JOURNAL OF MATERIALS SCIENCE LETTERS 10 (1991) 1291–1292

Enhanced superplastic deformation of 2 mol % yttria-stabilized tetragonal zirconia polycrystals–alumina composite by liquid-forming additives

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A recent study has shown that the addition of a small amount (0.3–1.0 mol %) of liquid phase-forming CuO greatly enhances superplastic deformation of 2 mol % yttria-stabilized tetragonal zirconia polycrystals (2Y-TZP) [1]. The superplastic deformation in that system is accompanied by changes in the stress exponent and in the activation energy across the melting temperature of the liquid phase, corresponding to a shift in deformation mechanism from diffusion-control to interface-control [1]. In this letter we report the superplastic deformation behaviour of a 2Y-TZP–20 wt % Al_2O_3 composite (denoted as Z20A) doped with 0.7 mol % liquid-forming additives which exhibits a significantly enhanced deformability and a similar temperature-dependent characteristic to that of CuO-doped 2Y-TZP.

The starting materials were a high-purity (> 99.99%) alumina powder (TM-D, Taimei Chemical, Tokyo, Japan) and a high-purity (> 99.95%) 2Y-TZP powder (Tosoh, Tokyo, Japan). The powders were mixed (20 wt % alumina + 80 wt % 2Y-TZP) and dispersed in distilled water with a surfactant (Darvan 821A) by attrition-milling using zirconia milling media in an alumina jar. The composition of the additives was 0.3 mol % CuO + 0.3 mol % TiO_2 + 0.1 mol % B_2O_3. The equal amount of TiO_2 with CuO would form compensating dopants to alumina, which was reported to enhance greatly the deformation of alumina [3]. The additives were introduced by adding aqueous solutions of $Cu(NO_3)_2$ and H_3BO_3 and ethanol solution of Ti [O $(CH_2)_3CH_3]_4$ to the dispersed alumina/2Y-TZP suspension. The pH of the mixture was then adjusted to floc the suspension. The slurry was dried and calcined at 700 °C for 1 h. The powder thus obtained was attrition-milling dispersed again and then pressure-cast under optimal rheological condition into cakes using the same prodedure as described elsewhere [1,2]. The final dried cakes were sintered at 1250 °C for 1 h. The density of the sintered specimens as determined by a water-immersion method and further confirmed by scanning electron microscopic (SEM) examination was greater than 98% theoretical. Specimens for the deformation testing were cut from the sintered discs into square bars of aspect ratio 2.2. Deformation was conducted in uniaxial compression in air, between 1050 and 1300 °C, with strain rates ranging from 4×10^{-7} to 4×10^{-3} s^{-1}. Other experimental details were similar to those reported elsewhere [1,2].

The sintered sample features a fine-grained microstructure with alumina grains being evenly distributed in zirconia matrix as can be seen in Fig. 1a. The alumina grains appear dark compared with zirconia grains. Both have an average grain size of about 0.3 μm, which is smaller than the about 0.5 μm that usually obtained in undoped Z20A [4], and is a result of the relatively low sintering temperature enabled by the doping. The microstructure of the doped specimens remained relatively stable during deformation. However, at higher temperatures (>1250 °C) noticeable grain growth (particulary for alumina) occurred. Some abnormal alumina grains of a couple of micrometres in size, presumably grown by a dissolution–precipitation process through the liquid boundary phase, were observed within the zirconia matrix. Cavitation was negligible except at high strain rates to a large strain. Fig. 1b shows the microstructure of a specimen deformed at 1300 °C and at a strain rate of 1.2×10^{-3} s^{-1} to a true strain of −0.68, which shows as an example the abnormal alumina grains and some cavitations that could be developed under unfavourable deformation conditions.

The superplastic flow behaviour of the doped Z20A is summarized in Fig. 2. For comparison, also shown is the flow stress–strain rate relationship at 1250 °C of an undoped Z20A with an average grain size of 0.5 μm for both zirconia and alumina [4]. It can be noted that the additives greatly enhance the superplasticity of Z20A. At a given flow stress, a more than two orders of magnitude increase in the strain rate is produced. Whereas the finer grain size in the doped material could pormote the deformation by a factor of 2 to 4, the main contribution to this observed enhancement is clearly from the presence of the liquid-forming additives. In the case of 2Y-TZP, a deformation enhancement by a factor of about 100 has also been found with the doping of CuO [1].

It can be seen from Fig. 2 that the slopes (that is, the stress exponent, n) of these data lines for the doped Z20A change with temperature. It is about unity at 1050 °C, but it becomes near 2 at 1300 °C, approaching that (2.1) of the undoped Z20A. This is highlighted in Fig. 3: the value of n increases as the temperature increases, then goes through a transi-

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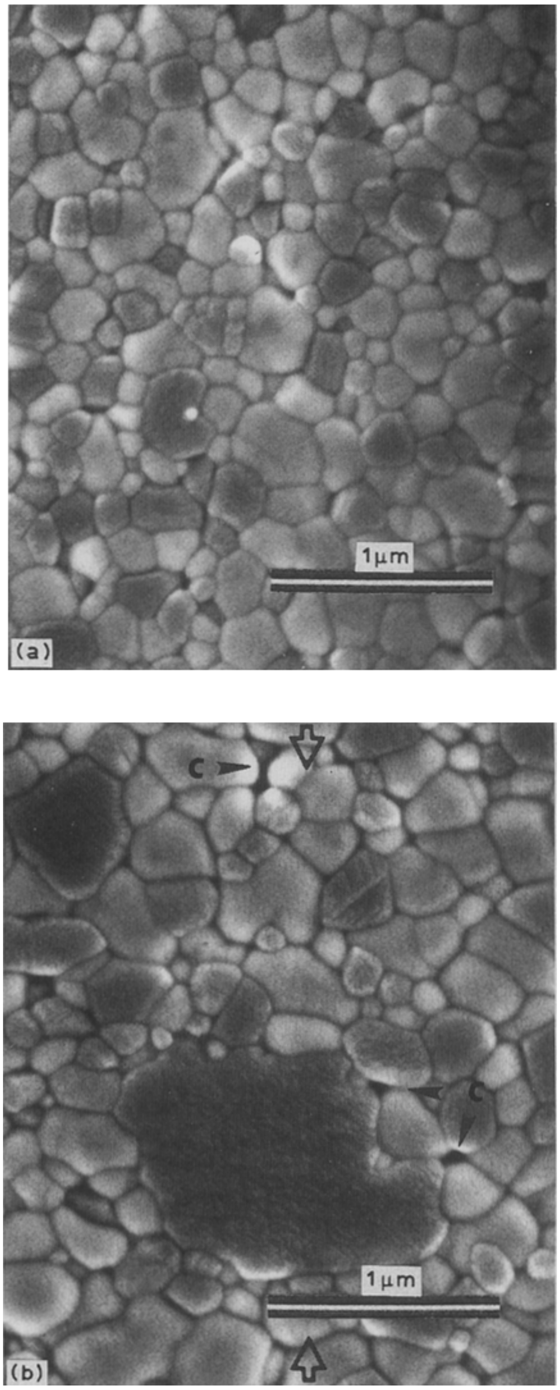

Figure 1 SEM micrographs of 2Y-TZP–20 wt % alumina doped with 0.7 mol % liquid-forming additives: (a) before deformation and (b) after deformation at 1300 °C to a true strain of − 0.68. The compression axis is shown by hollow arrows, and the cavitation sites are indicated by solid arrows.

tion and levels off at around 1200 °C. The n-value of between 1 and 2 found in the doped Z20A is relatively low compared with undoped Z20A of about 2.1, and is characteristic of superplastic flow. The temperature dependence of the deformation undergoes a corresponding transition. An activation energy of about 500 kJ mol^{-1} is obtained at the lower temperature range of 1050–1150 °C compared with that of about 420 kJ mol^{-1} at the higher temperature range of 1150–1300 °C. The values of the activation energy found for the doped Z20A are lower than that (about 600 kJ mol^{-1}) found for undoped Z20A [4].

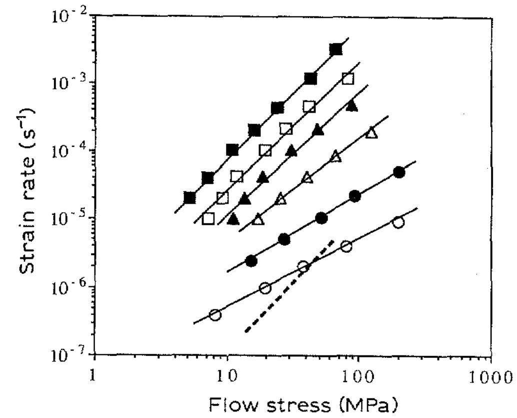

Figure 2 Flow stress–strain rate relationships for the doped Z20A at various temperatures: (○) 1050, (●) 1100, (△) 1150, (▲) 1200, (□) 1250 and (■) 1300 °C. The broken line is for an undoped Z20A at 1250 °C from [4].

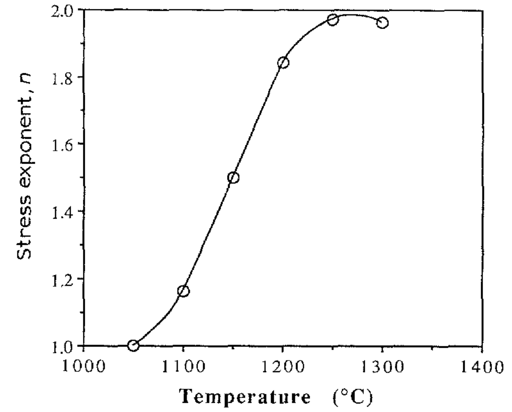

Figure 3 Temperature dependence of stress exponent, n, for the doped Z20A.

In the CuO-doped 2Y-TZP system, the increase in stress exponent and decrease in activation energy in the presence of a small amount of a liquid-forming phases has been identified to be associated with a shift of deformation mechanism from diffusion-control to interface-control. These two deformation mechanisms may also be operative in the doped Z20A in view of the similarities in the deformation characteristics between the two systems.

Acknowledgement

This research was supported by the US Army Research Office under Contract No. DAAL03-89-K0133.

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Received 12 February
and accepted 25 February 1991