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# Superplastic joining of alumina and zirconia ceramics

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**Abstract**

$Al_2O_3/ZrO_2$ composites with from 0 to 100% $ZrO_2$ content were bonded by superplasic flow at 1280, 1350 and 1400 °C under the pressure 30 or 50 MPa during the time from several min to several hours. FGM joints between $Al_2O_3$ and $ZrO_2$ parts and joints between non-superplastic coarse grained alumina bars with aid of thin superplastic plates were also obtained. All the joints were pore-free and strong. For $\Delta\alpha > 1 \times 10^{-6}\,°C^{-1}$ cracks parallel to interface arose near the joint. Residual stress distribution was evaluated by finite-element and Vickers indentation analysis. The calculations revealed that critical tensile stresses appeared only in perpendicular direction to the interface.


*Keywords:* Joining; $Al_2O_3$; $ZrO_2$; Superplasticity; Strength

## 1. Introduction

The necessity of using the ceramic elements with complicated shapes in many technical systems has caused the increasing interest of ceramics–ceramics joining. It seems to be the simple and economical way of obtaining of complex shaped components. There are many techniques for material bonding.[1,2] Some of them need the metal interlayer as a filler. It could create residual stresses in joints because of thermal expansion coefficient mismatch between metal and ceramics and could also cause poor thermal resistance. Another technique which enables us to avoid mentioned above faults is a diffusion bonding. The diffusion bonding[2] (often called diffusion welding) is a process of durable joining at a temperature lower than the melting of the materials as a result of plastic strain interaction and the diffusion of atoms or ions in the boundary layers of the elements under joining. The process of bonding proceeds at a high temperature with pressure onto the bonded elements. A discovery of superplasticity in fine grained tetragonal zirconia polycrystals[3] and in other ceramics[4] created new possibilities of making ceramics–ceramics joints in a similar way as the diffusion bonding.[5–7] During superplastic deformation, the grains of polycrystalline materials change their nearest neighbors, retaining their initial shape constant. Where two parts in contacts are superplastically deformed, the grains of one part interpenetrate the other producing a joint. Superplasticity enables to lower temperature and to shorten time of joining in comparison with diffusion bonding. Surface preparation of the parts to be joined is easier and faster because superplastic bonding, unlike diffusion bonding, tolerates surface asperities. Additionally, it will be shown in this work that for obtaining good joints the only one of the joining materials should be superplastic. Hence joining non-superplastic ceramic samples with aid of thin superplastic interlayers will become possible.

The objectives of the present work were following:

(1) obtaining joints between alumina and zirconia and different alumina/zirconia samples,
(2) obtaining joints between non-superplastic alumina samples.

## 2. Experimental procedure

For performing of the first aim $Al_2O_3/ZrO_2$ composites from 0 to 100 $ZrO_2$ content were used. Their preparation and some mechanical properties at room temperature are described by Boniecki et al.[8] but superplastic deformation data at high temperature are presented by Boniecki et al.[9] Some properties of the materials are summarized in Table 1.

From these materials cubic samples with side of 2.5 mm and plates 2.5 mm × 2.5 mm × 0.5 mm were cut and polished (cubes from one side and plates from two sides). The joining tests were performed using universal testing machine Zwick 1446 with a furnace at 1280 and 1350 °C at air under the compressive stress of 30 and 50 MPa. The pair of cubes were joined

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Table 1
Properties of alumina/zirconia ceramics used for joining

| Material | wt (%) | vol (%) | $d_{Al_2O_3} d_{ZrO_2}$ (μm) | $\rho$ (g/cm$^3$) | $E$ (GPa) | $\sigma_c$ (MPa) | $K_{Ic}$ (MPa m$^{1/2}$) | $\alpha$ ($\times 10^{-6}\,°C^{-1}$) | $\nu$ |
|---|---|---|---|---|---|---|---|---|---|
| *f*-$Al_2O_3$ | 0 | 0 | 0.87 (0.49) | 3.91 | 341 (20) | 190 (17) | 3.30 (0.05) | 8.46 | 0.22 |
| Al20 | 20 | 14 | 0.37 (0.19)<br>0.33 (0.19) | 4.21 | 319 (6) | 215 (17) | 3.95 (0.06) | 8.92 | 0.23 |
| Al40 | 40 | 30 | 0.35 (0.09)<br>0.27 (0.17) | 4.56 | 293 (8) | 245 (22) | 4.36 (0.05) | 9.09 | 0.24 |
| Al60 | 60 | 50 | 0.30 (0.08)<br>0.22 (0.19) | 4.88 | 269 (8) | 260 (36) | 4.40 (0.12) | 9.64 | 0.26 |
| Al80 | 80 | 72 | 0.31 (0.06)<br>0.18 (0.04) | 5.35 | 235 (5) | 265 (20) | 4.77 (0.32) | 10.71 | 0.27 |
| $ZrO_2$ | 100 | 100 | 0.47 (0.13) | 6.05 | 200 (4) | 800 (79) | 5.10 (0.35) | 11.26 | 0.29 |

wt and vol are weight and volume fraction of $ZrO_2$, respectively, $d$ the grain size, $\rho$ the density, $E$ the Young modulus, $\sigma_c$ the bending strength, $K_{Ic}$ the fracture toughness, $\alpha$ the thermal expansion coefficient, and $\nu$ is the Poisson's ratio. Numbers in brackets mean standard deviations. $ZrO_2$ is the zirconia in tetragonal phase (stabilised with 3 mol% $Y_2O_3$, made of powder obtained from Tosoh Co., Japan). *f*-$Al_2O_3$ is the fine grained alumina made of high purity (99.99%) alumina powder (obtained from Sumitomo Chemical Company, Japan) doped with 500 ppm MgO and 100 ppm $SiO_2$.

with polished surfaces (Fig. 1). The plates were stacked in the sequence: Al80–Al60–Al40–Al20, and next placed between $ZrO_2$ and $Al_2O_3$ cubes in order to obtain functional gradient material (FGM) samples (Fig. 1). The hold time was from several minutes to several hours (it depended on the temperature and the applied stress). The strain did not exceed 5%. Every joint was ground and polished from one side perpendicular to joined surface in order to reveal deeper parts of materials and next examined with aid of an optical microscope. Some joints were viewed on scanning electron microscope (SEM). In order to measure residual stresses, arising due to joining materials with different thermal expansion coefficients, Vickers indentation analysis (VIA)[7] was used. Several indentations were made in different distances from the interface with load 49.1 N. Crack lengths in the direction parallel and perpendicular to the interface were measured. Hence the residual stress distribution was calculated. This stress distribution was also evaluated by finite-element analysis (FEA). The calculation were made under the following assumptions:

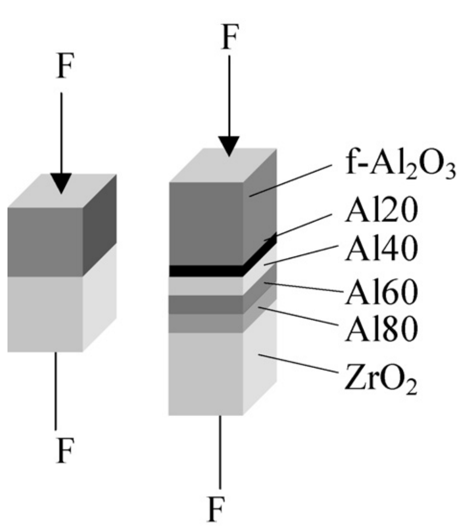


Fig. 1. Illustration of bonding of superplastic materials.

(1) the material properties are isotropic,
(2) residual stresses are not accommodated by plastic deformation for cooling from 1200 °C to room temperature,
(3) Young modulus and thermal expansion coefficient are function of temperature.

In the second part of the work, non-superplastic alumina (*c*-$Al_2O_3$) ceramic bars (4 mm × 4 mm × 14 mm) were joined with aid of superplastic interlayers (4 mm × 4 mm × 1 mm) (Fig. 2). The used materials are shown in Table 2. The joined surfaces (4 mm × 4 mm) were only ground. The joining tests were made at 1400 °C, in air, under the pressure 30 MPa during 3 h. After that the bonding strength was measured at room temperature by the three-point bending test (span = 25 mm) at a crosshead speed 1 mm/min.

## 3. Results and discussion

The results of the bonding tests for alumina/zirconia composites are shown in Table 3. When $\Delta\alpha > 1 \times 10^{-6}\,°C^{-1}$ between the bonded materials the parallel to the joining surface cracks appeared in the material with lower $\alpha$ (containing more alumina). The joints with these cracks were marked in Table 3 as cracked. An optical microscope photograph of the bonding interface of *f*-$Al_2O_3$–$ZrO_2$ (1350 °C, 30 MPa, 55 min) and a scanning electron microscope photograph of *f*-$Al_2O_3$–Al20

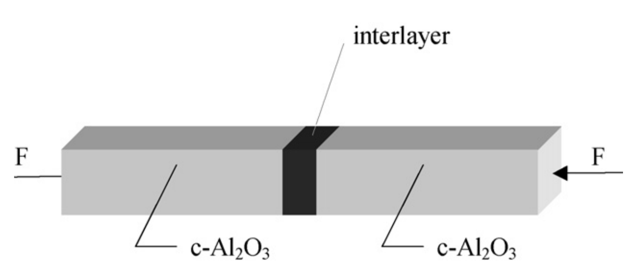


Fig. 2. Illustration of bonding of non-superplastic alumina bars with aid of superplastic interlayer.

Table 2
The materials used for obtaining joints between non-superplastic alumina ceramics

| Material | vol (%) | $d_{Al_2O_3} d_{ZrO_2}$ (μm) | $\rho$ (g/cm$^3$) | $E$ (GPa) | $\sigma_c$ (MPa) | $K_{Ic}$ (MPa m$^{1/2}$) | $\alpha$ ($\times 10^{-6}$ °C$^{-1}$) |
|---|---|---|---|---|---|---|---|
| $c$-$Al_2O_3$ | 0 | 4.27 (3.52) | 3.98 | 386 (5) | 351 (40) | 3.62 (0.05) | 8.50 |
| S15 | 15 | 2.18 (0.88)* 0.74 (0.26) | 3.95 | 247 (9) | 191 (16) | 4.49 (1.03) | 8.85 |
| E20 | 20 | 1.14 (0.41) 0.36 (0.12) | 4.38 | 312 | 319 | 4.8 | 9.10 |
| SAlZr | 40 | 0.16 (0.05)** | 4.63 | 241 (5) | 219 (5) | 3.86 (0.13) | 9.64 |

$c$-$Al_2O_3$ contains 99.5 wt% $Al_2O_3$, 0.2 wt% MgO, 0.25 wt% $Y_2O_3$ and 0.05 wt% other oxides, (S15) a composite of spinel $MgAl_2O_4$ and $ZrO_2$ (*, grain size of spinel), (E20) a composite of $Al_2O_3$ and $ZrO_2$ stabilised with 2 mol% $Y_2O_3$, (SAlZr) a composite of 30 vol% spinel, 30 vol% $Al_2O_3$ and 40 vol% $ZrO_2$ (**, grain size is an average for all the components), (vol) volume fraction of $ZrO_2$. The other materials used as interlayers are listed in Table 1.

Table 3
Results of the bonding tests for alumina/zirconia

| | $f$-$Al_2O_3$ | Al20 | Al40 | Al60 | Al80 | $ZrO_2$ |
|---|---|---|---|---|---|---|
| $f$-$Al_2O_3$ | G | G | G | C | C | C |
| Al20 | | G | G | G | C | C |
| Al40 | | | G | G | G | C |
| Al60 | | | | G | G | G |
| Al80 | | | | | G | G |
| $ZrO_2$ | | | | | | G |

G: Good bonding (without any visual pores and cracks), C: cracked.

(1350 °C, 30 MPa, 38 min) are shown in Fig. 3. Both joints seem to be well bonded without any pores, but in $f$-$Al_2O_3$–$ZrO_2$ the parallel crack in alumina is visible (at a distance about 140 μm from the interface).

In the Fig. 4, there are optical micrographs of a Al80–$ZrO_2$ (1280 °C, 30 MPa, 115 min) joint showing the crack pattern of indentations made near the interface. It is shown that cracks parallels to the interface are significantly larger in the material with lower $\alpha$ than in the material with higher $\alpha$. The reverse situation is with cracks perpendicular to the interface.

The phenomenon observed in Figs. 3a and 4 were explained by the stress calculations made by FEA (Fig. 5a) and by VIA (Fig. 5b). The residual tensile stress perpendicular to the interface appeared in material with lower $\alpha$ near the joint. When the difference between thermal expansion coefficients is too big the tensile stress becomes bigger than material strength and a crack parallel to the interface arises (Fig. 3a).

The stress analysis (made by FEA) showed that the normal stresses have an opposite sign than the corresponding parallel stresses. It means that if compressive normal stresses exist the parallel stresses are tensile and vice versa. The critical tensile stresses appeared only in perpendicular direction to the interface.

Four joints between $f$-$Al_2O_3$ and $ZrO_2$ with FGM interlayers at 1280 °C, 50 MPa, 40–125 min and four at 1350 °C, 30 MPa, 25–75 min were made. During grinding perpendicularly to the interfaces three joints made at 1350 °C and one made at 1280 °C cracked near Al60 plates. The residual stress analysis (made by FEA) showed that the maximum tensile stress arises in this interlayer (about 250 MPa). It is difficult to say why joints made at 1350 °C are weaker than those made at 1280 °C. Maybe the bigger pressure used during joining at 1280 °C caused arising of better joints.

In the second part of the work, several joints between non-superplastic $c$-$Al_2O_3$ with aid of superplastic interlayers (Tables 1 and 2) were made. The results of three point bending test of obtained joints are collected in Table 4.

Except the joint with $f$-$Al_2O_3$ all the samples cracked in alumina bars near the joint. The first listed in Table 4 sample cracked on the surface between the alumina bar and $f$-

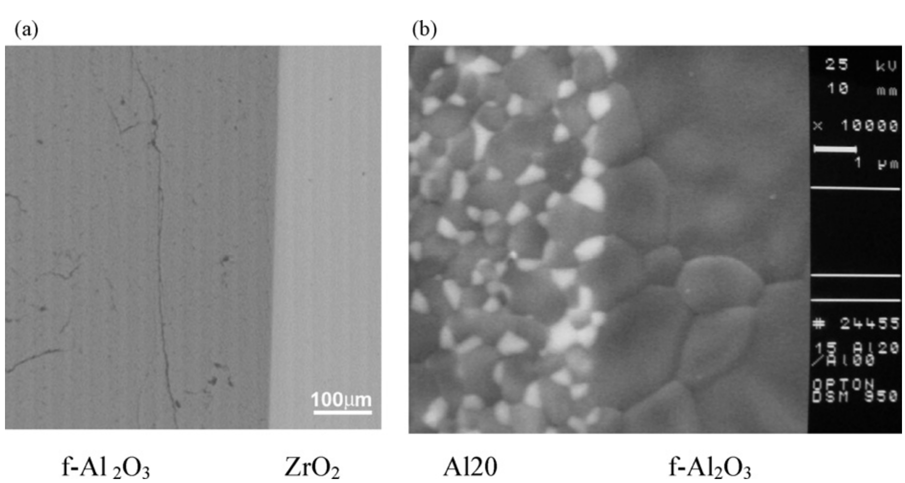


Fig. 3. Optical microscope photograph of $f$-$Al_2O_3$–$ZrO_2$ bonding showing the parallel to the interface crack in alumina (a) and SEM photograph of Al20–$f$-$Al_2O_3$ bonding (b).

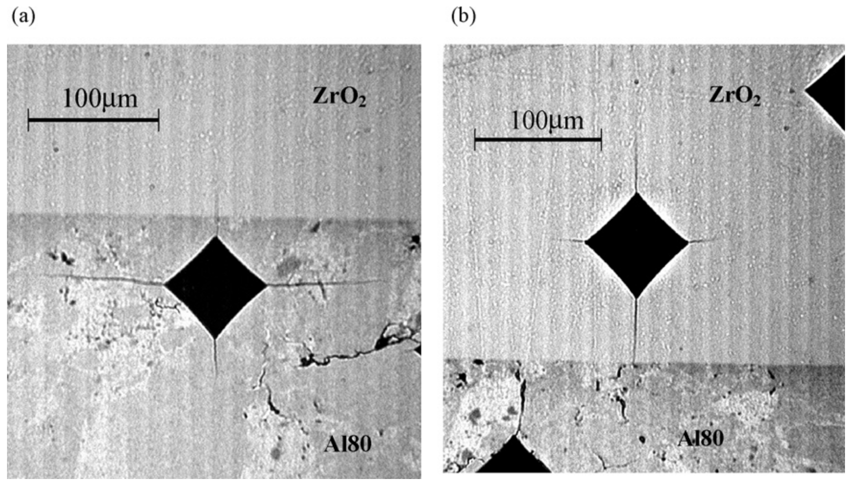


Fig. 4. (a and b) Optical micrographs of a Al80–$ZrO_2$ joint showing the crack pattern of indentations made near the interface.

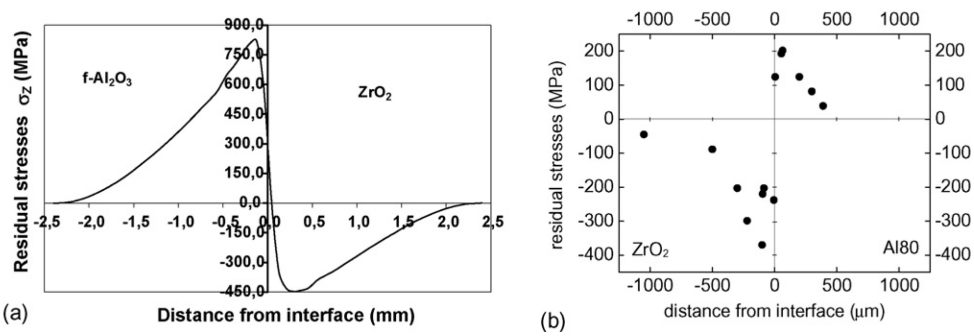


Fig. 5. Residual stress distribution (perpendicular to the interface) obtained by FEA for *f*-$Al_2O_3$–$ZrO_2$ joint (a) and by VIA for Al80–$ZrO_2$ joint (b).

$Al_2O_3$ interlayer. A SEM photograph of *c*-$Al_2O_3$–Al20 joint and an optical photograph of *c*-$Al_2O_3$–SAlZr joint are shown in Fig. 6.

Fig. 6 shows us good joining without any porosity between non-superplastic coarse grained alumina and fine grained superplastic interlayers. The crack in alumina (Fig. 6b) is caused by the residual stress larger than the material strength due to thermal expansion coefficient mismatch. Therefore, the joint with SAlZr interlayer is the weakest in Table 4. Other joints with $\alpha_{interlayer} > \alpha_{alumina}$ are stronger and crack in alumina. It is caused

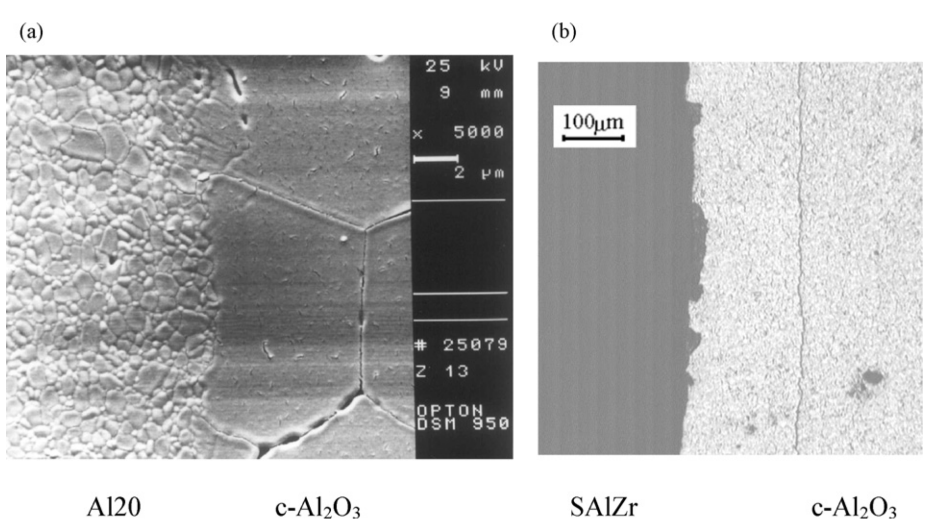


Fig. 6. SEM photograph of Al20–*c*-$Al_2O_3$ bonding (a) and optical microscope photograph of *c*-$Al_2O_3$–SAlZr bonding showing the parallel to the interface crack in alumina (b).

Table 4
Bending strength of the joints between non-superplastic alumina in function of the kind of interlayer

| Interlayer | Bending strength (MPa) |
|---|---|
| *f*-$Al_2O_3$ | 120.0 |
| Al20 | 154.6 |
| S15 | 195.4 |
| E20 | 166.1 |
| SAlZr | 60.8 |

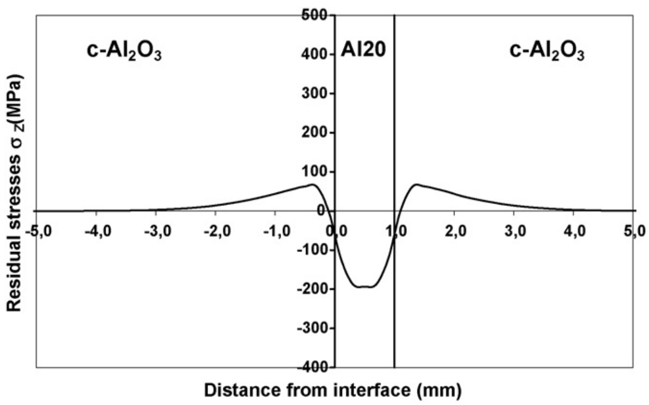


Fig. 7. Residual stress distribution (perpendicular to the interface) obtained by FEA for *c*-$Al_2O_3$–Al20–*c*-$Al_2O_3$ joint.

by the residual compressive stresses in the interlayer normal to the interface and tensile stresses in alumina (Fig. 7).

## 4. Conclusions

- Good, pore-free joints between pairs of different $Al_2O_3/ZrO_2$ composites with aid of superplastic flow were obtained.
- FGM joints between $Al_2O_3$ and $ZrO_2$ were also made.
- The joints were without cracks when $\Delta\alpha < 1 \times 10^{-6}\,{}^{\circ}C^{-1}$.
- Vickers indentations analysis and finite-element analysis were used for calculations of residual stress distribution in the joints. Critical tensile stresses appeared only in perpendicular direction to the interface.
- Strong joints were obtained between non-superplastic coarse grained alumina bars with aid of thin superplastic interlayers. The maximum bending strength of about 200 MPa was attained.

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