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Microstructure and Texture of Al–2Si–xSn (x = 0, 4, 8 mass%) Alloys Processed by Equal Channel Angular Pressing

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The influence of Sn on the microstructure, microstrain and grain morphology in Al–2Si–xSn samples processed by ECAP is reported and discussed. The pseudo-binary Al–xSn alloys (where x = 0, 4, 8 mass%) were produced by conventional ingot casting. Samples were characterised by X-ray diffractometry (XRD), transmission electron microscopy (TEM), dynamical mechanical analysis (DMA) and microhardness. Results showed that the initial texture was modified after several ECAP passes and the formation of subgrains was observed. The presence of Sn enhanced the tribological properties of the alloy but, the ECAP capacity for grain refining was reduced. Besides, it was also confirmed that the damping capacity and microhardness behaviour were dependent of the Sn contents.

Keywords: aluminium alloys, equal channel angular pressing, nanograins, transmission electron microscopy, Rietveld refinement

1. Introduction

In the recent years, there has been considerable scientific and industrial interest in severe plastic deformation produced by ECAP (Equal Channel Angular Pressing). The ECAP process in aluminium alloys can produce equiaxed grains of submicrometer sizes and, in some cases, a spectacular ductility enhancement can be achieved.¹ Some binary alloys with high plastic deformation that belongs to FCC and BCC cell types have been studied, especially under condition of extrusion, wire drawing and rolling. In the last two decades the main efforts have been focused on Cu or Al systems, with body centred cubic (bcc) second phase such as Nb or Ta, i.e., Cu–X (where X = Nb, Ta, Cr, V or Fe),²–⁸ having the objective of increasing the mechanical strength of these alloys. In the 1990’s, some studies started with hexagonal (hcp) metals including Ti–Y,⁹,¹⁰ Mg–Ti,¹¹ Al–Ti¹² and Al–Mg,¹³ however, the understanding of the fundamental mechanisms controlling the mechanical deformation and the microstructure evolution is still unclear.

At present, there are some studies on the deformation behaviour of two cell such as the combination FCC–BCC and FCC-Hex, however, the reports on the combination of FCC-Tetragonal cells are still underway. Previous studies have shown that the addition of Sn enhances the UTS of Al based alloys due to the Hall–Petch mechanism (an increase of 1.4 to 6.5%). These experiments were carried out under high deformation conditions by means of a process of rolling.¹⁴ In this study, we will continue these results with a series of ECAP experiments. The Al–Sn alloys are usually employed as bearings; this is due to their good combination of strength and frictional behaviour properties. The latter property could be interesting during the ECAP process, as this alloy has not been investigated in such conditions. Our objective is to characterise this alloy and investigate the role, beneficial or not, that the Sn could have. Several techniques concerning the characterisation of microstrain, grain size and crystal texture will be employed.

2. Experimental

Commercial aluminium (Al) containing 2% mass Si alloy was melted with Sn in order to achieve the nominal composition Al–2Si–xSn alloys (where x = 0, 4, 8 mass%). The ingots were machined into rod shape with cross section of 16 mm × 16 mm and length of 130 mm. The rods were then angular extruded at room temperature. The ECAP die consisted of two separated blocks of tool Cr–V tool steel (H13), which were held together to form a single internal channel having the same square cross section. The angle between the two channels was 110 degrees. The surface of the internal channel was lubricated with MoS₂ powder and oil in order to reduce the friction between the die wall and the alloy rod. The ECAP passes were performed with a rotation of 180 degrees between the passes.

Before starting the ECAP process, the samples were observed by optical microscopy. A standard metallographic preparation was performed over the samples prior to the observation. For higher magnification, microstructural characterisation by transmission electron microscopy was used with a FEI TECNAI G2. The thin foils samples were prepared by means of the focus ion beam technique (PHILIPS FIB 200).

The determination of the crystallographic texture was performed on a X’Pert MRD 4 circles diffractometer, with Kα—Cu at 40 KV and 40 mA. The device has a primary optical 1 × 1 mm² and point detectors with a Kα monochromator in the secondary position. The texture was determined on samples of Al alloy containing different Sn amounts, (0% Sn, 4% and 8% Sn) for several ECAP passes. For each sample, four hk1 plans have been explored: (111), (200), (220) and (311). A background correction and a correction of defocus were applied to the experimental files prior to
recalculation of pole figures. Samples were taken so that the normal line was parallel to the flow direction. Electropolishing (about 150 µm) is performed to remove the work hardened layer, due to the machining, prior to the texture analysis.

The experimental XRD spectra for the broadening peak analysis were determined using a Bruker D8 Advance Diffractometer equipped with a graphite Ka2 monochromator and a VANTEC 2000 2D detector. Relative variations of microstrain and size particle were evaluated by Williamson–Hall plots, this method assumes a Lorentzian XRD peak and a V ANTEC 2000 2D detector. Relative variations of Diffractometer equipped with a graphite K

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From the pole figures examination of the samples before ECAP, we found that the grains are too large, so there is not enough sampling to make proper measurement by this technique (grain size ranges from 20 to 50 microns in diameter).

After the 1st ECAP pass, it appears a strong crystallographic texture. The pole figures are quite similar for all the Sn contents. Figure 3 shows the results for 4% Sn, they are compatibles with a texture component \{001\} \{110\} and \{112\} \{110\}. The types of orientation and the intensity of the texture are the same for the different Sn contents studied (average intensity ratios are: 6.92 for 0% Sn, 6.5 for 4% Sn and 6.80 for 8% Sn). It should be noted that the grain number in the volume analysed is large enough, qualitatively demonstrating that the grain size of the material has certainly greatly reduced after this first pass of ECAP.

After 2nd pass, the pole figures are similar for the 3 sample types. On the contrary to what we observed after the 1st pass of ECAP, the intensity ratios vary from one sample to another, (see Fig. 4); which are:
- 0% Sn: average intensity ratio = 3.6
- 4% Sn: average intensity ratio = 5.5
- 8% Sn: average intensity ratio = 2.15
The type of texture is the same, \{145\} \{111\}, but the intensity of the texture is much higher for the sample with 4% Sn. The presence of 8% Sn has reduced the texture compared to a Sn-free material. This effect could be beneficial for the alloy.

As summary, on the flow plane, the 1st pass of ECAP generates two texture components: \{001\} \{110\} and \{112\} \{110\} in which the planes \{001\} and \{112\} are separated by an angle of 35°. On the other hand, the 2nd pass generates only
one texture component \{145\} \{11\bar{1}\}, which intensity depends on Sn content.

Figure 5 shows the effect of the microstrain and particle size on the breadth of the Al diffraction peak (311) for 0 mass% of Sn content, the referred peak was shifted in 2θ and normalised in order to appreciate clearly the XRD broadening effect of ECAP on these alloys. This figure clearly displays the effect of each pass on the diffraction peak broadening, where the main difference was observed between the undeformed sample and the first ECAP pass. However, the difference between the first and the second pass was very marginal and could lay within the experimental scatter.

Figures 6–8 show the Williamson-Hall plots for different Sn contents as a function of the ECAP passes, they show a clear correlation between the strain (slope), the size particle (reciprocal of the ordinate intersection) and the number of ECAP passes. Because of the large initial particle size at 0 pass, the instrumental integral breadth has the same magnitude order than the observed breadth, therefore the measured particle size are irrelevant for 0 ECAP pass, the same consideration applies for the Rietveld refinement results.

Table 1 summarises the data obtained from Rietveld refinement, it is noticed that Sn diminishes the microstrain in the material. At the same time, a tendency exists to increase the crystallite size with the Sn content. At identical chemical composition, the different passes of ECAP increased the microstrain. The cell parameter remains almost constant. Such observations tend to speculate that Sn plays a tribological role allowing an easier gliding of the surfaces but with a moderate detrimental effect on grain size.

Figures 9–11 show the damping capacity changes as a function of the temperature, it can be seen that there is quite similar behaviour for 4% and 8% Sn samples, where the damping capacity increases with the temperature and the...
number of ECAP passes (Figs. 10–11). For the sample without Sn, a different behaviour is observed. Figure 9 shows that the maximum damping occurs for the sample that does not contain Sn and at zero ECAP pass. For 1 and 2 passes of ECAP, the behaviour is very similar. These results confirm that DMA technique and damping behaviour could be used as a monitor of grain evolution during ECAP, but also it shows that Sn has an important effect on the elastic behaviour of the alloy. On the other hand, the curves split when the temperature increases, this result was not expected, and suggests a new study at higher temperatures is necessary, and looking for degradation conditions of these alloys will be the objective.

Figure 12 shows the evolution of the Vickers microhardness as a function of the Sn content and the number of ECAP passes. The ECAP process increases the Vickers microhardness of the alloy, if 2 passes of ECAP are performed, the microhardness value is duplicated. At the same time, it can be observed that Sn content has an effect of diminishing the microhardness. This behaviour is compatible with the DMA results.
4. Conclusions

The ECAP process can reduce significantly the grain size in only two passes. The texture induced by the process is modulated by the different passes but not destroyed. The formation of subgrains seems to be very local, indicating that strain heterogeneity exists in the sample (zones where grains are large with practically no dislocation and zones with a high dislocation density). The microstrain and grain size measured by XRD showed that Sn plays an important tribological role in the alloy but it reduces the ECAP capacity for refining the grains. Damping capacity and microhardness confirm that Sn affects the elastic behaviour of the alloys and proves that the DMA is a suitable technique for analysing the grain size evolution.

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