Mapping of the granular flow during superplastic deformation of microsample of Zn-20.2%AI - 1.8%Cu alloy at room temperature

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A new technique for Scanning Electron Microscopy (SEM), which provides a mesoscopic coordinate system inscribed on the surface of the center of a tension test specimen has been proposed. Such technique allows to establish in a repeatable way any angle relative to any axis of any coordinate system, or distances between grains, or to measure local or global true deformation in parallel or perpendicular directions relatives to the tension axis. The application of such technique for a Zn-20.2%Al - 1.8% Cu alloy tension test specimen with a total length between shoulders of 412 μ m length and a gage length of 371 μ m, deformed at room temperature, allows to obtain: The mapping of the granular flow during superplastic deformation; this result is in qualitative agreement with similar flow-diagrams obtained previously by Ashby and Verral (*Acta Metall.* **21**, 1973, p. 149) from their oil-emulsion model. © *2001 Kluwer Academic Publishers*

1. Introduction

Grain boundary sliding, GBS, between individuals grains which move relative to each other with little or no change in shape is an important mechanism of high temperature deformation, in particular for superplastic flow [1, 2].

Some open problems in, superplasticity, as the experimental determination of diagrams of granular flow during deformation require the development of new techniques which allow us to study the plastic deformation at three different microstructural levels: i.e., macroscopic (the entire deformated volume); mesoscopic (at the level of cooperative movement of grain groups) and the microscopic level (to study the grain boundry sliding between individual grains) [3].

The present paper deals with the development of a new technique (to be used in SEM) which provides the possibility of quantitative measurements about the superplastic flow at the three different microstructural levels before described. The new techniques provides a mesoscopic coordinate system inscribed on the surface of the center of the samples, in order to be capable to put a pair of sets of numbers that determines the location of any material point of interest on the surface of the sample under plastic deformation.

The technique schematically described was applied to the case of structural superplastic Zn-20.2%Al-1.8%Cu alloy (Zinalco) deformated at room temperature in order to obtain a diagram of granular flow for such alloy.

2. Experimental procedure

The material used for this investigation Zinalco was extruded to obtain strips of 20 mm wide and 5 mm thick. The extruded plates of Zinalco alloy were cold rolled into strips of 1 mm thickness. Tension test sample with

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total length between shoulders of $(412 \pm 4) \mu m$ (a gage length of 371 μm) parallel to the rolling direction were prepared. The geometry and size of specimen designed for tension test in SEM are shown in the microphotography given in Fig. 1a, the mean linear intercept was 3.5 μm . The specimen was mechanically polished on successively finer grades of emery paper and diamond paste.

By using of a pyramidal-shaped micro vickers indenter three sets of diamond pyramidal figures were inscribed on the surface of the specimen along on a longitudinal straight line as follow one pyramidal-shaped figure was inscribed approximately at the center of the specimen and a regular trapezoid delimited by pyramidal indention figures in each corner of such figure was inscribed on the surface of the tension sample. Centered on each trapezoid another pyramidal indentation was inscribed see Fig. 1b. The gage length size of the tension specimen is delimited by the two sets formed each by five diamond pyramidal figures. This length has been used for the determination of the macroscopical deformation.

The experiments were performed at constant cross head velocity, v = 0.1 mm/min, giving a nominal macroscopic initial strain rate of 4.04×10^{-3} s⁻¹. Before deformation SEM micrographs were taken from the sample. After each loading step, that provides a sample elongation (between shoulders) of 0.2 mm or 0.1 mm, the deformation was stopped and pictures were taken from the sample under different magnifications.

3. Experimental results

During deformation of a sample at constant crosshead velocity one end of the sample is fixed at rest in Laboratory, and the other end have the velocity of the mobile crosshead (v_c).

It is clear that the microscopical condition for homogeneous deformation during this type of test is given by:

$$v_{x_{\text{Lab}}}(x) = v_{\text{c}}\left(\frac{x}{L_0}\right) \tag{1}$$

where x is the distance between the end fixed at rest in Laboratory and the point of interest at the sample, L_0 is the initial gage length of the sample, and $v_{x \text{ Lab}}$ is the velocity of any material point on the surface of the sample at x.

Fig. 2 illustrate the experimental data for velocity of specific material objects choosed from Fig. 1b as a function of distance along x-axis as measured from coordinate system at rest in laboratory. It is clear from Fig. 2, that experimental data obey a linear relationship between velocity v_x and the x-distance.

Provided the deformation of the sample is mesoscopically homogeneous, we can build up a quantitative flow diagram for specific points of the specimen through deformation. Fig. 3a shows a quantitative flow map of velocities for specific point on the material through a true deformation of 24.0% (measured between the two sets formed each by five diamond pyramidal figures). The starting point in each arrow set is for denoting the original coordinates of the material before deformation taking as the initial microstructure which is given by the coordinate system depicted on Fig. 1b.

Also a quantitative flow-diagram of the strain at the surface of the deforming sample can be build up. The true deformation along the tension axis, at any point on the surface of the sample, can be determined by using the following equation

$$\varepsilon_x = \ln\left(\frac{L_x(t-t_0)}{L_x(t_0)}\right).$$
(2)

where it has been used that $L_x(t_0)$ is the *x*-distance of some material point of interest at time t_0 , and $L_x(t - t_0)$ is the *x*-distance of the same material object at the time $t - t_0$ during deformation at constant crosshead speed, or by using the equivalent equation too:

$$\varepsilon_x = \ln \left[1 + \frac{v_x(x)\Delta t}{L_x(t_0)} \right]. \tag{3}$$

A similar equation can be used for deformation along *y*-axis.

Fig. 4 shows the quantitative diagram of the strain flow at the surface of the deforming sample. For any point, the first deformation arrow occurs in 120 seconds and the second and third arrows occurs in 60 seconds each. Horizontal deformation, ε_x was calculated by using Equation 2 with $L_x(t_0)$ as the initial *x*-position of the choosed point on the surface of the sample before certain elongation occurs and $L_x(t - t_0)$ is the actual *x*-position of the material point after elongation. The *x* and *y* positions of any one of the 38 material points were carefull measured in order to build up Fig. 4. The flow of deformation follows the general trend of the flow of matter as suggested by Ashby and Verral [4], this was an expected result.

4. Analysis of experimental data

From ground principles of symmetry applied to plastic deformation, it can be expected that deformation in any of the four regions delimited by x and y axis are equivalent. Therefore it is possible to build up the Fig. 3b where the experimental data from Fig. 3a has been replotted. Also in Fig. 3b the idealized curves which are suggested by the experimental data are shown. The idealized curves are of hyperbolic nature; the experimental data can be described by,

$$y = \frac{a + (652.4 \pm 15.2)(\mu \text{m})^2}{x}.$$
 (4)

Where *a* is any asymptotical value for *y* choosed for $x \to \infty$, which allow the flow of material to cover any possible path on the surface of the material under deformation. At this point, we have not any theoretical model to explain the empirical Equation 1. As for as we now, granular flow maps for superplastic deformation has been not obtained before on experimental basis for metals. Our experimental results on Zinalco are in qualitative agreement with the oil-emulsion Ashby and Verral model [4]. As suggested by Ashby and Verral this granular flow maps are one way of identify non-uniform

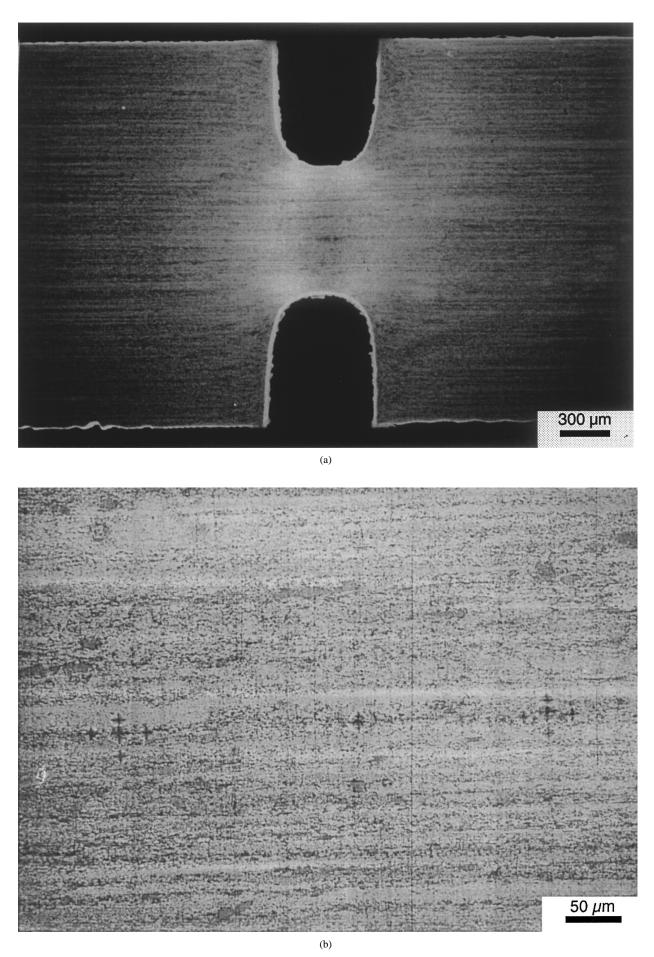


Figure 1 (a) Tension test specimen for SEM. (b) Initial Mesoscopic aspect of microstructure of the Zinalco alloy. The length size of the Tensile Sample are delimited by the two trapezoidal figures.

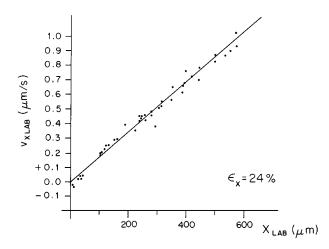


Figure 2 Linear relationship between velocity of material points in the sample and distance along tension axis as measured from coordinate system at rest in Laboratory. After a true deformation of 24.0%.

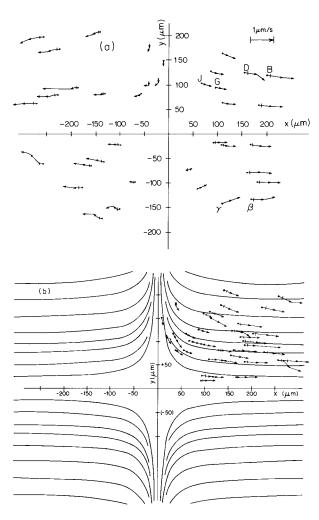


Figure 3 (a) Quantitative flow map of velocities on specimen through deformation. (a) Real flow in the four (x, y) regions of sample under deformation. (b) Flow lines obtained by using symmetry operations on real data on (a), denoted by arrows. Also are shown the idealized velocity flow map by which the experimental data can be described; denoted by the continuous curves.

flow. From Fig. 4 the following parameters can be calculated: the average deformation in each sector during a given elongation of the whole specimen, average strain rate, the average strain versus time curve (or local curves or values at any point of the specimen).

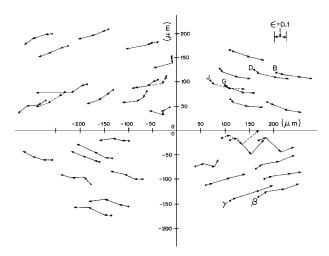


Figure 4 Quantitative diagram of strain flow at the surface of the deforming sample.

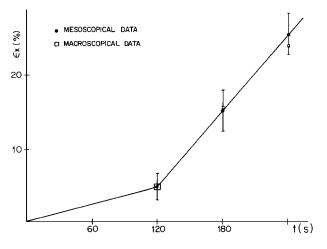


Figure 5 Average deformation ε_x versus time at mesoscopical scale denoted by black dots with error bars as indicated. Macroscopical ε_x versus time curve denoted by open square points.

For instance: see Fig. 5, which exhibits the deformation along the axis of tension versus the time: for the mesoscopical data. For comparative puposes in the same figure are shown the macroscopical data of the deformation (obtained from measurements on the increment of the distance between the diamonds figures above mentioned, which define the gage length of the sample). From this comparison it is clear that the mesoscopical data of the deformation for different times obtained by considering the movement of 38 grains statistically coincides with the macroscopical values, that which involve the movement about thirteen thousands of grains (where grain size length was calculated as 1.74 times the mean linear intercept $L = 3.5 \ \mu m$ [5, 6].

5. Conclusions

With the use of the new technique here presented the following problems can be approached:

1) As our technique allows to locate every grain on the surface of the of the sample during deformation gives the possibility to make mapping of the granular flow during superplastic deformation as suggested by Ashby and Verall [4]. 2) Our technique allows to measure rotation, translation, deformation and velocity of each grain on the sample surface all these measurements relatives to a coordinate system fixed at the center of the sample or relatives to coordinate system fixed at the laboratory. A very interesting application of this technique it is that in principle it allows the study of the processes of appearance of new external surface during the deformation of the sample.

3) Through the generation of maps for granular flow it is possible to obtain new data which, in principle, allow the development of more realistic models for superplastic deformation.

4) Also, this technique can be used to study the cooperative grain boundary sliding, in a complementary way than the usual one [7–10].

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