

Fracture of composite alumina/yttria-stabilized zirconia joints

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Abstract

Four-point bend tests have been performed on samples consisting of yttria-stabilized zirconia containing 0–80% alumina joined by plastic deformation to the same or different composition. The fracture strength of joints between the same composition was equal to the strength of the monolithic material. Fracture of joints made between different compositions occurred at the position of maximum tensile residual stress, as determined by finite-element analysis, not at the interface. Measured strengths were in accord with fracture mechanics and the calculated residual stresses.

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1. Introduction

Joining by plastic deformation has been applied to various advanced ceramics (yttria-stabilized zirconia (YSZ)/alumina composites,^{1–3} mullite,⁴ SiC and TiC whiskers in a zirconia-toughened alumina (ZTA) matrix,⁵ and La_{0.85}Sr_{0.15}MnO₃⁶). Techniques have been developed to reduce sample preparation procedures and the temperature at which joining takes place, amongst them a spray application technique² or use of nanocrystalline powders or dense interlayers^{2,7} stand out. Use of plastic deformation has many advantages over other joining techniques, such as brazing or diffusional bonding (for reviews, see Refs. 8–11), in terms of applicability or mechanical performance, especially at high temperatures at which a metallic interlayer, as, for example, one used in brazing, will exhibit diminishment of its mechanical or thermal properties.

Although plastic deformation has few, if any, serious deficiencies when joining similar materials,^{1,6} some issues remain to be addressed when dissimilar materials are to be

joined. In this case, the situation is quite different because of presence of residual stresses generated upon cooling if materials have different thermal-expansion coefficients (CTE). The thermal residual-stress distributions have been characterized in YSZ/alumina composites by finite-element analysis (FEA) simulation, and later contrasted with experimental observations from Vickers indentation measurements.¹

In this work, flexural strengths of joined YSZ/alumina ceramics have been evaluated. Failure mechanisms when joining dissimilar YSZ/alumina ceramics have also been identified. Experimental observations are compared to FEA simulations. Fracture mechanics principles, in conjunction with fractographic analysis, were used to explain strengths of joined ceramics in the presence of residual stresses.

2. Experimental details

Dense YSZ/alumina samples of various compositions (YSZ volume fractions ranging from 20 to 80%) were prepared by wet mixing commercially available alumina and 3 mol% tetragonal zirconia polycrystals (3Y-TZP) powders. Subsequently, the dried powder mixtures were cold pressed into cylindrical forms and sintered at temperatures ranging

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from 1450 to 1650 °C. Specific details related to sample fabrication are described elsewhere.¹ The resultant pieces were cylinders of approximate dimensions 1 cm diameter and 1 cm height. Joining experiments were carried out on as-sintered samples (without any further surface treatment) by compressing them together at constant crosshead speed in an Instron Model 1125 (Canton, MA) equipped with a high-temperature furnace. This joining technique has been fully described elsewhere.^{1–3} Typically, temperatures of 1250–1350 °C and strain rates of $\approx 10^{-5} \text{ s}^{-1}$ were used to ensure plastic flow during the joining process.¹

Interfaces of the joined samples were evaluated by scanning electron microscopy (SEM). Samples for metallographic observations were polished with 3 μm diamond paste and then thermally etched by annealing in air for 0.5 h at 1200 °C, which is lower than the temperatures used during joining in order to prevent microstructural evolution.

As-prepared and joined samples were cut into bars of $\approx 2 \text{ mm} \times 2 \text{ mm} \times 15 \text{ mm}$ for flexure testing. Samples were carefully polished to 1 μm diamond paste and the edges were beveled to avoid edge effects during testing. Four-point bending tests were carried out at constant crosshead speed in an Instron Model 4505 (Canton, MA), with a stainless steel four-point bending fixture. The inner load span was 9.5 mm and the outer load span was 14 mm; the loading rate was 1.3 mm/min. Strength was calculated from the maximum load at failure. At least four specimens were tested per sample type, with the exception of the zirconia-toughened 60 vol.% Al_2O_3 (ZT60A) joined to zirconia-toughened 40 vol.% Al_2O_3 (ZT40A) for which three samples were tested.

FEA was carried out with use of commercially available software ABAQUS 6.4 (Providence, RI). Residual stresses in the joined products were simulated by considering a rigid stress-free interface that is formed at high temperature, and during subsequent cooling (taken as 1200 °C to room temperature) stresses developed as a result of the mismatch in CTE values. One-quarter of flexure beam was modeled as shown in Fig. 1. Symmetry was used along 2–3 and 1–3 sides, as shown

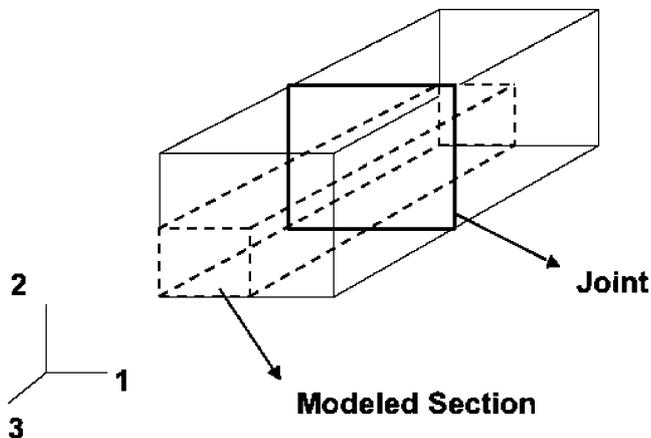


Fig. 1. Schematic of the joined beam sample showing the one-quarter section used in finite-element modeling.

in Fig. 1. The model comprised of 35,721 nodes and 32,000 hexahedral elements. The values of the elastic and thermal properties of the materials used in the simulation were determined experimentally and have been reported elsewhere.¹

To identify the failure-causing flaws in the ZT60A/ZT40A joined samples, fractographic analysis was conducted on the fracture surface of the flexure-tested samples with a Hitachi Model S-4700-II scanning electron microscope (Tokyo, Japan).

3. Results and discussion

3.1. Microstructure of the joined samples

Fig. 2 shows a typical interface for a ZT20A/ZT80A joint. The joint is well bonded with no residual porosity. Typical grains sizes for alumina and zirconia were 0.7 and 0.3 μm , respectively. As shown in the figure, grains of the two samples interpenetrate to form the pore-free bond. Similar dense interfaces were obtained for joints made with other YSZ/ Al_2O_3 composites. Thus, it is possible to join both similar and dissimilar YSZ/ Al_2O_3 composites using the plastic deformation technique.

3.2. Flexure testing of composite constituents and joints of similar materials

It was observed in previous work that when plastic deformation was applied to join similar materials, the interface was optically and electrically indistinguishable from the host ceramics.⁶ However, the mechanical response of the joined parts remains to be assessed. Flexural testing is probably the ideal technique to use since strength is sensitive to the presence of defects or stress concentration in the sample, and the specimens are too small for reliable tensile testing. Residual porosity due to incomplete joining should be revealed by frac-

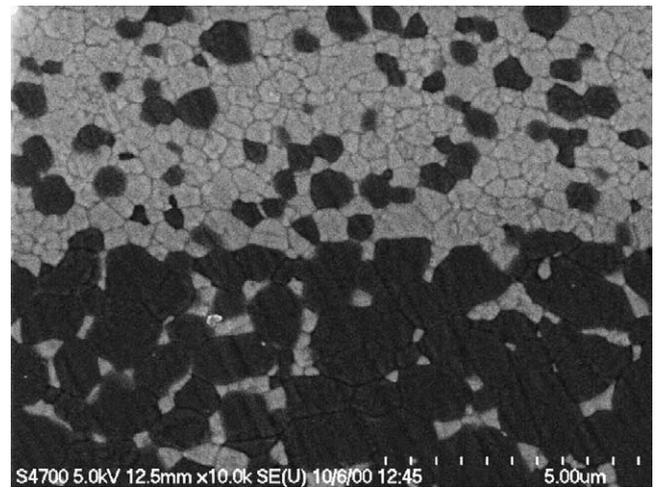


Fig. 2. SEM photomicrograph of YSZ/20 vol.% Al_2O_3 (ZT20A)–YSZ/80 vol.% Al_2O_3 (ZT80A) joint; Al_2O_3 is the dark phase.

Table 1
Flexure test data

Material	Joint	Flexural strength (MPa)	Fracture–interface distance (μm)
Al_2O_3 sintered ¹³		300	–
ZT80A		560 ± 70	–
ZT60A		580 ± 80	–
ZT50A		650 ± 100	–
ZT20A		1020 ± 150	–
YSZ sintered ¹⁴		1030	–
	ZT50A/ZT50A	620 ± 100	1949 ± 1544

turing along the interface, accompanied by a lower value of strength in the joined part when compared to the monolithic strength value.

Table 1 shows the measured strengths of monolithic alumina, YSZ, and their composites. Strength for monolithic samples versus alumina content follows a well-established trend.¹² The strengths varied from 300 MPa for alumina to 1030 MPa for fully sintered YSZ.^{13,14} As expected, the strength of zirconia-toughened alumina decreases monotonically with alumina additions.

Four-point bending tests were also carried out in joined zirconia-toughened alumina (50% volume fraction of each, ZT50A) samples. Joined and monolithic samples did not exhibit a significant difference in strength. The strength of the joined sample was 620 ± 100 MPa, within the experimental scatter for the monolithic material (Table 1), whose strength was 650 ± 100 MPa. Moreover, as indicated in Table 1, the failure locations for these joined ZT50A samples occurred on the average of 1949 ± 1544 μm away from the interface. This is further evidence of the efficacy of this technique for joining similar materials. The fact that the strength of the joined bodies and the monolithic samples was the same provides definitive evidence that the joining by the plastic flow technique is viable and bodes well for using the deformation joining technique for real-world applications.

3.3. Finite-element analysis

FEA was conducted to understand the role of residual stresses generated during joining of dissimilar materials on the mechanical behaviour of joined samples. The distribution of normal residual stresses generated when cooling from the joining temperature for ZT60A joined to ZT20A is shown in Fig. 3.^{1,15} A high tensile stress (~ 250 MPa), perpendicular to the joint interface, develops in the material with lower thermal-expansion coefficient. In our case, this material corresponds to the composite with lower YSZ volume fraction (ZT60A), due to the fact that the thermal-expansion coefficient of the alumina is considerably lower than that of YSZ.¹ Also, the location of peak residual tensile stress normal to the interface is 150–200 μm away from the physical interface in the ZT60A composite. The peak stresses are a finite distance away from the interface because of the discontinuity of the stresses at the interface. Moreover, normal stresses parallel

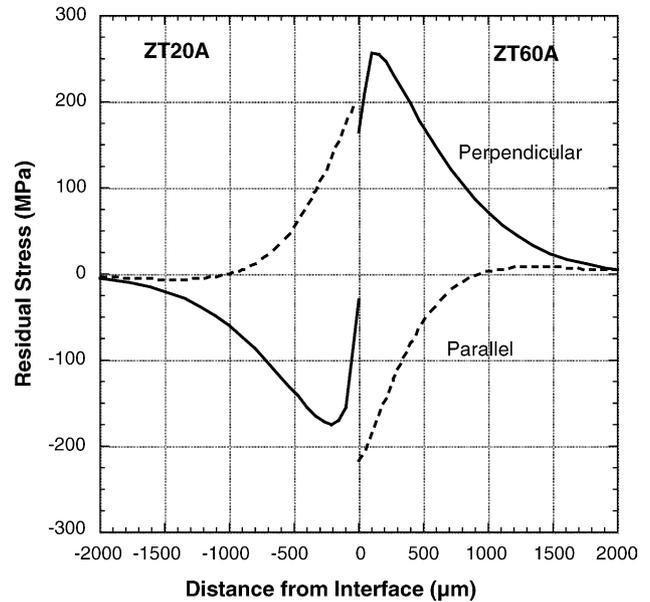


Fig. 3. Distribution of residual normal stresses per FEA simulation for a ZT20A/ZT60A joint.

to the interface are compressive in the material with lower CTE (ZT60A) and tensile for the material with higher CTE (ZT20A). This high tensile residual stress perpendicular to the interface has an important effect on mechanical performance of the joined dissimilar materials during flexure tests. Similar simulations were also conducted for ZT60A/ZT40A and ZT60A/ZT0A joined samples.

In addition to the normal stresses, shear stresses are also generated and their variation for ZT60A/ZT0A joined samples determined by FEA is shown in Fig. 4. Shear stresses are developed close to the interface; they fall off rapidly within short distance from the interface. The magnitude of shear

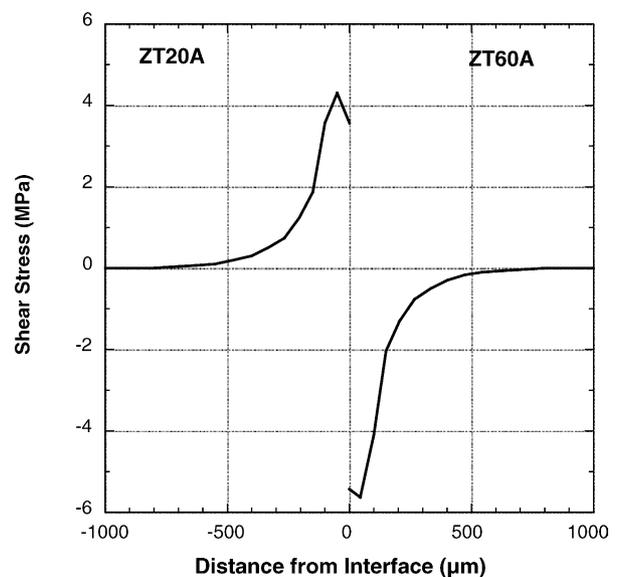


Fig. 4. Distribution of residual surface shear stresses per FEA simulation for a ZT20A/ZT60A joint.

Table 2
Flexure test data for dissimilar joined materials

Joint	Flexural strength (MPa)	Fracture–interface distance (μm)
ZT60A/ZT40A	364 ± 134	335 ± 148
ZT60A/ZT20A	500 ± 50	692 ± 108
ZT60A/ZT0A	440 ± 80	381 ± 98

stresses is small compared to the normal tensile stresses. Thus, it is expected that failure will be controlled by the normal tensile stresses of these joined samples.

3.4. Flexure testing of dissimilar joined samples

In order to study the influence on strength of joined parts with the stress distribution arising from CTE mismatch, a second series of joining experiments was performed. These experiments consisted in bonding parts with various alumina fractions (ranging from 0 to 40 vol.%) to another part of a fixed composition (60 vol.% alumina). Joined samples of ZT60A/ZT40A, ZT60A/ZT20A, and ZT60A/ZT0A were flexure tested and the results are presented in Table 2, along with the failure locations relative to the joint interface. Several key inferences can be made from the results of dissimilar joined samples. First, all samples failed in the ZT60A portion, the lower-strength material, and with tensile stresses normal to the interface. Second, strength for joined samples reduced as the difference in composition between the parts joined or as the CTE mismatch increased for ZT60A joined to ZT20A and ZT0A. For the experiment in which there was the largest difference in composition (YSZ joined to ZT60A), flexure strength decreased by 25% when compared to the strength of monolithic ZT60A. However, strengths for ZT60A/ZT40A joined samples showed lower mean strength and large scatter as compared to ZT60A/ZT20A and ZT60A/ZT0A joined samples. Reasons for this will be discussed further. Third, for all the dissimilar joints, failure was found to be away from the interface (as shown in Fig. 5), again confirming the

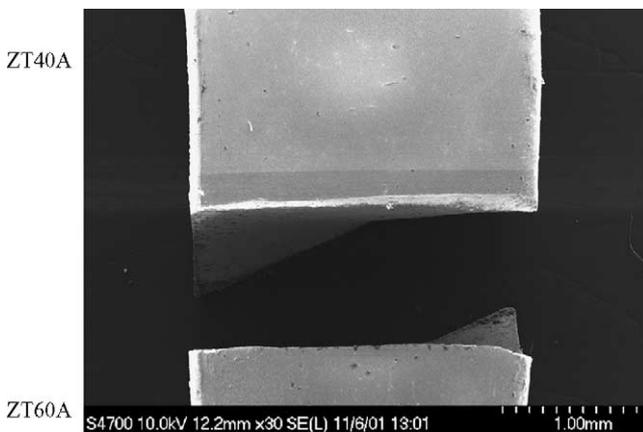


Fig. 5. Fractured ZT40A/ZT60A joint in a four-point bending test. The sample is broken in the portion with higher alumina content, while the interface (seen as the dark line in the upper part) retained its integrity.

strong interface and efficacy of the joining process for materials with dissimilar compositions and coefficient of thermal expansions.

The decreased strength of ZT60A when joined to compositions with lower volume fraction of alumina as compared to the strength of ZT60A can be attributed to the presence of tensile residual stresses, location, and the size of failure causing flaw. It is expected that the reduction in strength of the joined sample will depend on the region of near-maximum tensile residual stresses. In this regard, the distance from the interface at which fracture occurred is especially revealing. For ZT60A/ZT20A joint sample, the average failure location is about $700 \mu\text{m}$ away from the interface (Table 2) in the ZT60A section. This corresponds to about 100 MPa tensile residual stress as per FEA simulations as shown in Fig. 3. By simple superposition of the residual stress intensities on the applied stress intensity during flexure tests, the failure strength for ZT60A should be reduced by 100 MPa. This is in agreement with the reduction of approximately 80 MPa in flexural strength of ZT60A when joined to ZT20A. Simi-

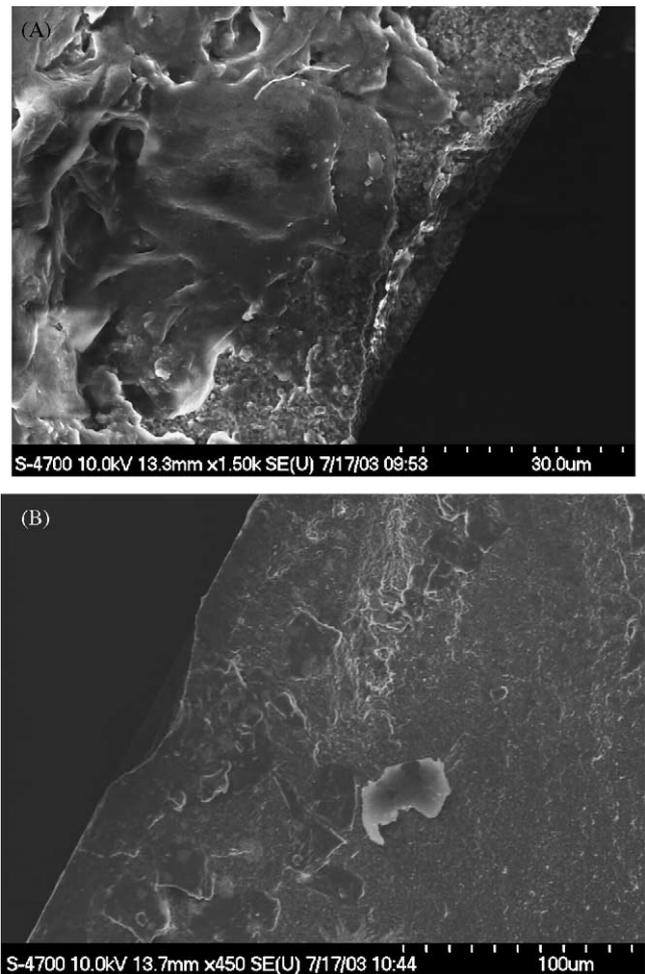


Fig. 6. SEM photomicrographs showing large flaws on the tensile surface of ZT60A section from two low-strength fractured ZT60A/ZT40A joined samples: (A) strength = 360 MPa; (B) strength = 201 MPa.

lar correlations between residual tensile stresses and flexural strength for ZT60A/ZT0A have been made.

Strengths of ZT60A/ZT40A were somewhat lower and were 201, 360 and 530 MPa for the three joined samples tested. Close inspections of the fracture sites were made for all samples. The lower strengths of 360 and 201 MPa observed for the two samples are believed to be due to the presence of larger flaws. To confirm this, fractography was conducted on the two low-strength samples. SEM photomicrographs (Fig. 6) reveals surface damage on the tensile surfaces in the two low-strength samples. It is speculated that this damage was introduced during the sample preparation steps or handling. Typical lengths of this damage were 70–90 μm . Using a nominal crack length of 80 μm , apparent toughness of 3.6 MPa $\text{m}^{1/2}$ for ZT60A, and fundamental fracture mechanics,^{1,16} the fracture strength was calculated to be approximately 350 MPa. This calculated fracture strength is consistent with the measured strength for the low strength ZT60A/ZT40A sample. Thus, in addition to the residual stresses, size and distribution of failure causing flaws in the ceramic materials will control the strength of the joint. This also explains why failure location in the joined samples does not necessarily coincide with the peak tensile residual stresses.

4. Conclusions

Strength of yttria-stabilized zirconia/alumina parts joined by high-temperature plastic deformation was measured by flexure tests. Samples with varying amounts of alumina (0–80%) were used in this work. Joints fabricated with same composition showed strength similar to that of base material. This observation demonstrates the efficacy of the joint and validates the joining technique. For joints made from dissimilar compositions, fracture occurred at locations away from the joint interface. This failure location has been shown to be consistent with the peak residual stresses calculated by finite-element analysis. Using fracture mechanics approach and the calculated residual stresses, strength of joined samples were predicted and found to be consistent with experimental observations. Presence of non-processing related flaws can also affect the strength of joined samples.

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