

D. Joining of Advanced Materials by Plastic Deformation

Principal Investigator: J. L. Routbort

Argonne National Laboratory

9700 S. Cass Avenue, Argonne, IL 60439-4838

630-252-5065, routbort@anl.gov

Co-investigators: F. Gutierrez-Mora, D. Singh, N. Chen, and K. C. Goretta

Argonne National Laboratory

9700 S. Cass Avenue, Argonne, IL 60439-4838

Technology Development Area Specialist: Sidney Diamond

202-586-8032, fax: (202) 586-1600; sid.diamond@ee.doe.gov

Field Technical Manager: Philip S. Sklad

630-252-506; routbort@anl.gov

(865) 574-5069; fax: (865) 576-4963; e-mail: skladps@ornl.gov

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Objective

- Join advanced materials such as ceramics, cermets, intermetallics, and composites by plastic deformation.
- Characterize the interfaces.

Approach

- Apply a modest compressive load to two pieces of similar or dissimilar materials that have had little surface preparation in the temperature region where the materials are known to deform by grain-boundary sliding.
- Examine interfaces by scanning electron microscopy (SEM).
- Measure residual stresses after joining dissimilar materials and compare the measurements with those from finite-element analysis (FEA).
- Measure the strength of the interface by 4-point bend tests.

Accomplishments

- Made strong, pore-free joints with various ceramics, cermets, intermetallics, and composites with and without various interlayers.
- Achieved joint strength equal to that of the monolithic.

Future Direction

- Join intermetallics to ceramics; join biomaterials.
- Use functionally graded materials to distribute and reduce interfacial stress concentrations.

- Measure in-situ grain rotation during deformation or joining using the Advanced Photon Source (APS), pending funding.

Introduction

Joining by plastic deformation has been successfully applied in this program to various advanced ceramics (yttria-stabilized zirconia (YSZ) –alumina composites, mullite, silicon carbide and titanium carbide whiskers in a zirconia-toughened alumina (ZTA) matrix, metal-matrix composites, and even an electronic ceramic, $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_3$ (1–5). Techniques have been developed to minimize sample preparation procedures and minimize the temperature at which the joining takes place. Among them, a spray application technique² and use of nanocrystalline powders or dense interlayers stand out.^{2,6} A patent application is pending. More recently, we have formed pore-free joints in Ni_3Al .

Although it is clear that joining by plastic deformation has few if any serious deficiencies when used to join similar materials, some issues remain to be addressed when dissimilar materials are to be joined. In those cases, when the materials joined have different thermal expansion coefficients at the high temperatures required for plastic deformation, residual stress are generated upon cooling. The thermal residual-stress distribution has been characterized in YSZ–alumina composites by FEA simulation and later compared with experimental observation from Vickers indentation measurements.¹

During the last quarter of FY 2003, we performed 4-point bend tests on joined pieces of the same and different compositions of YSZ–alumina ceramics. Fracture mechanics principles, in conjunction with fractographic analysis, are used to explain the strengths of joined ceramics in the presence of residual stresses. This report will concentrate on those results.

Experimental Details

Dense YSZ–alumina samples of various compositions (YSZ volume fractions ranging from 20 to 100%) were prepared.¹ The resultant pieces were cylinders ≈ 1 cm in diameter and 1 cm in height. As-sintered samples without any further surface treatment were compressed together at constant crosshead speed in an Instron Model 1125 equipped with a high-temperature furnace. Experimental conditions were those where plastic flow is known to occur in ZTA, temperatures of 1250–1350°C and strain rates of $\approx 10^{-5}$ /s.

As-prepared and as-joined samples were cut into bars of $\approx 2 \times 2 \times 15$ mm for flexure testing. Four-point bending tests were conducted on samples polished to 1 μm at a constant crosshead speed of 1.3 mm/min using an inner load span of 9.5 mm and an outer load span of 14 mm. Strength was calculated from the maximum load at failure, and at least four specimens were tested per sample type.

FEA was carried out using the commercially available software ANSYS. Stresses in the joined products were simulated by considering a rigid, stress-free interface that is formed at high temperature and during subsequent cooling. Residual stresses developed as a result of the different thermal expansion coefficients. The values of the elastic and thermal properties were measured experimentally.¹

Results and Discussion

Table 1 shows the strengths of monolithic alumina, YSZ, and their composites. Strength for monolithic samples vs. alumina content follows a well-established trend. The strengths varied from 300 MPa for alumina to 1030 MPa for fully sintered YSZ. The strength of ZTA initially

Table 1. Flexure data for monolithic ceramics. Al₂O₃ and ZTA from literature

Material	Strength (MPa)
Al ₂ O ₃	300
ZT80A	560 ± 70
ZT60A	580 ± 80
ZT50A	650 ± 100
ZT20A	1020 ± 150
YSZ	1030

increases and reaches a maximum for 20 vol % alumina and subsequently decreases with further alumina additions. Four-point bending tests were also carried out on ZTA (50 vol % each of zirconium and aluminum, designated ZT50A). Table 2 presents flexure test data for joined samples. Joined and monolithic samples did not exhibit a significant difference in strength. The strength of the joined samples was 620 ± 100 MPa, within the experimental value for the monolithic material. whose strength was 650 ± 100 MPa.

Table 2. Flexure data for joined ceramics

Joint	Strength (MPa)	Distance—fracture to interface (µm)
ZT50A/ZT50A	620 ± 100	
ZT60A/ZT40A	530	195
	360	270
	201	540
AT60A/ZT40A	500 ± 50	
ZT60A/ZT0A	440 ± 80	

The fact that the strength of the joined bodies and the monolithic samples was the same (compare ZT50A from Table 1 with ZT50A/ZT50A from Table 2) provides definitive evidence that joining by the plastic flow technique is viable and bodes very well for using the deformation joining technique for real-world applications.

Results were quite different when dissimilar materials were joined. However, as shown in Figure 1, a fracture occurred not at the interface but at some distance away from it, as indicated in Table 1. This is the result of

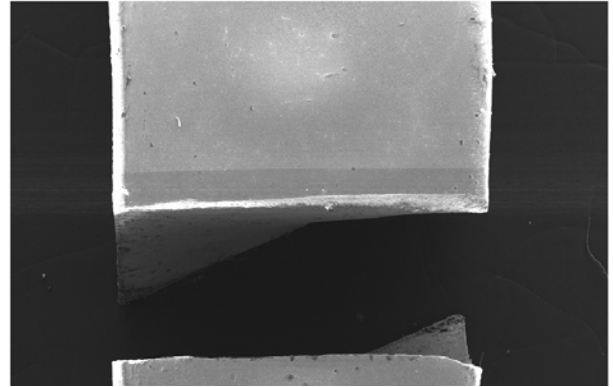


Figure 1. Fracture between ZT40A (upper) and ZT60A (lower). The fracture occurred in the ZT60A piece, not at the interface.

the residual stresses due to the differences in thermal expansion coefficients of the two materials. The distribution of stresses was calculated by FEA¹ and is shown in Figure 2.

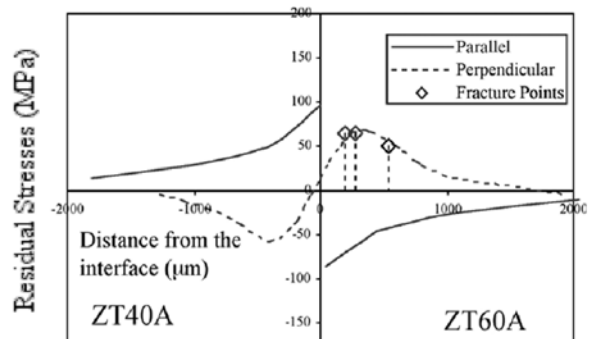


Figure 2. Plot showing the distribution of residual stresses as simulated by FEA for a ZT40A/ZT60A joint. Experimental data correspond to the distance from the interface at which the fracture took place in the flexure test.

A high tensile stress developed in the materials with the lower thermal expansion coefficient. The residual tensile stress resulted in fracture in the weaker material, away from the interface. The decreased strength compared with the value of the monolithic ZT60A can be attributed to the presence of residual stresses. In this regard, the distance from the interface at which fracture occurred is especially revealing.

To study the influence of the stress distribution, a series of joining experiments on the strength of the joined parts was conducted. These experiments consisted of bonding parts with various alumina fractions (ranging from 0 to 40 vol %) to a part of a fixed composition (ZT60A). Measured flexure strengths are shown in Figure 3, along with strength values for the monolithic composites. All samples failed in the ZT60A part, the weaker material, as discussed earlier. Strength was reduced as the difference in composition between the joined parts increased; consequently, the thermal-expansion coefficient and residual stresses increased. However, the reduction in strength was not dramatic. For the experiment involving the largest difference in composition (YSZ joined to ZT60A), flexure strength was decreased by 25% compared with the strength of monolithic ZT60A. These results provide a good indication of the applicability of the plastic joining technique when dissimilar materials with different thermal-expansion coefficients are bonded. It needs to be emphasized again that fracture did not take place at the joint.

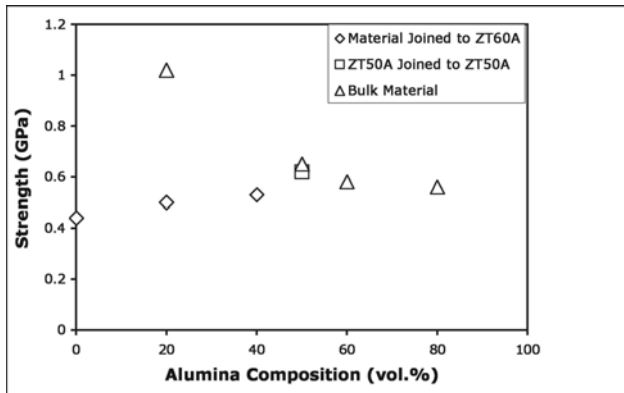


Figure 3. Representation of strength vs. alumina composition for monolithic materials. In the joining experiments, the alumina fraction corresponds to the composition of the materials being joined to ZT60A, while the strength is that of the ZT60A where fracture occurs.

The fracture strength of brittle materials without any surface residual stress can be given by

$$\sigma_f = K_{ic} / Y(a)^{1/2} \quad (1)$$

where K_{ic} is the fracture toughness, Y is a geometrical factor and is $=2/\pi^{1/2}$ for a sample tested in the four-point-bend mode, and a is the length of the crack. Using the measured physical properties of the monolithic ZT60A and ZT40A, one can calculate that the critical flaws are 37 and 25 μm , respectively. These values are consistent with typical processing flaws.

The influence of residual stresses on the fracture from inherent processing flaws may now be considered. For ceramics with surface residual stresses, the apparent fracture toughness (K_{ic}^{app}) can be given by the expression

$$K_{ic}^{app} = K_{ic}^o + K_I^R \quad (2)$$

where K_{ic}^o is the fracture toughness of the stress-free material K_I^R is the stress intensity from the surface residual stress and is given in terms of residual stress (σ_r), half-penny crack length (a), and non-dimensional stress intensity factor (Y) as $K_I^R = \sigma_r Y(a)^{1/2}$

For a four-point-bend test configuration, as used in this work,

$$K_I^R = 2\sigma_r (a/\pi)^{1/2} \quad (3)$$

Therefore, the apparent fracture toughness is related to the flaw size by

$$K_{ic}^{app} = K_{ic}^o - 2\sigma_r (a/\pi)^{1/2} \quad (4)$$

Using a flaw size of 40 μm and the apparent fracture toughness of ZT60A (3.6 $\text{MPa}\sqrt{\text{m}}$) as calculated from Eq. (1), the fracture strength is calculated to be 500 MPa, certainly consistent with the measured fracture strength of 530 MPa observed for one of the three samples tested.

The lower strengths of 360 MPa and 201 MPa observed for the two samples were believed to be due to larger flaws. To confirm this assumption, fractography was conducted on the two low-strength samples. SEM, as shown in Figure 4 shows surface damage on the tensile surfaces in two samples. It is likely that this damage was introduced during the sample preparation steps or handling. Typical lengths of this damage range from 70–90 μm . Using a nominal crack length of 80 μm and an apparent toughness of 3.6 $\text{MPa}\sqrt{\text{m}}$ for ZT60A, the fracture strength was calculated to be approximately 350 MPa. This value is consistent with that observed for the samples with surface damage (Table 1).

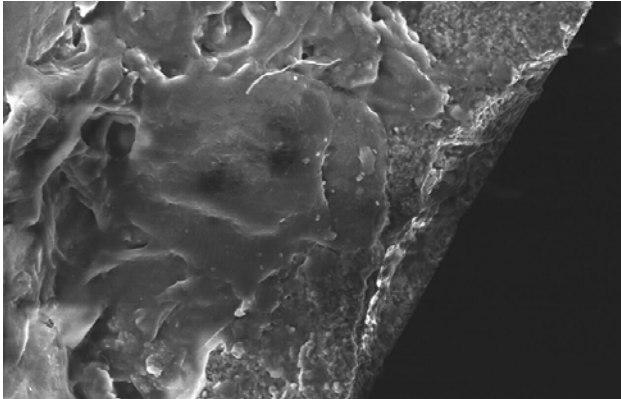


Figure 4. SEM micrograph showing a large flaw on the tensile surface of the ZT60A section from a fractured ZT60A/ZT40A joined sample.

Differences in thermal-expansion coefficients influence not only the mechanical response of the joined part, but also the joining process itself. Figure 2 represents an FEA simulation of the stress distribution along the center of joined pieces of ZTA ceramics. The stresses parallel (shear) and perpendicular (tensile) to the joint interface vary from the center to the edge of the two sections. The stresses parallel to the interface increase in magnitude at locations approaching the interface, whereas the perpendicular stresses decrease near the interface. It is believed that if the magnitude

of the shear stresses is high enough and if there is a stress concentration at the interface, spontaneous fracture can occur in the joining process. This is clearly seen in Figure 5, which shows a YSZ-ZT60A sample that fractured spontaneously during joining.

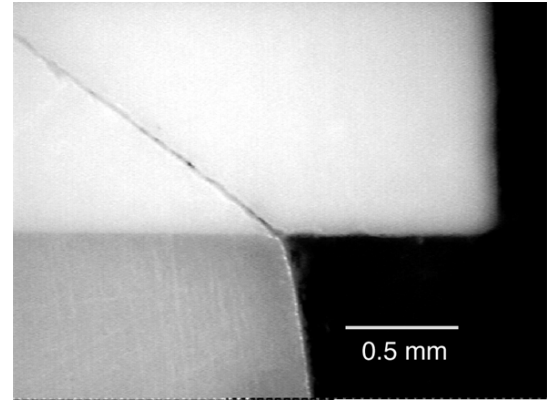


Figure 5. Micrograph of as-joined sample of YSZ/ZT60A. Fracture starts at the free surface and propagates, deviating away from the interface through the weaker material (ZT60A, top).

The fracture originates from the corner that acts as a stress concentrator. At the surface, because of the high shear stresses, the crack is parallel to the interface. As the crack traverses toward the center section, tensile stresses are more dominant and the crack becomes perpendicular to the interface. It is this change in stress intensities from shear to tensile that causes the kinking of the cracks observed for spontaneously cracked joint samples. Moreover, spontaneously cracked samples evince that the interface is strong, resistant, and pore-free, otherwise the sample would have fractured along the line of the joint.

However, the fact that residual stresses can make the joint fail spontaneously imposes a significant restriction upon the materials that can be joined by plastic deformation. The primary objectives are to control stresses, increase the strength of the joint, and, above all, prevent fracture during joining.

An obvious solution was to use an interlayer of an intermediate composition to reduce the residual stresses. However, FEA indicated that the stress reduction was only 30% (Figure 6)

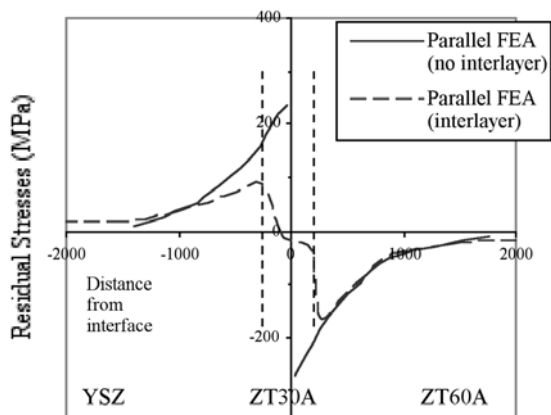


Figure 6. Comparison between residual stress distribution for a direct joint (YSZ/ZT60A) and the same joint using an 0.5 mm-thick interlayer of ZT30A.

ZT60A was jointed to YSZ with ZT30A interlayers of thicknesses of 10, 100, and 1000 μm . All samples exhibited failures similar to those in the direct joining experiment (Figure 7). The failure pattern is consistent with the stress profiles discussed earlier. In future work, we will use functionally graded materials to overcome this problem.

Conclusions

We have shown that we can form pore-free, very strong joints in a wide-variety of important materials by plasticity. We have joined structural and electronic ceramics, metal-matrix composites, Ni_3Al , whisker-reinforced ceramics, and cermets. Previous work has shown that experimental determinations of the residual stress agree with calculated values. Little surface preparation and modest temperatures are required for deformation joining. Those

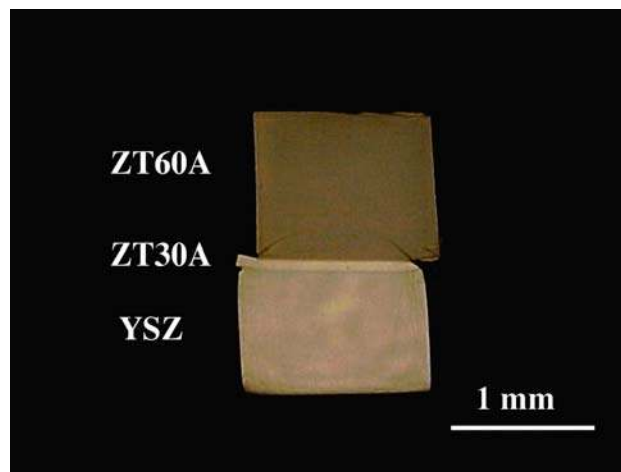


Figure 7. Fracture in a joined sample of YSZ bonded to ZT60A with an interlayer of ZT30A.

factors make the process attractive for commercialization.

It was originally assumed that the materials to be joined were superplastic and that the joining occurred by grain-boundary rotation. Grains would rotate and interpenetrate, resulting in a strong bond. However, many of the materials successfully joined are not superplastic. It is assumed, however, that most of the materials deform by grain-boundary sliding. So we believe that the critical process is the grain rotation that must occur in a grain-boundary sliding process in order to maintain strain continuity and avoid cavitation.

This assumption can be verified by performing in-situ experiments using the APS. Preliminary ex-situ experiments on 2.3- μm grain-sized YSZ have shown that we can identify single grains. Small rotations should allow us to determine the grain orientations. A recent proposal to perform more ex-situ APS experiments received extremely high marks. The ultimate goal would be to measure grain rotation while joining and to compare the rotations with those predicted from deformation theories.

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