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Direct observation of grain rearrangement during superplastic creep of a fine-grained zirconia

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Abstract

A 2 mol% yttria partially stabilised zirconia specimen has been deformed at 1300 °C under a constant stress of 100 MPa by strain increments of 14% up to an ultimate true strain of −0.56. At each stage of creep test, grain arrangement inside two zones of a side face of specimen was investigated by scanning electron microscopy. Grain rearrangement proceeded essentially by two elementary transformations: the neighbour switching event and the grain disappearance corresponding to grain movement perpendicularly to the analysed surface. These observations highlighted unambiguously that grain boundary sliding was not simply an accommodation mechanism but acted as the deformation mechanism, excluding that a pure diffusion creep may account for deformation. Influence of cooperative grain boundary sliding was very limited. Grain redistribution analysis showed that, on average, each grain was implied in four neighbour exchange events during test and that topological stability was nearly reached after a strain of 28%.

Keywords: Creep; Grain boundary sliding; Scanning electron microscopy; Superplasticity; ZrO_2

1. Introduction

Superplastic deformation of zirconia ceramics has been widely studied from the mechanical point of view, which allowed analysis of strain rate in terms of various parameters like stress, temperature or grain size.[1–3] From the microstructure point of view, it is generally accepted, but not consensual[4,5] that achievement of large strains retaining a quasi equilibrium structure, as those induced by a superplastic flow, mainly results from grain boundary sliding (GBS).[1,2,6,7] On the other hand, the origin of intragranular deformation, requisite for accommodation of GBS, is not yet clearly elucidated.[5,7,8] As a deformation process, grain boundary sliding may concern either individual grains or on the contrary rigid groups of grains behaving like entities, called then cooperative grain boundary sliding (CGBS).[9]

Experimental evidences supporting the role of CGBS have been reported by several authors. Zelin et al.[10] and Zelin and Mukherjee[11] studied superplastic deformation of two metallic alloys: a Mg alloy and a Pb-Sn eutectic respectively. In each case these authors observed surfaces of strain localisation, separating less deformed zones. These features were explained from the viewpoint of CGBS associated with rotation of entire grain groups. Chen and Huang[12] have equally noted presence of CGBS during superplastic forming of a 8090 Al alloy. In these experiments, the size of grain groups at the origin of cooperative sliding never exceeded a few grain sizes.

In fine-grained ceramic materials, the respective role of GBS and CGBS is not really known. In a recent work Muto et al.[13,14] investigated the tensile deformation of a 3 mol% yttria stabilized zirconia up to a strain of 0.75. The periodical surface corrugation observed after deformation was analysed as the result of a cooperative sliding mechanism, acting at the scale of the entire specimen,[15] able to explain a large part of the macroscopic strain. That conclusion was partly deduced from the observation of no changes in size and shape of individual grains implying, according to these authors, the insignificance of diffusion mechanisms. Nevertheless, in their experiments, authors did not investigate deformation at

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the mesoscale and consequently the role of grain boundary sliding of individual grains or small groups of grains was not determined.

The above conclusion is somewhat surprising since measurements of grain boundary sliding in alumina,[16,17] zirconia,[18] alumina-zirconia[19] or spinel–zirconia[20] ceramics have shown that boundary sliding of individual grains was able to explain about 70% of the macroscopic deformation. Besides, in a recent work devoted to grain rearrangement during deformation of an alumina-zirconia composite[21] it has been demonstrated that deformation of small zones (about 20×20 μm) was homogeneous, independent of their location in the specimen and nearly homothetic to that of the macroscopic sample. If cooperative sliding of groups of grains was not absent, such groups were rare and in any case CGBS was able to account for the main part of deformation.

This paper has been motivated by the need to clarify the various stages of grain rearrangement in a superplastically deformed yttria partially stabilized zirconia in order to precise (1) the exact role of grain boundary sliding in deformation and the possible contribution of CGBS and (2) consequences on deformation mechanisms.

With this object, the grain rearrangement inside two areas on a side face of a fine-grained zirconia specimen has been followed throughout a superplastic deformation test by scanning electron microscopy. This kind of investigation presents the undeniable advantage to directly visualise structure transformations in a quasi-continuous way, which avoid usual speculations when different zones are compared prior to and after deformation.

2. Experimental

A 2 mol% yttria stabilized zirconia was used in this work. It corresponds to material TS2/2 used by Nauer and Carry.[22,23] No amorphous intergranular phase was present owing to a low impurity content of less than 250 ppm (mainly Na_2O). Disks were sintered at 1400 °C for 3 h,[22,23] conditions entailing a grain size of about 0.35 μm. The need to examine the same zones at various strains led us to select a material with a slightly larger grain size ($d_0 = 0.7$ μm) obtained by annealing at 1500 °C. Fig. 1 shows the initial microstructure; grains are about equiaxed, an aspect ratio of 0.93 ($d_{0//}/d_{0\perp}$) being noted. This grain size is still consistent with the observation of a superplastic behaviour in this material and with grain boundary sliding as the main deformation process. The creep properties of this material have been already studied in the stress range 2.5–140 MPa, mainly at 1350 °C.[22–24] Schematically, it was shown that matter diffusion is the accommodation mechanism of grain boundary sliding, an activation energy of about 420 kJ mol^{-1} being measured above 40 MPa.

A sample of this material (2.7×2.7×6 mm) was crept at 1300 °C under a constant compression stress of 100 MPa. These conditions lead to a creep rate of 7×10^{-5} s^{-1} and a stress exponent in the range 1.5–2. The ultimate true strain was −0.56. At this strain the grain surface began to be rounded and consequently information obtained above this level could be invalid. Strain increments of 14% were performed between each observation series. This increment value is adequate to promote noticeable modifications of the grain arrangement without too large perturbations able to impede comparison between two consecutive observations, the lack of markers increasing location difficulties of investigated zones. These changes were imaged by scanning electron microscopy (SEM).

Prior to creep test, a side face of the specimen was diamond polished and boundaries were thermally etched. Deposition of a thin carbon film prevents from electrical charge during SEM analysis. Such a carbon film allowed us to recover a clean surface before atomic force microscopy experiments used to investigate the surface topography. Two zones (15×20 μm), in the central part of the side face, were examined from 0 to 56%.

From SEM images, two kinds of results were obtained: (1) qualitative ones showing the different ways in which grains move to induce the macroscopic shape change of the specimen and (2) more quantitative results concerned with the surface topology; these inform about the mean neighbour distribution and the more or less extensive grain rearrangement.

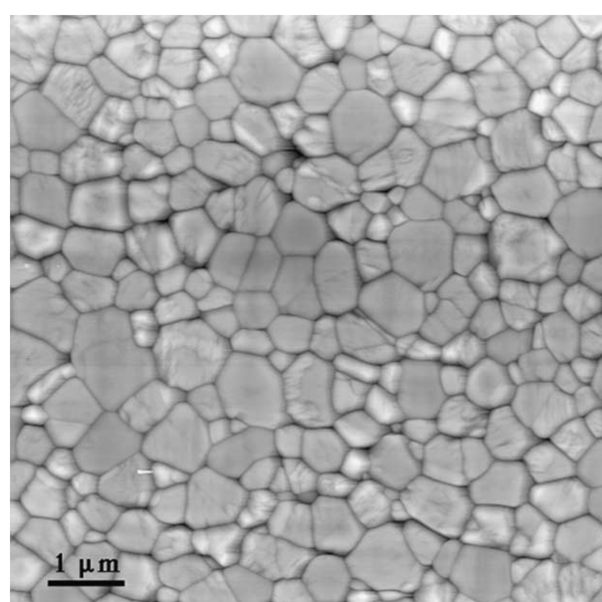

Fig. 1. Atomic force micrograph of typical microstructure of as sintered material. The compression axis is vertical.

3. Results

At the macroscale, the specimen surface kept a uniform aspect and no detail characteristic of a non-uniformity of deformation was noted. After the ultimate strain, a weak grain growth was noted ($d_f = 0.74$ μm) and the new aspect ratio ($d_{f//}/d_{f\perp}$) was 0.83. Such a change in the aspect ratio would imply an intragranular strain ϵ_g of about 7 to 8% [by using $\epsilon_g = (2/3)\ \ln\ (d_{//}/d_{\perp})$).[25] In each examined area, some recognizable grains were chosen and their position relative to a grain taken as origin was recorded for the five experimented strains. Fig. 2 shows some grain trajectories for the two selected areas. Grains move away from the ordinate axis, the compression axis, and results are independent of the examined area. In this figure, full lines represent followed trajectories when deformation of a given volume is assumed to be homothetic to deformation of the whole specimen. They correspond to relation:

$$y = y_0(x_0/x)^2 \tag{1}$$

in which x_0 and y_0 are the coordinates of a grain prior to creep, y/y_0-1 being equal to ϵ_0, the nominal strain.

At the mesoscale, the attention was focused on transformations implied in the achievement of high elongations. Two transformations, called afterwards topological events, were noticed: the neighbour switching and the grain disappearance.

An example of the neighbour switching mechanism is presented in Fig. 3 that shows the successive steps of the process. Two small grains, 2 and 4, in a configuration parallel to the compression axis and initially separated by two larger grains, 1 and 3, became nearer and nearer and finally separated grains 1 and 3 that moved perpendicularly to the compression axis. Each grain, in the surface plane, kept about the same area. One may note the good similarity between the different sequences of experimental switching event (Fig. 3) and those proposed by Ashby and Verrall[26] for this transformation. Fig. 4 is another example in which several switching events took place simultaneously in a small group of grains, some grains being able at the same time to be involved in three intercalation processes. For instance, grains 4 and 6, initially separated from grains 1 and 9 and from grains 2 and 11 respectively, moved towards those grains up to the contact, while they moved apart from grains 5 and 7 respectively.

Concerning grain disappearances, numerous ones have been observed. One of them is presented in Fig. 5. It corresponds to migration of grain 4 towards the specimen interior and does not result from absorption of the grain by adjacent grains, owing to grain growth. This grain disappearance may be analysed according to schemas proposed by Gifkins[27] or Langdon,[28] or also as a switching event arising in a plane perpendicular to the

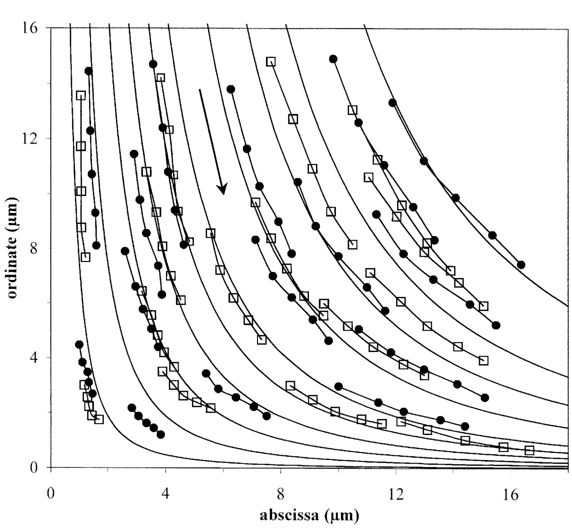

Fig. 2. Trajectories of some grains inside the two areas investigated between 0 and 56% (full circles: zone 1; open squares: zone 2). The compression axis is vertical. Arrow indicates displacement direction. Full lines represent trajectories when a homogeneous deformation is assumed.

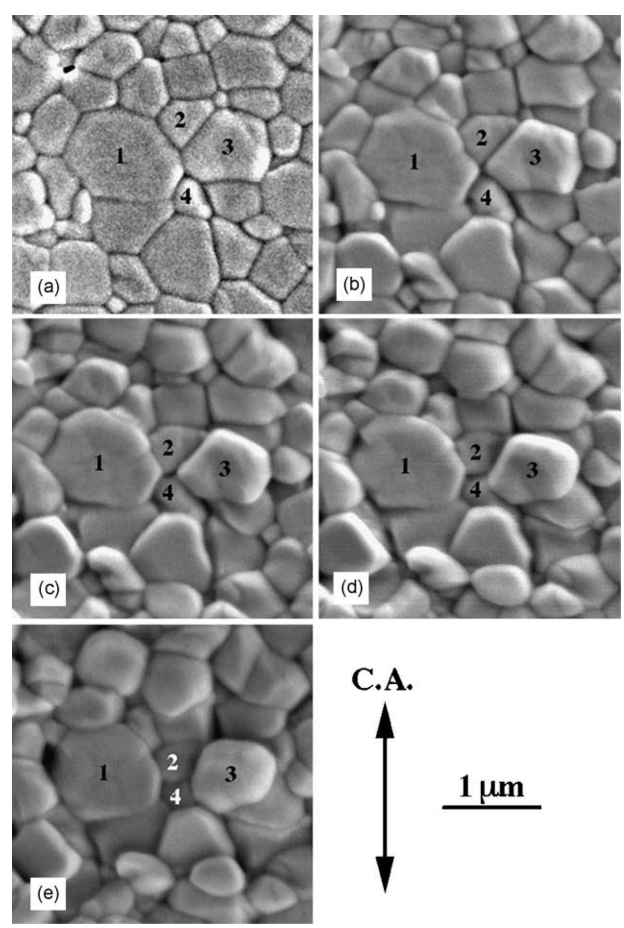

Fig. 3. Neighbour switching event in grain group 1–4; (a) 0%; (b) 14%; (c) 28%; (d) 42%; (e) 56%; 1 μm scale bar, vertical compression axis.

specimen surface since grain 1 and grains 6 and 7 come in contact after the ultimate strain at 56%.

From SEM images, the neighbour distribution of more than 600 grains has been analysed prior to creep and after strains of 28 and 56% and compared. The objective of this topological investigation is to provide quantitative information about the relative movement of grains and not only a subjective one. Distribution functions of the number of grain sides are shown in Fig. 6. Unless the maximum of distributions shifts from 6, in the non-deformed state, to 5, after strains of 28 and 56%, the average grain side number is nearly insensitive to strain, only slightly decreasing from 5.85 (0%) to 5.76 (28%) and 5.73 (56%). In contrast, neighbours of a given grain may change considerably in the strain range 0–56% since, in certain cases, 70–80% of nearest neighbours have been modified. Thus, in average, among the six neighbour grains present prior to creep, only five and then four are still present after true strains of 28 and 56% respectively. Fig. 7 is one of the rare examples presenting a grain that retained the same nearest neighbours during the test. Even in that case, deformation inside the grain group is clearly discernable.

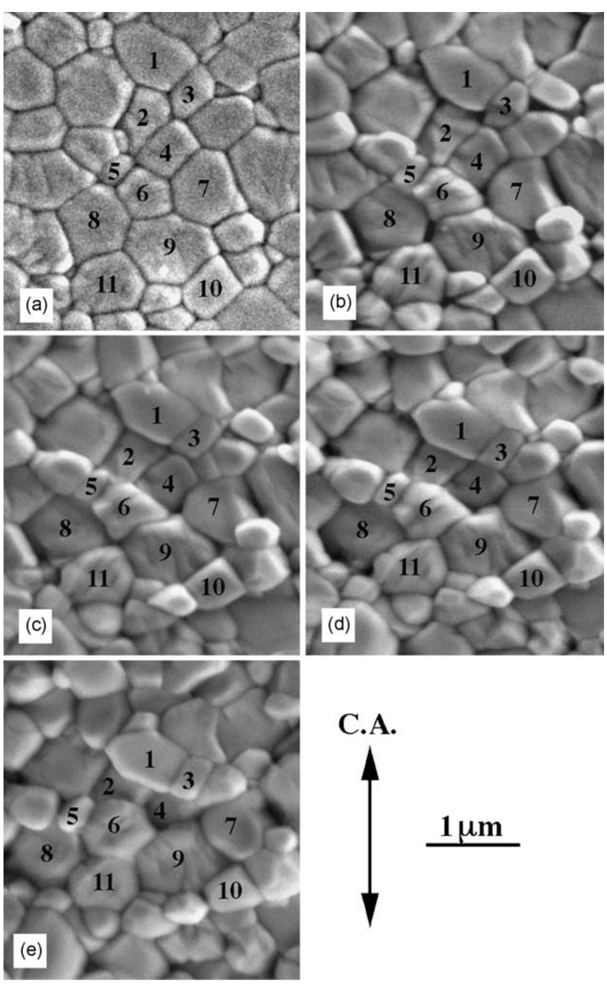

Fig. 4. Multiple neighbour switching events in grain group 1–12; (a) 0%; (b) 14%; (c) 28%; (d) 42%; (e) 56%; 1 μm scale bar, vertical compression axis.

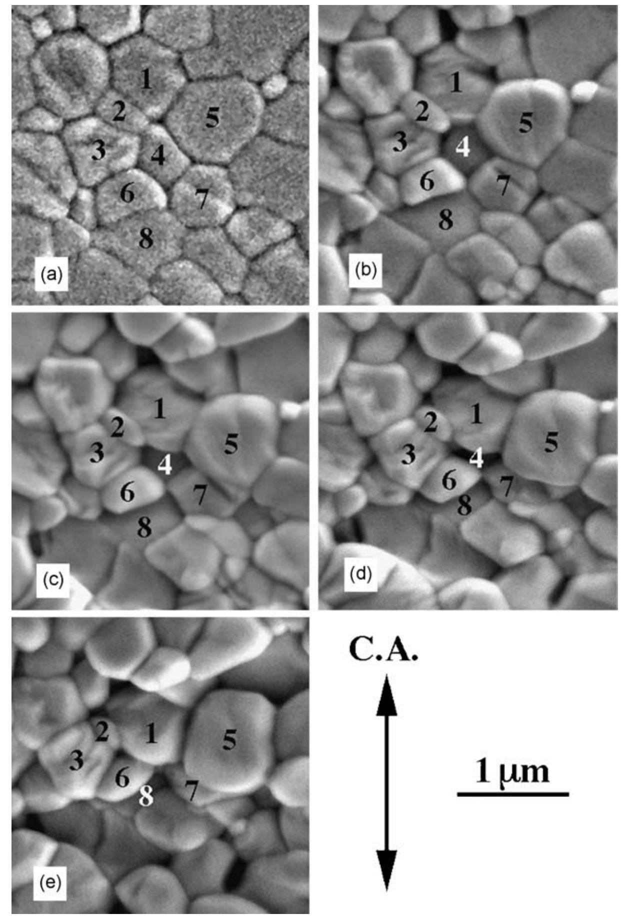

Fig. 5. Progressive disappearance of grain 4 from grain group 1–8. It can be analysed as a switching event in a plane perpendicular to the observation surface, entailing that grains 1, 6 and 7 became close to each other at strain of 56%; (a) 0%; (b) 14%; (c) 28%; (d) 42%; (e) 56%; 1 μm scale bar, vertical compression axis.

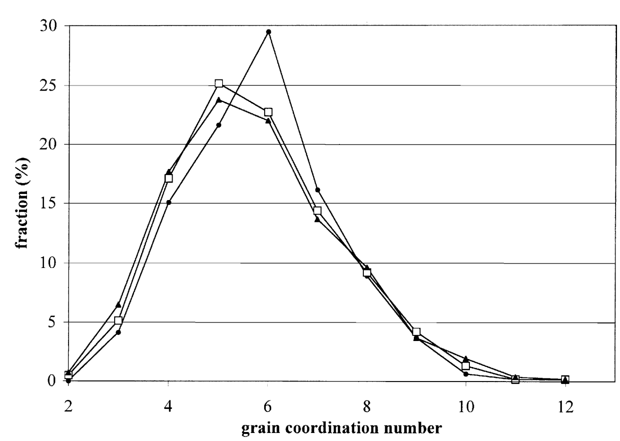

Fig. 6. Distribution functions of grain coordination numbers at strains of 0, 28 and 56%.

To complete the surface analysis and obtain information in the perpendicular direction to the face, topography has been analysed by atomic force microscopy in the central part of specimen. Surface profiles have been recorded on a distance of 150 μm in a parallel direction to the compression axis. It emerges from this investigation that image depth and roughness regularly increased with strain (table), without any coarse change as those reported by Muto et al.[14] Data in Table 1 do not strongly depend upon the analysed area when its size exceeds 5×5 μm.

4. Discussion

At first, information obtained from above results directly concerns grain rearrangement occurring in fine-grained ceramics during a superplastic deformation test.

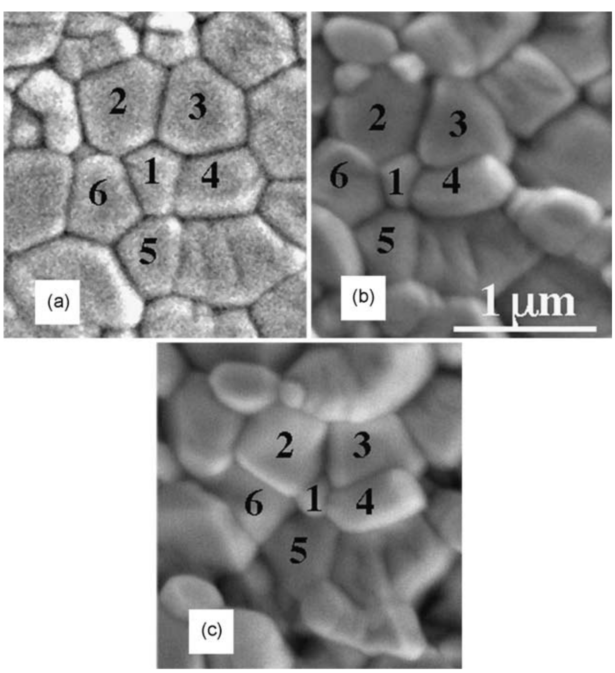

Fig. 7. A rare observation of a grain retaining the same neighbours throughout deformation; (a) 0%; (b) 28%; (c) 56%; 1 μm scale bar, vertical compression axis.

Table 1
Relief amplitude (perpendicular to the analysed face) and surface roughness as a function of true strain

True strain (%)	Relief amplitude (nm)	Roughness Ra (nm)
0	153±29	7.2±1
14	338±42	32.6±2
28	516±68	57.6±4.3
42	719±72	80.4±4.8
56	869±94	96±5

Subsequently, a number of consequences, related in particular to deformation mechanisms, may be drawn.

At present, grain growth being nearly absent, grain rearrangement proceeded by the neighbour switching mechanism. All micrograph series in Figs. 4–6 well illustrate the way in which deformation proceeded. Grains are not rigid and their three-dimensional movement makes easier deformation continuation after a switching event occurred.

Switching process may arise either in the observed surface plane or in a perpendicular one. This last case leads to grain disappearance inside specimen. This is a requisite condition since, during a compression deformation test, the area S of a side face decreases as:

$$S = S_0(1 + \varepsilon_0)^{1/2} \tag{2}$$

S_0 being the initial area of the face. Therefore, matter must leave external surfaces to penetrate specimen volume and increase the cross-section area.

The neighbour switching mechanism was especially active and led to pronounced grain redistribution but not necessarily to a similar topological change.

The first aspect may be described by the surrounding grain stability, i.e. by the number of grains remaining in nearest neighbour position during the test. The continuous decrease from 6 to 4, when strain increased from 0 to 56%, implies that on average any grain was involved in two switching events ending with a grain separation or a grain disappearance and two other ones leading to a contact (see grain 6 in Fig. 2), since each grain retained about six neighbouring grains throughout deformation.

The second aspect may be depicted by considering distribution functions of the grain coordination numbers. If distribution functions slightly evolve in the strain range 0–28%, the maximum of distribution shifting from 6 neighbours to 5, they are very similar at 28 and 56%. This evolution is correlated to a change in the

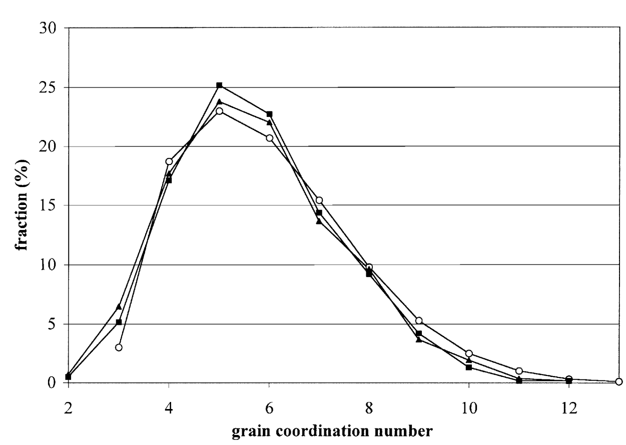

Fig. 8. Comparison of distribution functions of grain coordination numbers at strains of 28 and 56% with the theoretical one for a defect concentration of 0.69 after Carnal and Mocellin.[31].

topological defect concentration[29] C evaluated from f_n, the fraction of grains having n sides, by:

$$C = \frac{1}{2}\sum |n\text{-}6| f_n \quad (3)$$

Defect concentration increases from 0.57, prior to creep, to 0.65 and 0.68 after strains of 28 and 56% respectively, corresponding to an evolution toward a greater topological stability. Changes between 0 and 28% attest the need to produce an adequate amount of topological defects at the basis of the grain switching process, mechanism by which macroscopic strain occurs.

Models dealing with topological stability of plane sections of polycrystals, and concerning processes like grain growth or deformation, have been proposed by Blanc and Mocellin[30] and Carnal and Mocellin.[31] These models describe distribution of grain coordination numbers under the assumption that only two elementary transformations take place: the grain disappearance or appearance and the neighbour switching event. In addition, distributions were assumed to correspond to a steady state characterising a topological stability. When the two elementary events are allowed to occur simultaneously, invariance of distributions requires that a fixed ratio be maintained between the two kinds of events. Thus, for any disappearing grain, there should be about four neighbour interchanges.[31]

In the present case, as pointed out earlier, after a strain of 56% each grain retaining four of the six initial neighbours, this means that in average a given grain is implied in four neighbour switching events. As each switching event implies four grains in the observation plane, the total number of neighbour exchanges occurred during the test is equal to the grain number. During the same period, Eq. (2) shows that the area of a lateral face decreased of 25%, leading to the disappearance of one out of four grains. This is in agreement with above inferences.[31]

Fig. 8 compares experimental distribution curves at 28 and 56% with a theoretical one obtained by Carnal and Mocellin for the above assumptions and a defect concentration of 0.69.[31] The concordance is rather good, which suggests that after a strain of 28% a topological steady state was likely reached.

As noted previously, groups of grains behaving as rigid entities have not been observed. At best, groups of grains inside which no grain disappearance or neighbour switching took place were present. They generally contain a few grains and deformation in these groups is discernable (Fig. 7). In close correlation with these observations, AFM study did not reveal the presence of relief or asperities able to suggest that large parts of the specimen have slid on macroscopic shear surfaces. From roughness measurements, a value of GBS contribution to total strain has been estimated. This estimation refers to similarities found between GBS amplitude and roughness in a previous paper[17] and to the ratio of 1.43 noted between these two parameters for a zirconia with a grain size of 1 μm.[18] From values in Table 1, a GBS contribution to total strain of about 80% is obtained. This should be confirmed by direct measurement of average sliding amplitude. Under these conditions, the role of cooperative grain boundary sliding in the deformation process of the present zirconia appears to be minor contrary to conclusions of Muto et al for a similar material.

The main differences between this investigation and that of Muto et al.[14] reside in (1) the deformation mode, compressive test versus tensile test, and (2) the specimen size. These two differences act in the same way. The larger the specimen size, the larger the probability to find surface defects whose influence on macroscopic deformation is more sensitive during a tensile test than a compressive one. Nevertheless, in Muto et al.'s work no attempt was made to consider if corrugation height and density were able to account quantitatively for the main part of deformation and the influence of grain boundary sliding at the level of individual grains remains to be examined.

The last consequence of this investigation concerns the deformation mechanism. Even if grain trajectories are in accordance with those expected from a pure diffusion creep, in which shape change of any volume is similar to that of the macroscopic specimen, investigation of the same areas at different strains has shown that grain boundary sliding is present as a deformation mechanism, the neighbour switching event contributing to the maintenance of a nearly equiaxed grain configuration, but entailing simultaneously important alteration in the distribution of nearest neighbouring grains. Such observations are in conflict with structure evolutions ensuing from Nabarro-Herring or Coble diffusion creeps that predict a change shape of grains reflecting the overall strain and no change in grain environment in the absence of grain growth. Consequently, a pure diffusion creep is not able by itself to account for deformation of such a fine-grained ceramic, even if creep rates of some zirconia samples[6] have been theoretically reproduced within one order of magnitude by a Coble diffusion creep model.[4] In this work, authors bypassed the apparent discrepancy between the measured grain aspect ratios and the total strains of specimens by resorting to boundary migration in order to minimise boundary energy, which would maintain an equiaxed grain structure.

In the same way, if the possibility of accounting for neighbour switching by grain growth[32] cannot be totally excluded, it has not been noticed. The entire observed neighbour switching events produced grain rearrangements inducing local deformation consistent with the macroscopic strain. This would not systematically be the case if grain growth induced such neighbour exchanges since this would suppose that grain growth arises on certain grains in particular configurations, which seems unlikely.

5. Conclusion

A 2 mol% yttria partially stabilised zirconia specimen has been crept at 1300 °C under a constant compressive stress of 100 MPa up to an ultimate true strain of −0.56. Every 14% of true strain, the test was stopped and two same zones of a side face of the specimen were investigated by SEM and their grain arrangement compared in order to describe unambiguously modifications at the origin of macroscopic deformation. This method presents an undeniable advantage, compared to the observation of different zones prior to and after creep, since it shows transformation kinetics and thus allows a better understanding of deformation mechanisms. It is the first time that such an investigation is reported in a so large strain range for a fine-grained single-phase ceramic.

Two elementary transformations at the origin of grain rearrangement were noted: the neighbour switching event and the disappearance of grains by migration inside specimen, which is similar to a switching event occurring in a perpendicular plane to the analysed surface. These transformations maintained a nearly equiaxed grain configuration. In agreement with AFM measurements of roughness, that allowed us to estimate GBS contribution to total strain to about 80%, grain boundary sliding acted as the deformation source and not simply as an accommodation mechanism.

From the topology viewpoint, characterised by distribution functions of grain coordination numbers, structure slightly evolved from 0 to 28%. At this strain, a topological steady state seemed to be reached corresponding to nearly stable distribution functions. Nevertheless, this stability implies that on average each grain was involved between 0 and 56% in four neighbour exchange events, which significantly modified nearest neighbours of a given grain. These remarks induce that a pure Coble diffusion creep is not able to account for deformation of the zirconia in the conditions of the present work.

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