Preferred crystallite orientations depth profile in the two phase alloy Zn-22% wt Al, determined by X-ray and neutron diffraction

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Recibido el 8 de enero de 1996; aceptado el 24 de agosto de 1998

In order to observe the texture inhomogeneity of the Zn-22% wt Al alloy, polar figures for the α-phase (111) and β-phase (002) reflections were measured by X-ray diffraction at four different depths in a hot rolled sheet sample. Also a sample in the form of a cube was assembled with several pieces of the sheet, with the same degree of deformation, to make it suitable for the measurement of its polar figures by means of neutron diffraction. In both phases, the corresponding typical rolling texture was observed. Therefore, it does not seem to exist any strong correlation between preferred orientations in both phases, as it might be expected. β-phase polar figures show a homogeneous texture, with a very small increasing orientation dispersion related to depth. The α-phase polar figures are very weak and they vary statistically but retaining the main characteristics of hot rolling polar figures. Neutron diffraction polar figures were also obtained and the results are in good agreement with the X-ray polar figures. Probably, easy grain boundary sliding, which is one of the main mechanisms of superplasticity in this alloy, is also responsible for a homogeneous distribution of strain and stress in the bulk of the sample.

Keywords: Texture; Zn-22%Al; X-ray and neutron diffraction

Para observar la inhomogeneidad de la textura de la aleación Zn-22% en peso Al, se midieron las figuras polares de las reflexiones (111) de la fase α y la (002) de la fase β por difracción de rayos X, a cuatro profundidades diferentes en una muestra laminada en caliente. También se construyó un cubo con varias láminas con el mismo grado de deformación para poder medir las figuras polares por difracción de neutrones. En ambas fases se observaron las correspondientes texturas típicas de laminación. Por lo tanto, no parece existir alguna correlación fuerte entre las orientaciones preferenciales de ambas fases, como podría haberse esperado. Las figuras polares de la fase β muestran una textura homogenea, con dispersión de orientación ligeramente creciente como función de la profundidad. Las figuras polares de la fase α aparecen muy débiles y varían estadísticamente pero retienen las características principales de la textura de laminación. Las figuras polares obtenidas con la difracción de neutrones presentan un buen acuerdo con los resultados de los rayos X. Probablemente, el deslizamiento fácil de las fronteras de grano, que es uno de los principales mecanismos de la superplasticidad en esta aleación, también es responsable de una distribución homogénea del esfuerzo y la deformación en el interior de la muestra.

Descriptores: Textura; Zn-22%Al; difracción de neutrones y de rayos X

PACS: 81.40.eF

1. Introduction

Preferred crystallite orientations, or texture, and the microstructure in polycrystals, are very important characteristics of all materials, because they control their physical properties and specially, the mechanical ones. Texture is measured through the so called polar figures.

A polar figure is a stereographic plot of the intensity of a Bragg reflection, as a function of sample orientation expressed by two angles, one measured as a rotation around an axis normal to the sample face, varying from 0° to 360°, and the other one measured as a rotation around an axis in the diffraction plane, varying from 0° to approximately 80° [1].

Polar figures have also been measured by neutron and electron diffraction. The neutron technique is advantageous for large grain sizes and bulk characteristics and the electron one is suitable for local microtexture determination.

To measure a polar figure, the sample is placed at the center of a special goniometer, which rotates around both axes, as mention above, at the same time as the diffraction intensities are being recorded. As the diffractometer is placed at a certain diffraction angle, two theta, only a family of planes is explored. Here only two plane families were studied, namely, the α-phase (111) and the β-phase (002).

Unfortunately, X-rays are strongly absorbed by matter and only a small zone in the neighborhood of the surface can be measured. This is a problem when texture is inhomogeneous: it varies with the sample depth. This is not an uncommon situation, since hot rolling deformation of the sample sometimes affects the surface in a different way than the bulk. Other minor agents, as dynamic and kinematic recrystallization, could also modify the texture. Obviously, physical properties of alloys depend mainly on the bulk rather than on surface texture.

Because the neutrons are only weakly absorbed by matter, polar figures measured with neutron diffraction deliver an average value of the bulk of the sample. For this reason we measured the texture also with this technique.

On the other hand, the Zn-22% wt Al alloy has also been widely studied because of its superplastic properties. Several studies have been performed [2, 3] relating texture with superplasticity, but they have been strongly criticized [4]. These texture studies have been made mainly with X-ray diffraction and it would be very interesting to see if texture is actually homogeneous in depth.

In this work, we intend to characterize texture inhomogeneity with depth for the Zn-22% wt Al alloy which could result from deformation of the samples when prepared to exhibit superplasticity. This means that a very fine grain structure, less than 10 microns in average grain size, is present in the whole alloy sample.

2. Experimental details

An ingot was prepared with commercially pure Al and Zn bars at the desired proportion, namely 22% weight Al and 78% weight Zn; and homogenized for 72 h at 350°C in an electric furnace. The ingot was then rolled 93% at 240°C in steps of about 4%, down to a sheet thickness of 0.75 mm.

An X-ray diffractogram revealed a superposition of the characteristic Bragg peaks from the cubic Al α-phase and those from the hexagonal Zn β-phase, indicating that the sample was the intended two phase alloy.

A piece of about 2 cm^2 was then cut from the sheet and attacked with diluted HNO_3, in stages of several hours in order to exhibit several depths. In each stage, the sample was polished and the α-(111) and β-(002) polar figures were measured. A linear depth parameter 's' was defined as 1 at the surface and 0 at the middle of its thickness. Polar figures were recorded with our updated Philips PW 1078/24 texture goniometer. Details of this instrument are given elsewhere [5].

Neutron measurements were made at the powder diffractometer of the Mineralogy Institute in the research reactor DIDO at the Research Center in Jülich, Germany [6].

A textureless powder sample, with the same phase composition was prepared for absorption and defocusing corrections. From this sample, spectra of α-(111) and β-(002) reflections were also measured. Polar figure evaluation was made using the poPLA (preferred orientation Package Los Alamos) software [7].

3. Results and discussion

Figure 1 is an X-ray diffractogram of the rolled Zn-22% Al sheet, showing a superposition of the cubic α-phase an the hexagonal β-phase. Figures 2a to 2d show the polar figures of the β-(002) reflection at four different depths: $s = 1, 3/4, 1/2$ and $1/4$ respectively, and Figs. 3a to 3d show the corresponding polar figures for the α-(111) reflection.

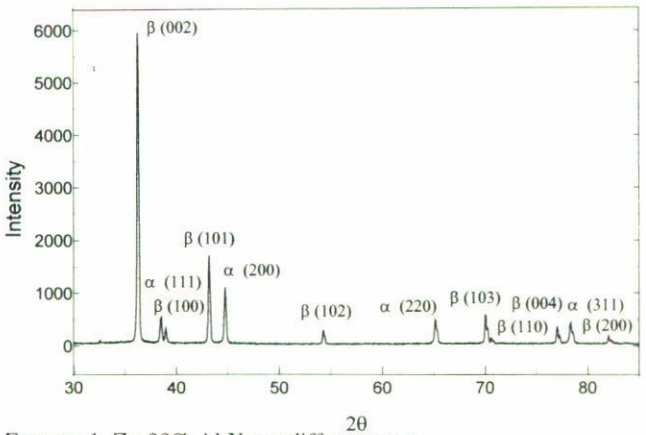

FIGURE 1. Zn-22% Al X-ray diffractogram.

From Figs. 2 we can see the typical rolling texture of the pure Zn policrystals, consisting of two peaks along the rolling direction, about 13° away from the origin. Very clear details can be seen here since this reflection is very strong. A very small peak broadening can be observed for decreasing s-values indicating that in the middle of the sample a slight orientation dispersion takes place. This can be better observed through a plot of the maximum of the peaks *vs.* depth, as shown in Fig. 5, with the assumption that the volume under the peaks is constant.

From Figs. 3, we see the α-(111) polar figures, which are weak mainly due to the fact that Al peaks are very small. However, the main details of the rolled aluminum can be recognized. Here we can find noticeable differences, but there is no clear evolution in a special direction: *e.g.* to dynamic recrystallization. Figs. 4a and 4b show the neutron diffraction results. Figure 4a is the α-(111) reflection and Fig. 4b is the β-(002) reflection. We observe here features very similar to the X-ray results.

4. Conclusions

Preferred crystallite orientations for the hot rolled Zn-22% Al are, to a large extent, homogeneous in depth, specially in the β-phase, although a very small orientation dispersion is apparent at the center of the sample. α-phase varies statistically but, in the average, it retains the same rolling features. Neutron polar figures, which take into account the average over the bulk, confirm this observation. On the other hand, those polar figure variations from X-ray diffraction, could indicate a small amount of orientation dispersion along the depth.

Normally, it is difficult to obtain a homogeneous texture because strain is modified in the bulk of the sample. On the other hand, this alloy, prepared for superplasticity, is fine grained and it is very likely that easy grain boundary sliding, which is one of the main mechanisms of superplastic deformation, could also be responsible for a homogeneous distribution of strain and stress in the bulk. Also, since both phases show their characteristic rolling textures, no strong correlation seems to exist between both phases orientations

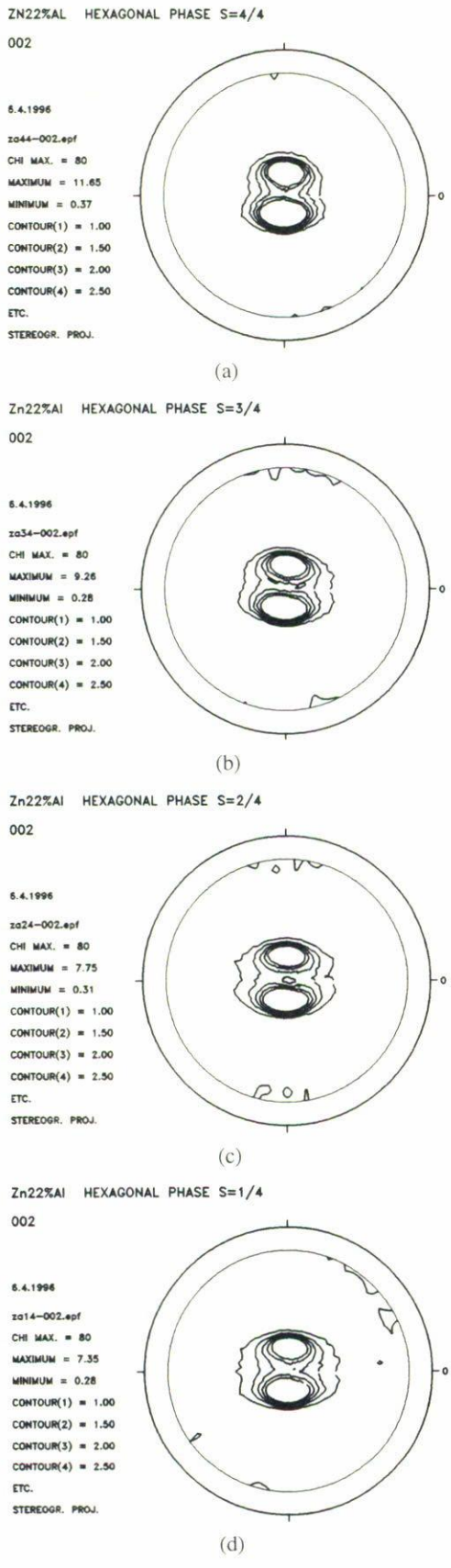

FIGURE 2. (002) β-phase polar figures at different depths: (a) $s = 1$, (b) $s = 3/4$, (c) $s = 1/2$, and (d) $s = 1/4$.

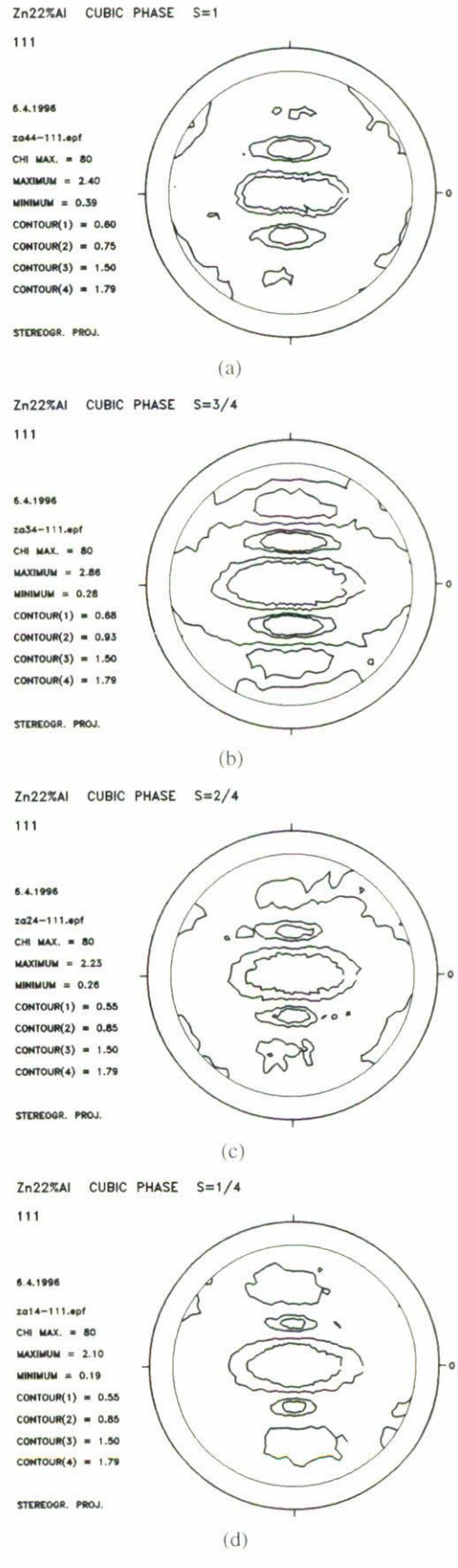

FIGURE 3. (111) α-phase polar figures at different depths: (a) $s = 1$, (b) $s = 3/4$, (c) $s = 1/2$, and (d) $s = 1/4$.

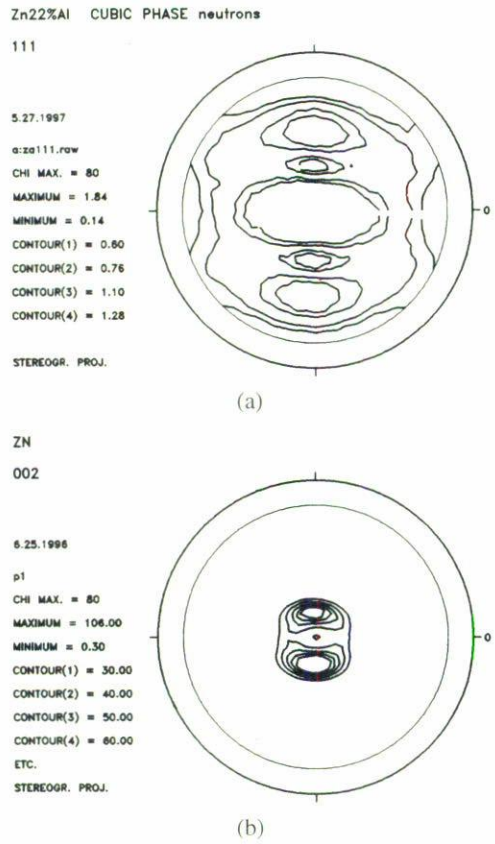

FIGURE 4. Neutron diffraction polar figures: (a) α-(111) reflection and (b) β-(002) reflection.

by simultaneous deformation. Nevertheless, an actual correlation function should be determined by measuring other reflections.

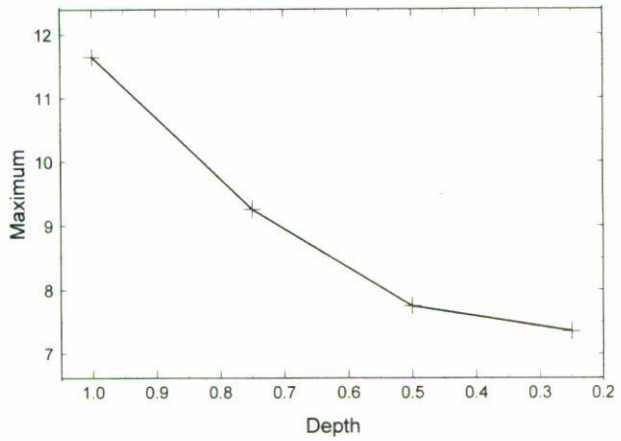

FIGURE 5. Height of the peaks *vs.* depth parameter s from Fig. 2, showing that at low values of s the peaks are lower, and therefore slightly broader than for $s = 1$.

Acknowledgments

This Project was funded also by CONACyT Mexico, through the contract 3329-E. The authors would like to thank Ing. Jorge Araujo from the Metallurgy Laboratory of the Escuela Superior de Ingeniería Química e Industrias Extractivas of the IPN, for his kind help with the hot rolling of the sample. Also we acknowledge the assistance of M. en C. Carlos Vega R. with the preparation of the alloy. We are also indebted to Dr. W. Schäfer and Dr. E. Jansen of the Mineralogy Institute of the University of Bonn for measuring the neutron polar figures. We are grateful with Prof. J.E. Rosado Chauvet of the Foreign Languages Center of the IPN, for his assistance with the english redaction.

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