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## A NOVEL TECHNIQUE TO GENERATE SHARP CRACKS IN METALLIC/CERAMIC FUNCTIONALLY GRADED MATERIALS BY REVERSE 4-POINT BENDING

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### Introduction

Functionally graded materials (FGMs) containing metallic and ceramic phases offer a viable solution to modern design problems requiring structural materials with high fracture toughness and elevated temperature resistance. Ideally, the ceramic side of the FGM provides thermal and corrosion resistance while the metallic side gives the necessary strength and fracture toughness. Careful grading of the metallic/ceramic interfaces in a continuous or stepwise manner can minimize thermal shock damage to layered FGMs. Cracks originating in the brittle, ceramic side would be prevented from catastrophic propagation through an increase in fracture toughness as the metal content increases. This behavior has been demonstrated by recent experiments with a titanium/titanium monoboride (Ti/TiB) FGM (1).

Fracture toughness testing of metallic/ceramic FGM single-edge-notched-beams (SENB), with the starting notch in the ceramic rich side, requires the initiation of a sharp, short precrack at a specific desired location in a brittle material. Stresses required to initiate crack growth in brittle materials are determined primarily by the distribution, orientation and size of existing flaws, as shown by the classic studies of Griffith (2). A review of fracture toughness testing of brittle materials by Sakai and Bradt (3) notes that creating a starting crack of the proper sharpness and length is critical for accurate fracture toughness measurements. The new standard for fracture toughness testing of advanced ceramic materials describes the requirement of a sharp, well-characterized precrack as well as the difficulty in obtaining one (4). Use of machined notches or blunt cracks can lead to fracture toughness values that are much larger than those obtained from specimens with sharp precracks. Sharp precrack initiation by the bridge-indentation method was devised by Nunomura and Jitsukawa (5). It was used on hard metals by Warren and Johanneson (6) and on ceramic materials by Nose and Fujii (7). The single edge precracked beam method (SEPB), based on standard 4-point bending with the notch tip in tension, can lead to longer than desired popped-in precracks in multi-layered FGMs (1) even when displacement is limited by a rigid testing machine. Long precracks can also give values of fracture toughness that are too high due to crack wake effects. The method of axial compression precracking developed by Ewart and Suresh (8) for ceramic materials can lead to extensive microstructural damage in metallic/ceramic FGMs and can give values of fracture toughness which are too low (9). The goals of our current

research are to demonstrate rising crack resistance curve (R-curve) behavior in a layered metallic/ceramic FGM and accurately measure  $J_{Ic}$  values for this material. To accomplish these goals, it was necessary to develop a method of generating a short, sharp precrack at the root of a machined notch in the brittle side of the FGM.

### **Experimental Procedures**

A functionally graded Ti/TiB material was prepared by Cercom Incorporated, Vista, CA, using a commercially pure (CP) titanium plate and six tape cast mixtures of titanium (Ti) and titanium diboride (TiB<sub>2</sub>) powders. The assembled green material was placed in a graphite die in an induction furnace and heated to 500°C in vacuum to remove organic tape materials. The material was then heated further to 1100°C to remove surface adsorbed gases from the TiB<sub>2</sub> and then heated to 1305°C under a pressure of 13.8 MPa. Subsequent x-ray diffraction revealed that reactions between the Ti and TiB<sub>2</sub> during the 36 hour pressing and cooling time resulted in the formation of titanium monoboride (TiB) with less than one percent of residual TiB<sub>2</sub>. The compositions of the layers in the FGM varied from 15%Ti/85%TiB on the brittle side to pure Ti on the ductile side. The intermediate compositions of the FGM were chosen to reduce the thermal residual stress during cooling from the sintering temperature as described by Gooch et al. (10). Single-edged-notched bend specimens (SENB) of the FGM were cut using electro-discharge machining (EDM) with the machined notch on the side containing the highest concentration of TiB. The notch was oriented for crack propagation through the layers toward the CP-Ti. The specimens were machined to a width of 14.73 mm, a thickness of 7.37 mm and a length of 82.55 mm, with a notch 5.08 mm deep (initial  $a/W = 0.345$ ) with integral clip gage knife-edges in accordance with ASTM E1820-96 (11).

### **Precracking by ASTM Tension Fatigue**

Precrack initiation was attempted using a standard tension fatigue method by loading the beams in three-point bending, specified by ASTM methods (11) for metallic specimens, with a starting load of 490 N, a compressive load ratio of 10 (minimum/maximum load) and a span of 63.5 mm. Crack growth was monitored by crack mouth opening displacement (CMOD) compliance and no growth was detected after 75,000 cycles. The load was increased to 540 N and application of that load resulted in the pop-in of a compliance measured crack 6.29 mm in length, a crack length to width ratio ( $a/W$ ) of 0.427. This crack was longer than the desired starting crack length of 5.28 mm ( $a/W = 0.358$ ) and prevented the fracture toughness testing of two layers in the FGM specimen. This method of precrack initiation proved to be uncontrollable, as another specimen fatigue cycled using the same stress intensity cracked to a length of 14.65 mm ( $a/W = 0.995$ ).

### **Fracture Testing without Precracking**

The difficulty in initiating a short, sharp precrack led to an attempt at J-R testing a notched specimen without any precrack. The test crack initiated with pop-in to 7.53 mm ( $a/W = 0.511$ ) measured optically with a traveling microscope. This allowed the measurement of the fracture toughness of only the final 85%Ti/15%TiB and the pure titanium layers. The test was repeated with a resulting popped-in crack 7.85 mm ( $a/W = 0.533$ ) in length, again much longer than the desired crack length and allowing fracture toughness measurements of only a few layers of the FGM.

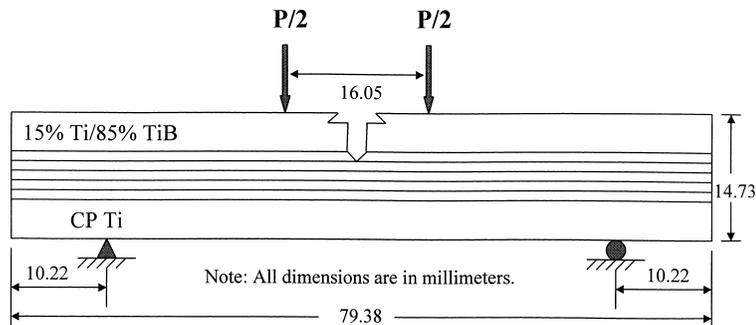


Figure 1. Seven-layer Ti/TiB reverse 4-point bending specimen. The composition for each layer is (1) 15/85, (2) 21/79, (3) 38/62, (4) 53/47, (5) 68/32, (6) 85/15, and (7) 100/0 where the number in parentheses indicates the layer number.

### Pre-Cracking by Axial Compression

Pre-crack initiation using the uniform axial compression method of Ewart and Suresh (8) was attempted on SENB specimens 50mm in length. Shorter specimens and a hemi-spherical fixture were necessary to prevent buckling and shear failure of the beams in axial compression. The stress required to initiate precracking at the machine notch was found to be 212 MPa, larger than the ultimate tensile strength of the CP titanium layer (179 MPa). Short cracks were developed in axial compression at the machined notch, predominantly along the direction of the loading axis rather than in the desired perpendicular direction. Cracking was also developed in the Ti layer due to excessive plastic deformation and residual tensile stresses. Experiments done with smaller loads failed to produce any precracks. The average elastic modulus calculated by beam compliance based on load/CMOD measurements was 145 GPa prior to application of axial compression loads and was 96 GPa after precracking. The large decrease in modulus of the axial compression precracked specimens indicates extensive microstructural damage (cracking and debonding of the TiB particles from the matrix in the titanium rich layers) which was observed by scanning electron microscopy (9).

### The Novel Technique: Reverse 4-Point Bending

To improve the quality of the pre-crack, the SENB specimens were cyclically loaded in reverse four-point bending using a 6,800 N load, resulting in initiation of a controlled, short, sharp crack at the machine notch. The loading configuration of the specimens is shown in Figure 1.

The load was applied with a compressive load ratio ( $R = 10$ ) at a cyclic frequency of 5Hz. The load was chosen to develop the precrack in approximately 5,000 cycles. The target crack length was 0.2 mm from the starting notch which gave the desired  $a/W = 0.358$  for the fracture experiments. The starting notch and resulting pre-crack developed by reverse four-point bending fatigue are shown by optical micrographs, Figures 2(a) and 2(b), respectively.

Figure 3(a) is a scanning electron micrograph (SEM) of the pre-crack fracture surface showing crack fronts resulting from successive large monotonic load applications. The crack is generated by a compressive shear overload at the machined notch resulting in a crack straight through the thickness of the beam, parallel to the machined notch. Relatively large, monotonic loads may result in larger than desirable crack growth increments. When the correct cyclic loads are applied in reverse 4-point bending, the precrack is short and sharp as shown in Figure 3(b).

Average modulus values, measured before and after the precracking were 145 GPa, indicating that the overall microstructure of the FGM was undamaged by reverse 4-point bending. After heat tinting

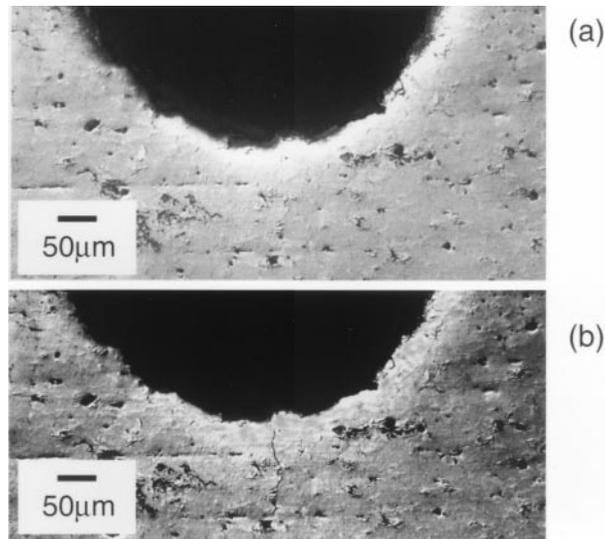


Figure 2. Face of FGM SENB specimen (a) before precracking and (b) precracked in reverse 4-point bending fatigue.

at 400°C, the specimens were cooled overnight to room temperature and fracture tested in three-point bending according to ASTM E1820–96 (11).

### Results and Discussion

Precrack generation by cyclic, tensile fatigue loading of metal specimens provides a convenient method of crack initiation in ductile materials. Application of that technique to brittle ceramic materials leads to rapid, uncontrolled crack growth at crack initiation and failure of the test specimens. This is due to the large stresses required for crack initiation and the very small stresses required to drive a sharp crack through the brittle material. In FGMs, tensile fatigue loading causes cracks to pop-in. However, the thin layers in multi-layered FGMs require a more precise location of the precrack than that provided by

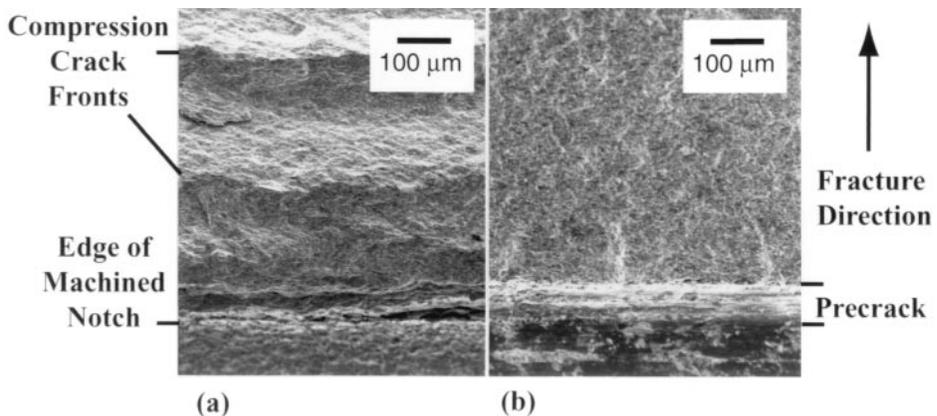


Figure 3. Reverse 4-point bending precracks generated by (a) large monotonic loads and (b) fatigue.

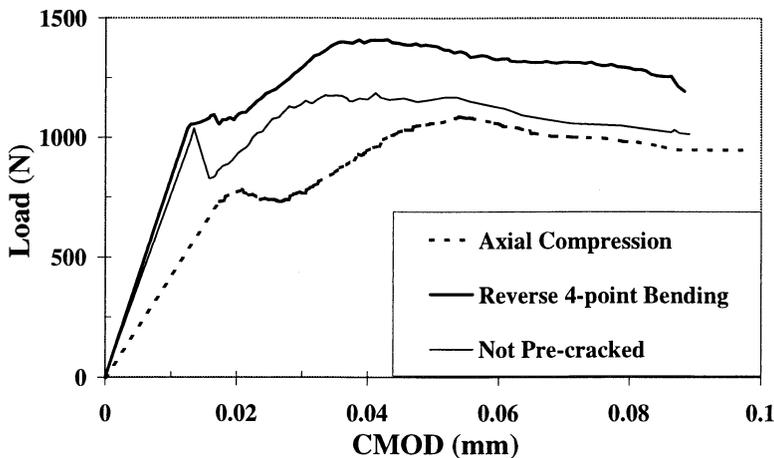


Figure 4. Load vs. CMOD plot for Ti/TiB J-R tests.

pop-in methods. Precracks at locations with small  $a/W$  ratios are also desired in FGMs, and these are difficult to achieve with pop-in methods.

Axial compression of ceramic materials leads to precrack initiation by residual tensile stresses during compressive unloading according to Suresh and Brockenbrough (12). However, the layered metal/ceramic FGM has a decreasing modulus with increasing metal content. Application of uniform axial compressive stresses to the FGM beams can cause cracking at the machined notch and plastic deformation of the metal-rich matrix layers. This generates cracks between the matrix and the ceramic particles in those layers. The resulting damaged microstructure provides for decreased crack propagation resistance (lower fracture toughness). The novel reverse 4-point bending fatigue method overcomes the limitations of the above mentioned methods by placing the notch tip region in compression while maintaining tensile stresses in the metal-rich layer. The results indicate that the mechanism of crack initiation involves residual tensile stresses at the notch tip, similar to the processes by which cracks are nucleated in ceramics under uniform compression loading (8,12,13).

The load vs. CMOD data for J-R tests of the beams precracked by uniform axial compression, reverse 4-point bending and an uncracked specimen are shown in Figure 4. The beam without a precrack exhibited rapid, unstable crack growth when loads were increased to the point of crack initiation and gave no useful data for the FGM layers near the machined notch.

The specimen precracked by uniform axial compression showed stable crack growth with no pop-in during the J-R test indicating that a sharp crack was initiated during compressive loading. The load values and the resultant J values, however, were only 70% of those obtained by the specimen that was precracked by reverse 4-point bending. The lower values for that specimen are due to extensive microcracking during application of the axial compressive loads. The J vs. crack extension result for the reverse 4-point bend precracked specimen is shown in Figure 5 demonstrating a rising R-curve behavior as the metal content increases in successive layers of the FGM. The results of these J-R tests, along with finite element modeling of crack growth, will be described in greater detail in a subsequent paper (14).

### Conclusions

Accurate measurement of fracture toughness in brittle and ductile materials requires a sharp pre-crack at the machined notch. Cyclic tensile fatigue methods work well on ductile, but not brittle materials.

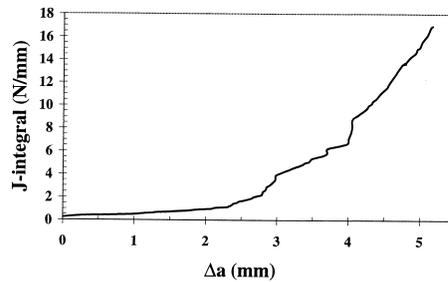


Figure 5. J vs. crack extension for Ti/TiB J-R tests.

Uniform axial-compression methods developed for ceramic materials introduce microstructural cracks leading to lower than expected fracture toughness. Pop-in methods usually develop longer precracks than desired for FGM beams and are difficult to control. Thus, precracking methods applicable to homogeneous metal and ceramic composite materials are not appropriate for FGMs. By using the novel reverse 4-point bending method, a controlled, sharp, short precrack can be developed at the machined notch in metallic/ceramic FGM beams allowing accurate J-R measurements. This precracking method can be used in fatigue or monotonic loading.

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