained to elevated temperatures, is ductile (~ 50 % plastic deformation prior to failure in polycrystalline samples), and has good oxidation and corrosion resistance.[16,17] In addition, excellent wetting by the aluminides is achieved in a variety of carbides and borides. This and the relatively low melting point of the aluminides (< 1400°C) are quite attractive from the standpoint of component fabrication.

The fracture resistance of the Ni_3Al-bonded TiC cermets rises as the existing short (< 100 µm long) cracks are extended with plateau (steady-state) values approaching 14 MPa√m achieved to date using the AMDCB geometry, Figure 5. From these results, it is not clear what the fracture resistance values would be reduced to for much shorter cracks (e.g., < 20 µm). For those compositions where the binder phase is continuous, the location of the tip of very short cracks (i.e., in the TiC grain or in the ductile Ni_3Al phase) would influence the fracture resistance for near zero crack lengths. In-situ observations of the crack during loading reveal that interface debonding and cleavage of the TiC grains accompany the deformation of the Ni_3Al binder in the wake of the crack, Figure 6.

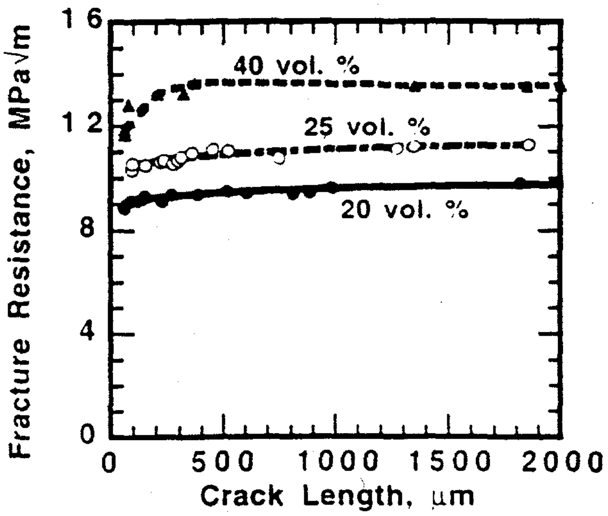

Figure 5. The fracture resistance increases with crack extension in the Ni_3Al-bonded TiC cermets. The toughening effects become greater as the Ni_3Al content is raised.

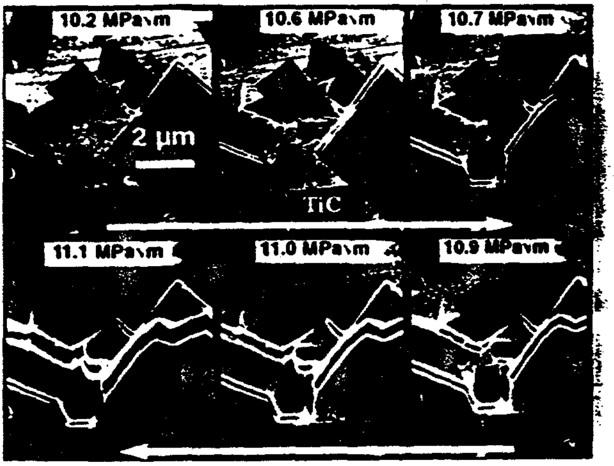

Figure 6. Evidence of deformation of the Ni_3Al phase (white bands) and interface debonding plus TiC fractures is noted as the applied stress intensity increases (TiC-40 vol. % Ni_3Al).

The steady-state toughness of the Ni_3Al-bonded TiC cermets rivals that of commercial Co-bonded WC cermets and exhibits a similar increase in toughness with amount of the ductile binder phase, Figure 7. (For comparison, measurements of the materials shown in Figures 5 and 7 using the chevron notched beam geometry gave toughness values that were twice the values shown here.) Interface debonding and TiC cleavage would reduce the triaxial constraint imposed on the binder phase by the rigid TiC structure and, thus, enhance the deformation of the ductile binder phase. The retention of the triaxial constraint would raise the stress for yielding in the Ni_3Al phase and reduce the toughening contribution of the ductile phase.[6,7]

The potential for these aluminide-bonded cermets in applications over a broad range of temperatures is suggested by the retention of their high flexure strengths to upwards of 1000°C in air, Figure 8. Similar response is noted over a range of Ni_3Al-TiC compositions and with other carbide constituents. Macroscopic plastic deformation is observed in the cermets at temperatures above 850°C in air. This is related to the reduction in the yield point of the Ni_3Al alloys at temperatures above 700°C. In the presence of the embedded TiC grains, global yielding of the ductile Ni_3Al must be restricted in order to retain the high strengths of the cermets to temperatures approaching 1000°C. Thus the temperature limit for the cermets may be a function of composition.

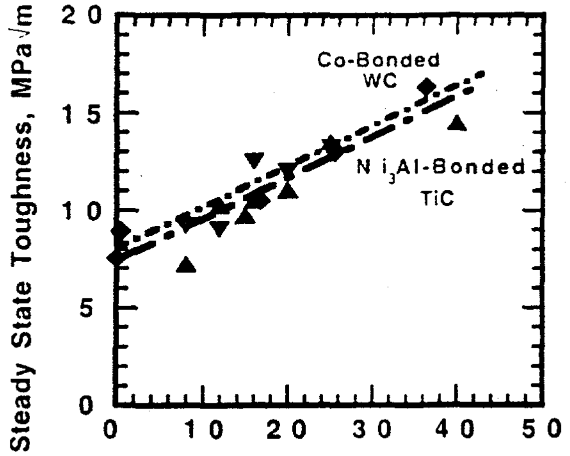

Figure 7. The steady-state toughness of both the Ni_3Al-bonded TiC (filled triangles) and the Co-bonded WC (filled diamonds)are comparable, increasing with ductile binder phase content.

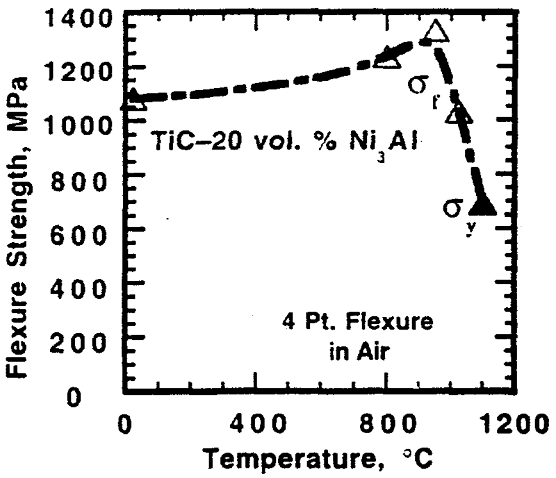

Figure 8. The high fracture strength of the Ni_3Al-bonded TiC cermet is retained to temperatures approaching 1000°C in air.

IV. Summary

Two approaches are being explored in the development of high toughness ceramic systems that also exhibit high strengths. The first employs crack wake bridging by large prismatic shaped grains dispersed in a fine grain matrix as typified by the self-reinforced silicon nitride ceramics. The development of larger elongated grains alone is not sufficient to achieve high strength with high toughness in this system; a distinctly bimodal distribution of grain diameters is required. Debonding of the interface between the larger elongated grains and the amorphous intergranular phase is also necessary to form bridging grains in the crack wake and to initiate deflection of the crack tip. Thus the strength of this interface must be reduced to promote interface debonding over fracture of the large reinforcing grains. With the use of a combination of yttria and alumina additives to promote densification, increasing the Y:Al ratio in the additives is shown to enhance interfacial debonding and, hence, the toughness of the self-reinforced silicon nitrides. Combining microstructural control with compositional tailoring can be used to develop high toughness (> 10 MPa√m) silicon nitride ceramics with high strengths (> 1 GPa) and excellent damage resistance.

The second approach incorporates a ductile aluminide (e.g., Ni_3Al) binder phase for carbides (e.g., TiC) and borides to obtain high toughness (> 10 MPa√m) and strength (> 1 GPa). In this case, debonding of the TiC-Ni_3Al interface and cleavage of the TiC grains accompany plastic deformation within the Ni_3Al binder phase in the crack wake resulting in the increase in toughness. The fracture toughness values of the TiC-Ni_3Al cermets are comparable to those of the WC-Co cermets and increase with ductile binder phase content. In addition, the high strengths of the TiC-Ni_3Al cermets are retained in air to temperatures of ~ 1000°C which suggests the potential for a broad range of applications.

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