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Deformation behaviour of iron-doped alumina

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Abstract

Compressive creep tests in air were carried out on 1 cat.% Fe-doped alumina at a temperature $T = 1400\,^{\circ}C$. Iron doping affected the plastic deformation by different ways in relation with Fe^{2+} cations population. Fe^{2+} cations sped up the deformation rates. $FeAl_2O_4$ spinel precipitates were identified and they were found (i) to interact with alumina grain boundaries (ii) to limit the grain growth within a range of strain. The Fe^{2+} cations underwent oxidation and this resulted in the dissolution of the some precipitates and in the decrease of deformation rates. It was suggested that deformation sped up this evolution through mass transport and that time was not a dominating parameter.

Keywords: Al_2O_3; Doping; Creep; Grain size; Grain boundaries

1. Introduction

Useful information can be generated on the nature and on the consequences of ion transport characteristics in alumina and on potential deformation capabilities by studying the effects of substitution impurities on the diffusion creep. The sintering and the creep of transition metals doped alumina have been studied before in terms of defect structure and diffusional processes.[1–7] It has been established that Fe atoms in substitution solid solution change the diffusion properties of alumina.[2,3,5] These effects are related to an enhancement of the cation lattice diffusion (via vacancies and interstitials) with rapid diffusion of oxygen in the grain boundaries.[3] The diffusion changes are associated to the presence of Fe^{2+} cations, which have a relative low solubility in alumina[5,8] whereas Fe^{3+} cations present a large solubility in alumina[9] but have only little influence on the species diffusion properties.[3]

Here, we report the plastic deformation in air of Fe-doped polycrystalline alumina, for a doping level of 1 cat.%, at $T = 1400\,^{\circ}C$ and under different stresses. The plastic deformation presented systematically two sequences: the first one corresponding to a high creep strain rate and the second one following an important decrease of the strain rate. The strain rate change occurred always in the narrow range of compressive true strains [−13%, −17%]. The alteration of strain rate and the grain size evolution have been analysed in terms of porosity, Fe valence evolution, iron aluminate spinel particles precipitation and dissolution.

2. Experimental procedure

The iron-doped alumina was prepared from a high-purity α-alumina powder (SM8, Baikowski, Annecy, France) for which the chemical impurity levels are listed in Table 1. The mean particle size is 0.30 μm.

The powder was mixed with deionised water with the object of obtaining a stable suspension. In order to achieve a homogeneous distribution of iron, aliquots of a high purity $Fe(NO_3)_3{\cdot}6H_2O$ solution were added to the aqueous slurry of alumina powder to yield Fe cation doping level of 1 cat.%. This cation doping level was thought to be large enough to be dominant over the impurity content of the processed powder as listed in Table 2.

The powder was freeze–dried before being calcined in air at $T = 800\,^{\circ}C$ for 2 h to remove nitrogen and possible carbon contaminant. Hot-pressing the calcined powder in vacuum, in a graphite die, for 30 min at 50 MPa produced material discs. The dwelling temperature was 1350 °C. The

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Table 1
Chemical impurity content of the Baikowski alumina powder

	Na	Si	K	Ca	Cr	Fe
Wt.-ppm	17	38	38	8	4	10

Table 2
Chemical impurity content of the processed alumina powder

	Na	Mg	Si	K	Ca
Wt.-ppm	20	300	100	40	1000

obtained alumina discs had a diameter of 30 mm and a height of 8 mm. The density was measured using the Archimedes' method with methanol as the immersion medium.

Deformation samples were cut from the discs and they had a typical height of 7 mm and a square cross section of $3 \times 3\,\text{mm}^2$. The height was parallel to the hot-pressing axis. Compressive creep tests were carried out in air at $T = 1400\,^{\circ}\text{C}$ and in the true stress range 50–100 MPa. From length change versus time curves, true strain rate versus true strain curves were obtained. A sample was annealed 1200 s at $T = 1400\,^{\circ}\text{C}$, prior to creep testing, to assess the time effect on the evolution of Fe populations and on deformation behaviour.

Microstructure of the different samples were revealed by thermal etching in air at $T = 1400\,^{\circ}\text{C}$ for 1.5 h. The grain sizes (d) were determined, using scanning electron micrograph (SEM) with at least 300 grains counted for each sample, by the relation ($d = 1.38 \times A^{1/2}$),[10] where A is the corrected mean grain surface. The evolution of the grain size during creep under a true stress $\sigma = 75$ MPa was investigated. Reference samples were systematically positioned next to the creeping specimens. This procedure allowed us to observe by SEM the microstructure evolution in a free-strain condition in order to assess the static grain growth. This evolution was compared to the one obtained for the strained specimen, for which both static and dynamic grain growths were active.

Analytical transmission electron microscopy (ATEM, Philips CM30 equipped with an energy dispersive spectroscopy (EDS) Noran X-ray microanalysis) investigations were performed on specimens, which were hot-pressed or crept at different true strains under $\sigma = 75$ MPa.

3. Experimental results

3.1. Compressive creep behaviour

The effect of the compressive true stress on true strain rate behaviour is presented in Fig. 1. Samples exhibited specific deformation behaviour, i.e., a quasi steady state creep was reached during a first stage up to a compressive true strain in the range $\varepsilon = [-13\%, -17\%]$ then, the true strain rate decreased strongly and a second creep stage was observed. Note that the compressive true strain range $\varepsilon = [-13\%, -17\%]$ corresponded to quite different durations according to the applied true stress. Under the true stress $\sigma = 75$ MPa, that time was in a range t (500 s, 1000 s).

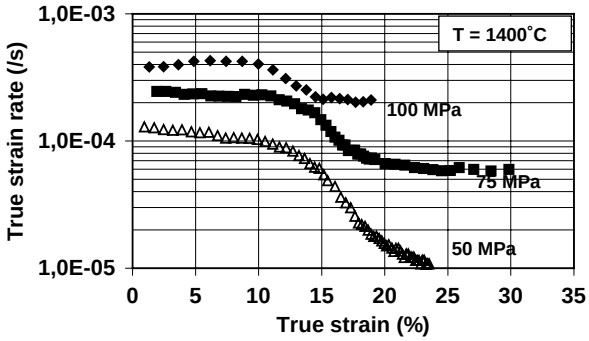

Fig. 1. True strain rate vs. true strain under different true stresses for a 1 cat.% Fe-doped alumina.

For the annealed sample, the annealing time (1200 s) was a bit more than the one corresponding to the time observed to reach the decrease of strain rate in the true strain range $\varepsilon = [-13\%, -17\%]$. No significant difference was observed in its deformation behaviour. The corresponding strain rates were similar and the strain rate belonged to the same true strain range.

3.2. Microstructural features

The hot-pressed materials were densified to more than 98.8% relative density. Fig. 2 presents the microstructure of the hot-pressed materials observed by SEM. Microstructure was homogeneous and the mean grain sizes was 1.6 μm.

Fig. 3 shows the evolution of the mean grain size versus strain. After a short period of grain growth observed at the beginning of the deformation, the grain size remained constant up to about a strain corresponding to the second creep stage. Then the grain grew on. The sample had final

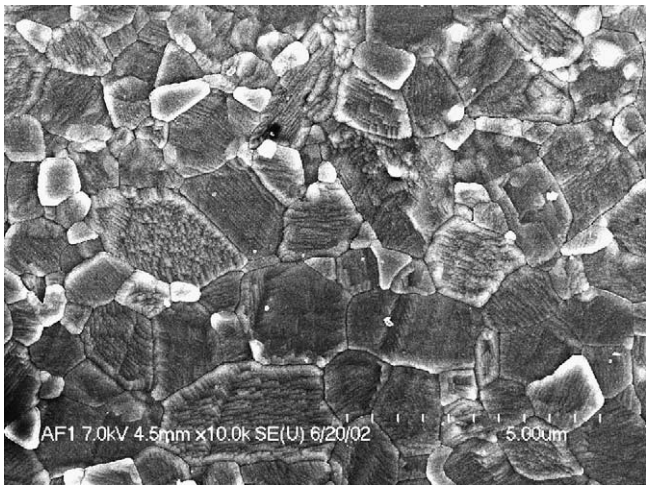

Fig. 2. Microstructure of the as hot-pressed material.