***In-situ* SEM Observation of Microstructural Evolution of High Purity Aluminum Deformed at Elevated Temperatures**

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The subgrain evolution in high purity aluminum (4N) has been observed *in-situ* during high temperature deformation, by means of the crystallographic contrast obtained from the backscattered electron signal of the scanning electron microscope (crystallographic contrast technique). It was observed that the substructure on the surface of the deformed sample represents the substructure developed inside the material. Evidence of increasing misorientation between adjacent subgrains and grain boundary migration with increasing strain is reported. This subgrain structure remains unchanged at room temperature when the applied stress is maintained during cooling. The crystallographic contrast technique demonstrates to be appropriate to measure subgrain size. Subgrain size values similar to those obtained by transmission electron microscopy are reported when the method of images superposition is incorporated to this technique.

(Received July 10, 1995)

Keywords: *in situ* subgrain evolution, creep, scanning electron microscopy, aluminum, microstructure

I. Introduction

Subgrain formation is one of the microstructural changes most widely studied among those occurring during high temperature deformation in high stacking fault energy materials. Subgrains formation is a strengthening mechanism of many metals⁽¹⁾, consequently, it is of great interest to understand in detail its evolution and its dependence with test variables, such as applied stress and temperature, and with strain. For example, several creep studies carried out by a variety of techniques such as transmission electron microscopy (TEM)⁽²⁾⁻⁽⁷⁾, scanning electron microscopy (SEM)⁽⁷⁾⁽⁸⁾, and optical microscopy (OM)⁽²⁾⁽³⁾⁽⁵⁾⁽⁹⁾⁻⁽¹¹⁾ have been conducted on aluminum.

The majority of these studies were executed under static conditions on samples that were previously deformed. To keep the same microstructure developed during the test, the samples were either quenched from the test, or slow cooled while maintaining the applied stress. The second alternative is based on experimental studies which demonstrate that the subgrain size, λ , is proportional to the inverse of the applied stress, σ , ($\lambda \propto \sigma^{-1}$)⁽¹²⁾. These studies⁽²⁾⁽⁶⁾⁽⁷⁾⁽¹⁰⁾ show some discrepancies on the strain dependence of subgrain misorientation. Whereas some investigations maintain that misorientation between subgrains increases with strain⁽²⁾⁽⁶⁾⁽¹⁰⁾, others conclude that the misorientation remains constant⁽⁷⁾. The later result is defended in further investigations⁽⁴⁾⁽¹¹⁾.

In order to have a better knowledge of the microstructural changes and the subgrain evolution during high temperature deformation, some of the above investigations were conducted *in-situ*; i.e. the observation of the microstructure was carried out during deformation of the samples. Results were obtained by TEM regarding the formation/evolution of cell formation and subgrain

boundary migration occurring during straining⁽⁵⁾. Although this research concluded that the *in-situ* deformation observed was the same as in the bulk, the reliability of this results must be, however, questioned since the thickness of the sample, 3 μm , was of the order of the subgrain size.

In the present work, a study of the subgrain evolution during high temperature deformation has been conducted on high purity bulk aluminum. This is the first investigation carried out *in-situ* by SEM for bulk material using the crystallographic contrast technique. The source of this contrast is due to the diffracted electrons coming from the first surface layers of the backscattered signal. Since these electrons are sensitive to crystal orientation, they can be used to identify different subgrains⁽⁷⁾⁽⁸⁾ and to monitor the misorientation between subgrains.

II. Experimental Procedure

The material used in the present investigation was a high purity (4N) aluminum ingot. To eliminate the as-cast structure, the ingot was subsequently rolled (20% reduction) and annealed (20 min, 500°C) to a final thickness of about 2 mm. This thermomechanical treatment led to a structure of equiaxed grains of about 1 mm in diameter. From the rolled plate, tensile specimens of 3 $\times$ 2 mm² section and 15 mm total length were machined for mechanical test. To grip the specimen, 5 mm of each extreme of the sample were used; the 5 mm of the centre were the gage section.

Tensile tests were performed in a micro-tensile-testing machine which operated inside the SEM. This allowed to account for the *in-situ* evolution of subgrain size during tensile testing at different stages of deformation as well as during cooling when the sample was maintained under stress. The capacity of the load cell was 196 N. A heating

system attached to both grips allowed the tensile test up to 500°C. Temperature gradient along the sample was minimized by controlling the heating power independently in each grip. Surface preparation of the sample involved conventional metallographical preparation and final electro-polishing. This surface finishing is required to remove the distorted layer originated in the mechanical polishing (Beilby layer). The crystallographic contrast technique was also used to measure subgrain size. In order to distinguish between adjacent subgrains with different orientation but showing similar brightness, images with different Bragg conditions were compared. For this purpose, photographs with the sample tilted at various angles at a given electron beam energy, or different elec-

tron beam energies at a given inclination of the sample were taken from the same area. The subgrain structure revealed from the different micrographs were superimposed (method of images superposition) and the subgrain size was measured using the linear intercept method.

To determine whether the microstructural evolution observed on the surface of the sample describes that occurring in the bulk material during plastic flow, metallographical sections of deformed samples were prepared and the microstructure compared to that observed on the surface.

Fig. 1 Evolution of subgrain structure in high purity aluminum (4N) with strain: (a) transient regime. (b)–(f) steady state. Strain of sample in each micrograph is expressed in percent.

III. Results and Discussion

From the comparison of the microstructure of deformed samples, both on the surface and in the bulk, it was seen that similar subgrain size structures were observed. Therefore, the substructure observed on the surface of the sample represents that developed in the bulk material during deformation, in accordance with the previous studies carried out at room temperature in aluminum⁽¹³⁾.

1. Microstructural evolution

Figure 1 is a sequence of micrographs obtained during a test performed at 300°C and at an initial strain rate of $2.8 \times 10^{-5} \text{ s}^{-1}$. Figure 1(a) was taken in the hardening region after yielding, whereas Figs. 1(b) to (f) correspond to the steady state region. For these testing conditions, the flow stress in the steady state was 6.2 MPa. The following points can be drawn from the photomicrographs of Fig. 1.

(i) The onset of subgrain formation occurs inhomogeneously before the steady state is attained. As can be seen from Fig. 1(a), which correspond to a tensile strain of 2.5%, the top centre grain (arrow) is developing subgrains whereas surrounding grains are still free from subgrains. On the later, extensive shearing leading to band formation occurred. This inhomogeneous subgrain formation has been previously reported⁽³⁾⁽⁵⁾⁽⁸⁾⁽⁹⁾ and is associated to the anisotropic nature of each grain and their different orientations with respect the tensile direction⁽⁹⁾. From this figure it is also seen that triple points are preferential sites for subgrain formation.

(ii) Once steady state is attained, $\varepsilon > 5\%$ (with ε tensile strain), subgrain size and morphology remain constant with strain, Figs. 1(b) to (f).

(iii) Subgrain boundaries are seen to migrate during plastic deformation in the steady state region, which agrees with previous investigations⁽⁴⁾⁽⁶⁾⁽¹⁰⁾. Arrows A in Figs. 1(c) to (f) indicate examples of subgrain boundaries which migrate during deformation. This migration leads to the annihilation and generation of subgrains. Other subgrains, however, remain unaltered in the course of deformation, but their orientation changes (arrow B).

(iv) As deformation increases, the misorientation between adjacent subgrains, especially during the initial 10% deformation, also increases as deduced from the increasing image contrast of subgrains. This corroborates some previous studies⁽²⁾⁽⁶⁾⁽⁹⁾, and is in disagreement with others⁽⁷⁾.

2. Changes of subgrain structure during cooling under stress

In order to account for the possible evolution of the microstructure of loaded samples during cooling, the subgrain structure developed at high temperature was continuously monitored after the heating system was switched off, but maintaining the applied stress. It was seen, for example, that the subgrain structure developed

during creep of a sample deformed 30% at 6.3 MPa and at 300°C was very well defined and it was not modified during cooling under constant load.

3. Subgrain size measurement

An example of subgrain size measurement using the method of images superposition is shown in Fig. 2. This figure shows the substructure of a sample which was tested at $2.9 \times 10^{-5} \text{ s}^{-1}$ and 300°C and elongated 30%. Both micrographs are of the same area and were taken at the same tilt but with different electron beam energies; 10 keV and 15 keV for Figs. 2(a) and (b), respectively.

As can be seen, subgrains which are not identified in Fig. 2(a) can be detected in Fig. 2(b) and vice versa. It is seen, then, that the actual subgrain size must be smaller than that measured from a single picture. By using the method of images superposition from 10 different Bragg conditions, to pick up as many subgrain boundaries as possible, a subgrain size of $18.5 \mu\text{m}$ was measured under the mentioned testing conditions inside the material. On the other hand, a subgrain size of $19 \mu\text{m}$ was measured on the surface of the sample.

Figure 3 shows a plot of subgrain size as a function of

Fig. 2 Subgrain structure taken at (a) 10 keV and (b) 15 keV and at the same tilt of the sample.

Fig. 3 Logarithm of the subgrain size as a function of logarithm of the Young modulus compensated stress. Lines denoting subgrain sizes determined by etch-pit, optical technique, and MET are reported in Ref. (5). Filled circles correspond to data of this work.

the Young modulus compensated applied stress from the present study together with data collected in a previous investigation⁽⁵⁾ and obtained using several techniques. Young modulus data at different temperatures are taken from Simmons and Wang⁽¹⁴⁾. As can be seen, the data from the present study follow the well-known relation $\lambda = \sigma^{-1(12)}$, and are very similar to those obtained by TEM⁽⁵⁾. The present values are smaller than those obtained by the etch-pit technique or optical microscopy. Previous studies on the subgrain size of aluminum measured both by SEM and TEM led to different values (smaller by TEM than by SEM)⁽⁷⁾. This discrepancy is attributed to the fact that the method of images superposition was not considered in this study, i.e. all measurements in the SEM were made under the same Bragg conditions.

IV. Summary

The formation/evolution of subgrains during high temperature deformation in high purity aluminum (4N) has been studied by means of *in-situ* tensile tests in the scanning electron microscope. The following conclusions

have been obtained:

(1) The subgrain structure observed on the surface of the sample accounts for that in the bulk.

(2) The subgrain structure developed in the early stage of deformation is rather inhomogeneous and evolves to a more homogeneous structure as deformation progresses.

(3) During steady state deformation the subgrain boundaries migrate, leading to formation an annihilation of subgrains, but the average subgrain size remains constant with strain. The misorientation between adjacent subgrains increases with strain.

(4) The subgrain structure developed during high temperature deformation does not change when the sample is cooled but the applied stress is maintained.

(5) The crystallographic contrast technique proves to be adequate to measure subgrain size when the method of images superposition is incorporated. Subgrain sizes measured at different stresses lead to similar values obtained by TEM.

Acknowledgement

Support from CICYT (Spain) under project MAT91-0685 is gratefully acknowledge.

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