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# Superplastic joining of 3Y-TZP

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#### **Abstract**

The microscopic processes and mechanisms for the superplastic joining of 3Y-TZP (3 mol% yttria partially stabilized tetragonal zirconia polycrystals) were examined. The cooperative grain-boundary sliding associated with superplastic deformation and flow plays an essential role in the joining at the interface between 3Y-TZP blocks. The mechanical strength of the joined interface was determined by three-point flexural loading, where three different types of joined interfaces were studied; the interfaces between (1) fine-grain-size 3Y-TZPs, (2) coarsegrain-size 3Y-TZPs, and (c) a fine-grain-size and a coarse-grain-size 3Y-TZPs. The strengths of these interfaces were examined by changing the compressive stresses applied to the interfaces during joining. It was concluded that the mutual migrations of grains across the interface through cooperative grain-boundary sliding (CGBS) dominates the strength evolution at the interface along with thermally activated diffusion processes.

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# **1. Introduction**

Joining of structural ceramics has been recognized as a key technology to obtain complex shapes and large pieces of engineering components in low cost processing[.1](#page-5-0) Several techniques have been proposed for joining ceramic materials such as direct diffusion bonding, metal brazing and so on.<sup>[2,3](#page-5-0)</sup> In particular, the utilization of superplastic deformation and flow is very efficient not only for forming complex structures but also for joining segments in a large structure. $4-6$ 

The CGBS through cooperative movements of grain groups is one of the essential processes in superplastic deformation.<sup>[7–17](#page-5-0)</sup> Present authors have posed a novel microscopic process for CGBS in the superplastic deformation of polycrystalline materials. $11-17$  Theoretical considerations and experimental works for CGBS have also been made by the present authors, having been concluded from the view point of the principle of minimum energy dissipation that the cooperative sliding of grain groups along grain-boundaries is essential for refractory polycrystalline materials to accommodate large-scale superplastic deformation and flow.

The intent of this paper is to examine the microscopic processes and mechanisms for the superplastic joining of 3Y-TZP (3 mol% yttria partially stabilized tetragonal zirconia polycrystals) on the basis of CGBS processes.

#### **2. Experimental**

# *2.1. Materials*

A fully dense 3Y-TZP was used as a test material. The average grain size of as-received material supplied from Nikkato Co. Ltd., Japan was  $0.3 \mu m$ . Besides the as-received ceramic block, the same material was annealed at  $1500\degree$ C for 3 h to obtain a 3Y-TZP block with the grain size of  $1.3 \mu m$ . The test specimens with a dimension of  $4 \text{ mm} \times 4 \text{ mm} \times 4 \text{ mm}$ ([Fig. 1\(a](#page-1-0))) were cut out of the as-received and the annealed 3Y-TZP blocks using a diamond saw, and then, polished with diamond past.

#### *2.2. Creep joining*

Two pieces of the test specimens were sandwiched between SiC-made plates and joined under a constant

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Fig. 1. Details for the geometries and the dimensions of (a) test specimens in joining, (b) flexural test specimen, and (c) three-point flexural test.

compressive stress  $\sigma_0$  ranging from 5 to 20 MPa at 1200 °C in air to various compressive strains. During this creep joining, the applied constant load and the induced creep displacement were measured by a water-cooled load cell (TCLZ-200KA, Tokyo Sokki Co. Ltd., Tokyo, Japan) and an optical extensometer (UDM 5000, Zimmer GmbH, Darmstadt, Germany), respectively (see Fig. 2). Details of the testing machine used in the compressive creep test were reported in the literature.<sup>[18](#page-5-0)</sup> The resultant compressive strains  $\varepsilon$  were systematically controlled by changing the time span of creeping.

Three different types of joining were studied; the joining between (1) the fine-grain-size  $(0.3 \mu m)$  3Y-TZPs (Type I), (2) the coarse-grain-size  $(1.3 \,\mu\text{m})$  3Y-TZPs (Type II), and (3) the fine-grain-size and the coarse-grain-size 3Y-TZPs (Type III).

#### *2.3. Mechanical strength*

The joined bodies were machined into rectangular bars with the dimensions depicted in Fig. 1(b) (7 mm in length, 4 mm in width and 3 mm in height). The mechanical strength of the joined interface was determined by three-point flexural loading with the test span of 6 mm at room temperature in air. The bulk materials (as received and heat-treated, respectively) without a joined interface having the same dimensions of flexural bars were also tested for reference. The details of the compressive joining and the flexural test were shown in Fig. 1(a–c).



Fig. 2. Schematic drawing of the apparatus for compressive creep joining.

#### **3. Results**

#### *3.1. Microstructure of joined interface*

Microscopic observations of the joined interface and its vicinity were made by electron scanning microscopy (SEM). No changes in the size and the shape of grains were observed after compressive creep joining. The typical SEM images of the joined interfaces for Types I and III produced under the compressive strains of about 0.04 are shown in [Fig. 3\(](#page-2-0)a and b), respectively. The white arrows in these figures mark the interface. It was rather difficult to identify the joined interface of Type I when it is subjected to the compressive strains exceeding 0.03.

#### *3.2. Creep curves for joining*

The creep curves (creep strain  $\varepsilon$  versus time  $t$  curves) during joining at  $1200\degree$ C for Types I and II interfaces, and their creep compliance  $D(t)$  [= $\varepsilon(t)/\sigma_0$ ] curves were plotted in [Fig. 4\(a](#page-2-0) and b), respectively. For the superplastic 3Y-TZP (Type I), the creep compliance curves were greatly dependent on the applied stresses as shown in [Fig. 4\(](#page-2-0)b), while all of the creep compliance curves for the coarse-grain-size 3Y-TZP (Type II) were well coincident, implying that the microscopic mechanisms of creep deformations are significantly different in Types I and II interfaces.

#### *3.3. Mechanical strength*

Although the flexural test span of 6 mm used in this work is smaller than that in the conventional flexural test with the test span of about 30 mm, the inert strength in the present test configurations for the bulk specimens without a joined interface agrees well with the value in the conventional test (about 1.2 GPa).

The mechanical strengths of the interfaces for these three types of joining were plotted in [Fig. 5](#page-3-0) against the applied strains. The dashed lines in [Fig. 5](#page-3-0) indicate the range of inert fracture strength for the specimens without a joined interface. The strength of Type I interface highly depends on the applied strain, i.e., a rising linear relationship in the semi logarithmic plot between the flexural strength and the compressive strain. In the range of compressive strains exceeding 0.04, the flexural strength reaches the value of the inert strength. It is worthy of emphasizing that the total creep strain significantly affects the failure strength of Type I interface. Accordingly, by way of example, the interface jointed for 5 h under 20 MPa is stronger than that for 160h under 10 MPa, for the total strain of the former is larger than the strain of the later. On the other hand, the evolution of the interface strengths of Types II and III is insignificant even after the interfaces were exposed to the larger applied strains and/or the longer creeping times.

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Fig. 3. SEM images for (a) Type I and (b) Type III interfaces subjected to a compressive strain of about 0.04. The arrows indicate the interfaces.



Fig. 4. (a) Creep curves and (b) creep compliance curves of Types I and II creep joining for various compressive stresses at 1200 ◦C.

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Fig. 5. Relationship between the flexural strength and the applied compressive strain during creep joining for Types I–III interfaces.

#### **4. Discussion**

# *4.1. The mechanism of joining in superplastic materials*

Several attempts for joining advanced ceramic materi-als have been reported in the literature.<sup>[1–3](#page-5-0)</sup> Among them, the utilization of superplastic deformation and flow is very efficient.[4–6](#page-5-0) In superplastic joining, GBS plays an important role in achieving a stronger interface. As reported in the literature,  $11-17$  the CGBS is the most plausible candidate for the microscopic processes in superplastic deformations of polycrystalline materials. A schematic illustration of this CGBS for compressive loading is shown in Fig. 6. When a two-dimensional close-packed aggregate (Fig. 6(a)) is compressed by two parallel plates, the grain groups shaping regular triangles slide along the boundaries of each triangle (Fig. 6(b)). This CGBS goes on during compressive loading with a step-wise reduction in the size of triangles. This process requires the least deformation energy.[11](#page-5-0) As typically demonstrated in Fig. 6(a–c), the grains at the tip of gray triangles are migrating into the bottom layer comprising only white grains in the first step of CBGS. This sliding migration of grains into neighboring planes of grains is very efficient for strengthening the joined interface.

Suppose two pieces of materials are superplastically compressed. It will easily be expected that the cooperative mutual migrations of grains in the respective pieces across the interface efficiently strengthen the interface joined. As a matter of fact shown in [Fig. 3\(a](#page-2-0)), the resultant interface of Type I is hard to be identified, due to the mutual migrations of grains through CGBS. On the other hand, as shown in [Fig. 3\(b](#page-2-0)), the interface of Type III remains intact, even after a large-scale creep strain applied. This fact suggests that no CGBS takes place in Type III interface. The interface of Type II is very similar to that of Type III. In conclusion, a strong interface like as that of Type I is only created by the mutual migrations of grains across the interface via CGBS in a superplastic menner.



Fig. 6. Schematic illustration for CGBS process, (a) before and (c) after the first step of deformation of the two-dimensional model aggregate under a uniaxial compression via (b) an intermediate state.

#### *4.2. Effect of grain size on joining*

Superplasticity is very grain-size-dependent; polycrystalline materials with coarse-grains are more resistant to deformation than those with finer grains.[17](#page-5-0) In CGBS, the deformation energy  $\Delta U(\varepsilon)$  required for overcoming grain interlocking along the interfaces of cooperative grain groups is given by the following relation<sup>11,12</sup>;

$$
\Delta U(\varepsilon) \propto K \left[ \frac{R}{L(\varepsilon)} \right]^2 \tag{1}
$$

where *R* and  $L(\varepsilon)$  are the mean size of grains and the mean length of cooperative grain groups at a specific compressive strain  $\varepsilon$ , respectively. The frontal coefficient  $K$  is the bulk modulus of the polycrystal. The experimental examinations for Eq. (1) have been quantitatively conducted by the present authors in tensile tests.<sup>[17](#page-5-0)</sup> Eq. (1) implies that the grain size *R* is significantly affects the energy of superplastic deformation. When an externally applied energy is less than the energy of  $\Delta U(\varepsilon)$ , CGBS dose not take place due to grain interlocking, leading to small-scale deformations through diffusional creep.

The creep compliance curves measured at various applied stresses give a useful guideline in the study for the deforma-



Fig. 7. Schematic illustration for the migration of grains across the interface, (a) before and (b) after creep joining.

tion mechanisms of polycrystalline materials at high temperatures. As readily seen in [Fig. 4\(](#page-2-0)b), all of the creep compliance curves (closed symbols) of Type II interface for different applied stresses fall on a single creep curve, indicating linear viscoelastic deformations, and suggesting that its dominant deformation mechanism will be lattice and/or grain-boundary diffusions. In this case, the joining proceeds merely with thermally activated diffusions, resulting in a rather weak interface. For the superplastic joining of Type I interface [open symbols in [Fig. 4\(b](#page-2-0))], the creep compliance curves are significantly dependent on the applied stresses, meaning that the deformation and flow are very nonlinear viscoelastic. This non-linear creeping behavior is one of the important features in superplastic deformation and flow. A theoretical considerations for the non-linear creeping behavior in superplastic deformation will be given in the literature.<sup>[19](#page-5-0)</sup>

# *4.3. Dependence of the strength of joined interface on creep strain*

The strength of superplastic interface is greatly dependent on the distance of mutual migrations at the joined interface. A theoretical consideration for the distance of grain migration  $D_{\text{mig}}$  at the interface is made in this subsection to understand the evolution of the interface strength as a function of the applied compressive strain (see Type I interface in [Fig. 5\)](#page-3-0). The distance of migration  $D_{\text{mig}}$  is defined in Fig. 7(a). Before creep joining, the height of respective blocks is  $D_0$ , being equal to  $2RN_0$  in terms of *N*<sup>0</sup> (the number of grains along the height). After creep joining subjected to a compressive strain of  $\varepsilon$ , the height of blocks was reduced from  $2D_0$  to  $2D_f(\equiv 2RN_f)$ , when there are no changes in the mean size and the shape of grains during creep joining, as is the case of superplastic deformation and flow. The compressive strain  $\varepsilon$  is defined

by

$$
-\varepsilon = \ln \frac{D_{\rm f}}{D_0} \tag{2}
$$

The number of grains  $N_f$  of the crept block at  $-\varepsilon$  [see Fig. 7(b)] is given by

$$
N_{\rm f} = N_0 e^{-\varepsilon} \tag{3}
$$

On the other hand, the total migration distance after the creep deformation is given by

$$
D_{\rm mig} = 2R(N_0 - N_{\rm f})\tag{4}
$$

Accordingly, the dependence of *D*mig on the applied strain  $\varepsilon$  is expressed in Eq. (5) by the uses of Eqs. (3) and (4),

$$
\frac{D_{\text{mig}}}{D_0} = 1 - e^{-\varepsilon} \tag{5}
$$

It is clearly seen in Eq. (5) that the distance of migration across a joined interface increases with the applied stress increased. At the applied strain of 0.04 where the interface strength meets to the inert strength (see [Fig. 5\),](#page-3-0)  $D_{\text{mig}}$  is about  $0.04D_0$ , being about 150  $\mu$ m in the present specimens used, if the interface is ideally coherent at the onset of creep joining.

#### **5. Conclusion**

The strength evolution at the interface in superplastic joining has been well understood by considering the microscopic processes and mechanisms of CGBS. It was concluded that the strength of the interface is exclusively controlled by the mutual migrations of grains across the interface through CGBS processes. The distance of migration across the interface through CGBS has been theoretically estimated as a function of the applied strains in creep joining.

#### <span id="page-5-0"></span>**Acknowledgements**

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