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Microstructure and mechanical properties of superplastically joined yttria-partially-stabilized zirconia (Y-PSZ) ceramics

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Abstract

Joining of oxide ceramics has been recognized as a key issue in their successful utilization in a variety of demanding, high temperature applications. Superplastic joining of yttria-partially-stabilized zirconia (Y-PSZ) ceramics has been carried out to avoid the deleterious effects of joint interlayer materials. Microstructural characterization of superplastically formed joints indicate that the joint interface is indistinguishable from the bulk and that there is not grain growth across the interface. Compression tests conducted on double-notch specimens show that very good bonding was achieved in the superplastic joining process. From these experiments an underestimation of the shear strength was obtained as the specimens failed in the bulk. The room temperature flexural strength of joints measured had values up to 185 MPa. © 2000 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Ceramic joining has been recognized as one of the enabling technologies for the successful application of advanced ceramic materials in a wide variety of ground-based and aerospace applications. Many of these applications require fabrication of complex shaped components, which is quite expensive. It is often economical to fabricate simple shapes and join them together. Thus, it is very important to have a good joining technique that is practical, robust, and economically viable. A number of techniques to join advanced ceramics have been reported in the literature.[1–4] The main emphasis in these previous efforts has been developing joint materials and techniques for modest temperature and low stress requirements. Overviews of various joining techniques, e.g. mechanical fastening, adhesive bonding, welding, brazing, and soldering have been provided in recent publications.[1–4] The development of joining technology for oxide ceramics for high temperature and high stress applications, has been limited. The majority of the techniques used today are based on the joining of monolithic oxide ceramics by diffusion bonding, metal brazing, brazing with oxides and oxynitrides, or diffusion welding. In many instances, the joint use temperatures are lower than the temperature capability of the base oxide ceramics. The joints produced by brazing techniques can have different thermal expansion coefficients than the parent materials, which contributes to stress concentration in the joint area.

Ceramic joint interlayers have been developed as a means of obtaining high-temperature, high-strength joints. In the joining of fine grained ceramics, especially the yttria-partially-stabilized zirconia (Y-PSZ), the use of structural superplasticity for making joints has been very successful, achieving good joints at low deformation ($\varepsilon \approx 2\%$).[5–7] It is now accepted that Rachinger grain boundary sliding (GBS) is the mechanism controlling the superplasticity in these fine grained ceramics. It is a microstructural feature that during deformation, the grains of the polycrystalline materials change their nearest neighbors, retaining their initial shape constant, even after high deformation. When two parts in contact

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are superplastically deformed, the grains of one part interpenetrate the other producing a good joint.

In this paper, microstructural characterization results of superplastically joined yttria-partially-stabilized zirconia (Y-PSZ) ceramics will be presented. Shear strengths of the joints measured using compression double-notch tests will be discussed. Preliminary data on room temperature flexural properties of joints will be reported.

2. Experimental procedure

The starting material was 3 mol% yttria-partially-stabilized zirconia (3Y-PSZ) with an average grain size of 0.3 μm. Samples of dimensions 24×2.5×3 mm were cut using a low speed diamond saw and polished using diamond paste of decreasing sizes down to 3 μm. To produce the joints, two pieces were compressed at constant cross-head speed (5 μm/min≈1.5×10^{-5} s^{-1}) in air at 1400°C to final deformation of 5 and 10%. In order to determine the interlaminar shear strength of the joints, shear tests were carried out on notched specimens. Two centrally located notches, spaced a fixed distance apart on opposite faces, were machined halfway through the thickness of the sample. These samples were compressed parallel to the interface until final failure. A schematic and a photograph of specimen are shown in Fig. 1(a) and (b).

Compression tests, as shown in Fig. 1, were conducted on double-notched specimens to determine the shear strength of the joint according to ASTM C-1292.[8] The tests were carried out, for each type of pieces joined and final deformation of 5 and 10%, at room temperature and at a constant cross-head displacement of 5, 10 and 20 μm/s, corresponding to strain rate of 2, 4 and 8×10^{-4} s^{-1}, using a mechanical testing machine,[9] and a lateral support fixture to prevent buckling. Shear stresses were calculated using the relationship:

$$\text{Shear stress} = P_{\max}/A \tag{1}$$

where $P_{\max}$ is the applied maximum load and A is the shear stressed area. The area (A) is calculated as $A = t\times h$, where t is the average specimen width and h is the distance between the notches [Fig. 1(a)].

For the three point flexure test, two cylindrical specimens of 7 mm diameter and 24 mm length were joined with the same technique as described above to a final deformation of 5 and 10%. In this configuration, maximum stresses were concentrated in the joint regions in the middle of the bars. In the three point flexure test, the fracture stress (σ) was calculated as:

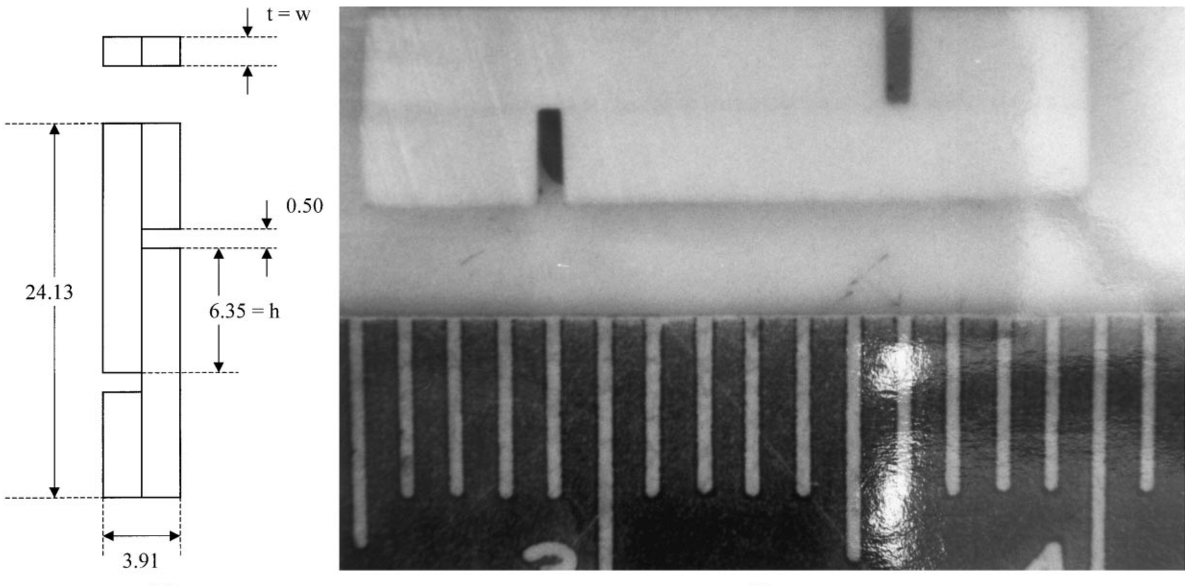
$$\sigma = 8LP/\pi d^3 \tag{2}$$

where L is the support span ($L = 19.89$ mm), P the applied load, and d is the diameter of the specimen. The flexural tests were conducted at a loading rate of 0.5 mm/min. After the mechanical testing, fracture surfaces were examined using optical and scanning electron microscopy.

3. Results and discussion

3.1. Joint microstructure

The microstructure of the superplastically joined ceramics were observed by SEM. Fig. 2 shows the interface of a junction achieved with $\varepsilon = 10\%$. In this



(a) (b)

Fig. 1. (a) Schematic of double-notch shear test specimen (dimensions in mm); (b) typical photograph of double-notch shear test specimen.

micrograph it is very difficult to distinguish the interface. In order to recognize the interface, the micrograph was taken showing the discontinuity at the end of the joined pieces. The vanishing of the interface, as observed in this figure, is the proof that GBS is very active in superplastically deformed yttria-partially-stabilized zirconia (Y-TZP) ceramics with small grain size (0.3 μm). No differences in the microstructure have been found when samples were deformed at 5%, indicating that the junction can be achieved as soon as the sample is deformed in superplastic regime. Further details of superplastic joining have been provided elsewhere.[5–7]

3.2. Mechanical strength

Compression tests were conducted on superplastically joined, double-notch specimens. The joint shear stresses were calculated from the load vs. cross-head displacement curves for all the specimens. Typical plots for specimens joined with 5 and 10% deformation are given in Fig. 3(a) and (b). The data indicate that the shear stress (load) increases monotonically up to its peak value, which was followed by a large load drop coinciding with the failure of the specimen. There was some evidence of small load drops prior to failure but it was not possible to relate these events with the occurrence of damage in the specimens. The specimens failed in several pieces but there was no evidence of shear failure in the bonded area. Some of the failure origins could be traced to the root of the machined notches. Both types of joined specimens and bulk specimen failed at relatively large stresses between 104 and 116 Mpa independent of the final deformation or of the cross-head displacement. The same type of experiment (same configuration as Fig. 1) has been conducted in a bulk 3Y-PZP and it is interesting to note that no differences in the compressive fracture stress values were found compared with the joined samples

The superplastically joined specimens also show good behavior in the flexural tests. The pieces joined with a final deformation of 5% showed fracture strength of

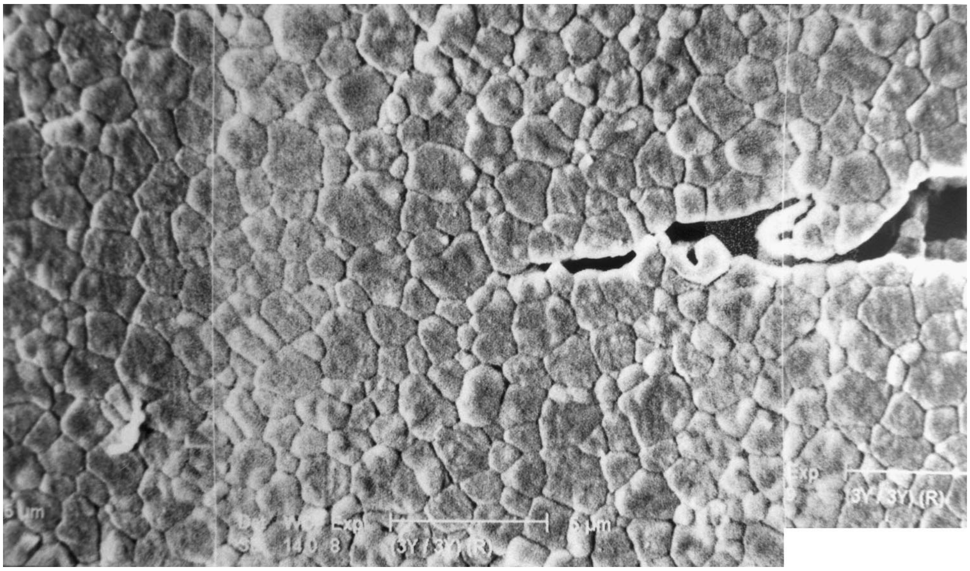


Fig. 2. SEM micrograph of the joint microstructure. In order to recognize the interface, the micrograph was taken showing the discontinuity at the end of the joined pieces.

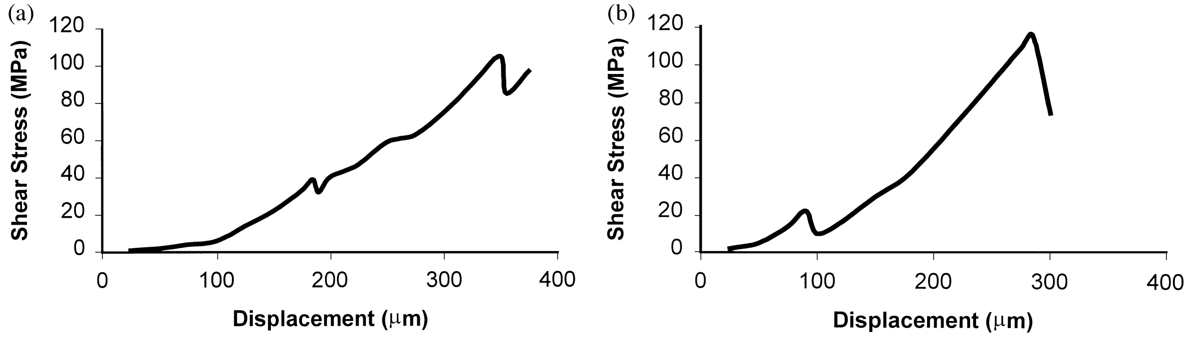


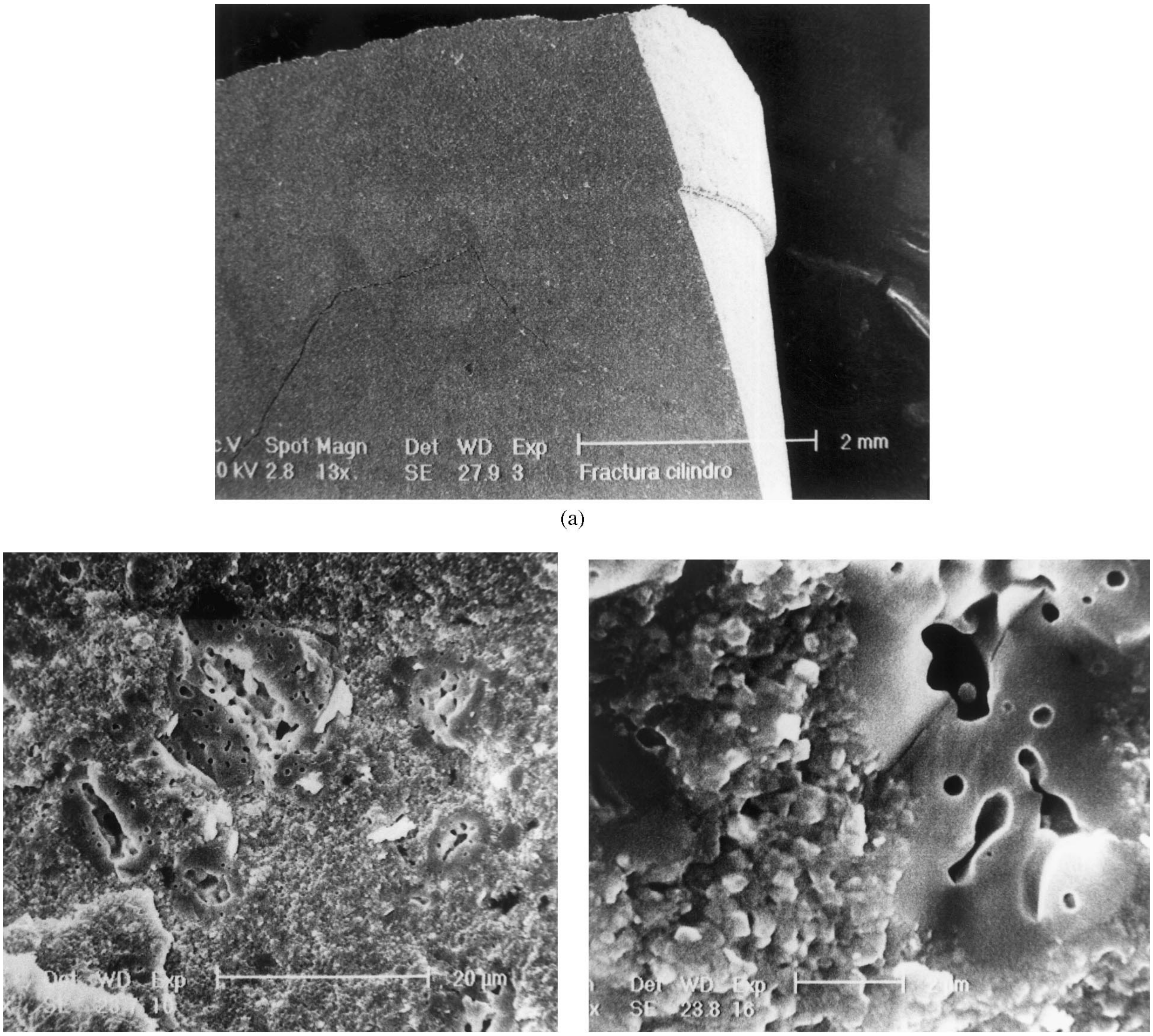
Fig. 3. Average shear stress in joint vs. cross-head displacement results from the compression of double-notch specimens: (a) 5% deformation, (b) 10% deformation (loading rate: 5 μm/s).

185 MPa at room temperature. The fracture occurred at the joint regions. The specimen joined with 10% strain broke away from the joint at 45 MPa, as shown in Fig. 4(a), due to a processing flaw in the bulk of the material. The fracture origins was identified using the scanning electron microscopy and are given in the following section.

It is important to mention that the shear and flexural strength values of joints and bulk materials reported in this paper are significantly lower than the values reported for monolithic YSZ-based materials in the literature.[10] A wide variety of factors influencing the ultimate strength of ceramics are well known. The majority of the strength discrepancies can be attributed to different fabrication methods and processing conditions (time, temperature, powder grain size and quality, etc.) as well as variation in testing conditions (three point vs. four point bend tests) with different specimen sizes. In addition, in the three point bend test configuration for joints, maximun stresses were concentrated in the regions in the middle of the bars. It will be preferable to test joints under four point bending conditions, where loads are redistributed throughout the bars. In the present work, four point bend tests could not be carried out because the specimens were too small.

3.3. Fractography

The fractographic analyses of tested specimens were carried out using SEM. Detailed analysis of the fracture surface [shown in Fig. 4(b) and (c), shows the presence of processing related flaws in this material. The scanning electron micrographs in Fig. 4(b) and (c) show the porosity



(a)

(b)

(c)

Fig. 4. (a) Micrograph showing specimen fracture away from the joints; (b) and (c) scanning electron micrographs showing processing related flaws and porosity in the bulk material.

in the material. These processing related flaws acted as the failure origins which resulted in low flexural strength (~45 MPa).

Efforts are underway to fabricate joints with more homogeneous microstructure and composition, vary the joint thickness, and evaluate the effect of high temperature heat treatment on the joint strength in these materials.

4. Conclusions

It has been demonstrated that the structural superplasticity can be successfully utilized as a joining approach to join yttria-partially-stabilized zirconia (Y-PSZ) ceramics. These superplastically formed joints have good shear and flexural strengths at room temperature. The specimens did not fail in shear but broke in many pieces. These joints show good flexural strength up to 185 MPa at room temperature. Further studies are underway to test these joints at high temperature under shear and flexure mode.

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