MICROSTRUCTURES AND MECHANICAL PROPERTIES OF SUBMICROMETER-GRAINED Al ALLOYS PRODUCED BY EQUAL-CHANNEL ANGULAR PRESSING

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ABSTRACT Grain refinement was attempted using the equal-channel angular (ECA) pressing technique for a wide range of commercial Al alloys such as 1100, 2024, 3004, 5083, 6061 and 7075. Transmission electron microscopy revealed that grain sizes less than 1 μm are attained in these alloys. Tensile tests at room temperature showed that the strength increases with an increase in the imposed strain during pressing but the elongation to failure remains little changed following a large decrease after the first pressing. Static annealing experiments demonstrated that extensive grain growth occurs above ~200°C in 1100, 3004, 5083 and 6061 but the submicrometer grained structures in 2024 and 7075 are stable even at 300°C.

Keywords: *equal-channel angular pressing, grain refinement, proof stress, elongation to failure, static annealing experiments*

1. INTRODUCTION

Grain refinement is effective for the enhancement of tensile strength and this effect is well described by the Hall-Petch equation [1,2]. The characteristic feature involved in this effect is that the strengthening of metallic materials is achieved without reducing ductility.

It was shown that equal-channel angular (ECA) pressing is capable of refining the grain size down to the submicrometer level [3]. Recent studies demonstrated that the Hall-Petch relationship holds for ECA pressed Al-Mg alloys [3-6]. There is also a report that the toughness was improved when an Al-Mg-Si alloy was subjected to ECA pressing [7].

The importance of the ECA pressing technique is that it is possible to apply to large bulk materials prepared by ingot metallurgy and thus, unlike powder metallurgy, no attention is necessary regarding the effect of residual porosity in the materials. It is then anticipated that the ECA pressing technique is more practical in industrial application. So far, there have been a limited number of applications of ECA pressing to commercial Al alloys [3,6,8-10] and especially these applications have been performed mostly at elevated temperatures. In this study, grain refinement is attempted using ECA pressing at room temperature on various commercial Al alloys and their subsequent tensile properties are examined.

2. EXPERIMENTAL PROCEDURE

This study used six different commercial Al alloys, 1100, 2024, 3004, 5084, 6061 and 7075.

All alloys were fully annealed and machined to dimensions of 10 mm in diameter and 60 mm in length for ECA pressing. The ECA pressing was conducted at room temperature (~20°C) using a solid die having a channel angle of 90°, which creates an equivalent strain of ~1 on one passage through the die [11]. The pressing was repeated up to a maximum of 8 passes but was terminated when breaking occurred in the samples as in 2024, 5083 and 7075 or when homogeneous equiaxed grained structures with high-angle grain boundaries were established as in 1100, 3004 and 6061. The samples were rotated about the longitudinal axis by 90° in the forward direction after each consecutive pressing. In an earlier paper [12], this rotation procedure was designated route B_C and it leads most efficiently to a homogeneous equiaxed grained structure [13,14]. The samples after ECA pressing were sliced perpendicular to the longitudinal axis with a thickness of ~0.4 mm. The slices were subjected to static annealing at selected temperatures in the range of 100°C to 300°C for 1 hour.

All slices were mechanically ground to a thickness of ~0.15 mm and subjected to twin-jet electropolishing in a mixture of 20%HNO_3 and 80%CH_3OH for 2024 and of 10%$HClO_4$, 20%$C_3H_8O_3$ and 70%C_2H_5OH for the remainder. The grain sizes were measured directly from grains having well-defined grain boundaries.

Tensile specimens having dimensions of 2x3 mm^2 in cross section and 5 mm in gauge length were cut parallel to the longitudinal axes of the ECA-pressed samples. For comparison purposes, tensile specimens with the same dimensions were also prepared from the alloys in the fully annealed conditions and from the cold-rolled 3004 by taking the rolling direction parallel to the tensile axis. Each specimen was deformed in tension at room temperature at a constant displacement rate equivalent to an initial strain rate of 3.3×10^{-4} s^{-1}. The load variation was recorded with respect to time and converted to the relationship of stress versus strain.

3. RESULTS AND DISCUSSION

Figure 1 shows typical microstructures after ECA pressing for all alloys together with selected area electron diffraction (SAED) patterns taken from a 6.3 μm region in the corresponding areas. The grain size appears to be largest in 1100 but, in any alloy, the microstructure consists of grains having sizes less than 1 μm. The SAED patterns indicate that these grains are separated by high-angle boundaries. In the microstructures, there are many grain boundaries which are wavy or not well defined and there are also many dislocations within the grains. All these features indicate that the microstructures of the ECA pressed alloys are in a high energy and non-equilibrium state and these features are similar to those reported earlier [3, 15-17].

The 0.2% proof stress and the elongation to failure are plotted in Fig.2 (a) and (b), respectively, as a function of equivalent strain imposed by the ECA pressing. It should be noted that the values of the equivalent strain given in Fig.2 (a) and (b) numerically correspond to the numbers of the ECA pressing since a die having a channel angle of 90° creates an equivalent strain of ~1 on each passage though the die. The values at the equivalent strain of 0 correspond to the results obtained from the specimens in the fully annealed conditions. The proof stress increases with an increase of imposed strain but the increase is significant only after the first pressing followed by a gradual increase with a further increase in strain. The level of the proof stress is highest in 5083 and lowest in 1100 and the order follows the Mg content in the specimen: 4.83% (5083), 2.56% (7075), 1.38% (2024), 1.15% (3004), 1.02% (6061) and 0.002% (1100) in mass%. The elongation to failure exhibits behavior which contrasts to the proof stress with a large decrease after the first pressing. With a further increase in imposed strain, however, the elongation to failure remains the same or even

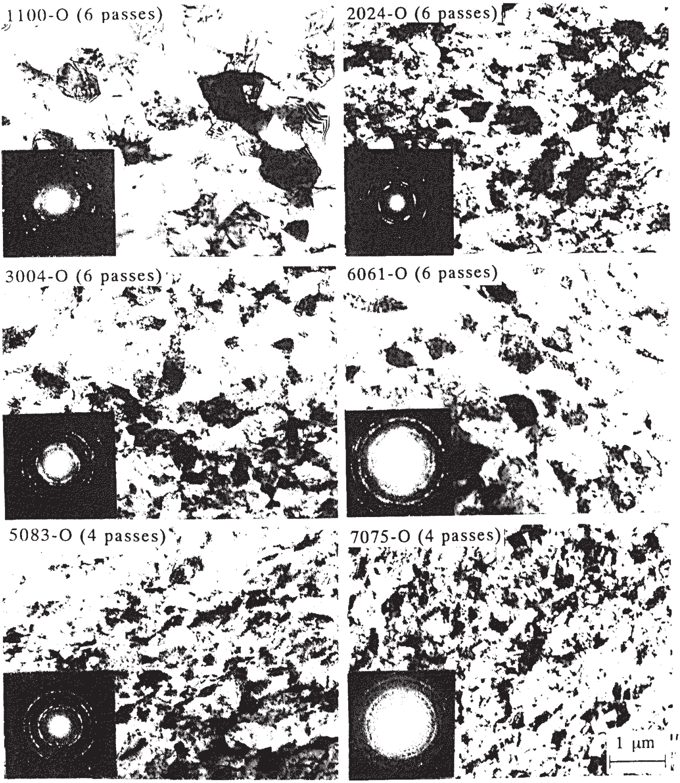

Fig.1. Microstructures of as-ECA-pressed alloys and corresponding SAED patterns.

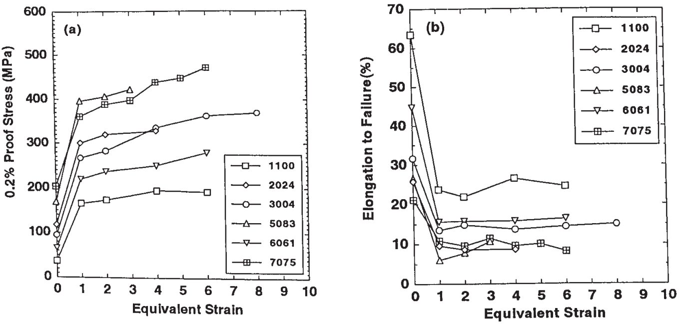

Fig.2. Plots of (a) 0.2% proof stress and (b) elongation to failure against equivalent strain. Note values of equivalent strain correspond to numbers of ECA pressing.

increases in 5083. The results of the tensile tests suggest a potential enhancement of toughness when the ECA pressing is applied to the alloys of this study. In fact, an improvement of toughness was reported by Kawazoe et al.[7] through Charpy tests on an ECA-pressed Al-Mg-Si alloy.

Figure 3 (a) and (b) compares the 0.2% proof stress and the elongation to failure, respectively, obtained on ECA-pressed 3004 (ECAP) with those on cold-rolled 3004 (CR). It should be noted that the tensile tests were conducted under the same conditions and with the same dimensions of specimens for both ECA-pressed and cold-rolled 3004. The strain introduced by the cold rolling was converted to the equivalent strain and the results were plotted on the same scale as those for ECA pressing. There is no appreciable difference between the proof stresses of the ECA-pressed and cold-rolled specimens. However, the difference seems significant when a comparison is made between the elongations to failure of both specimens. The total elongation is appreciably lower in the cold-rolled specimens than in the ECA-pressed specimens especially as the equivalent strain

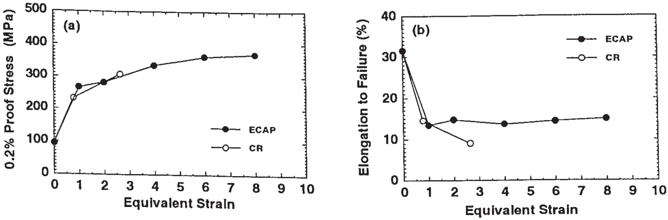

Fig.3. Plots of (a) 0.2% proof stress and (b) elongation to failure against equivalent strain for comparison between ECA-pressed 3004 and cold-rolled 3004.

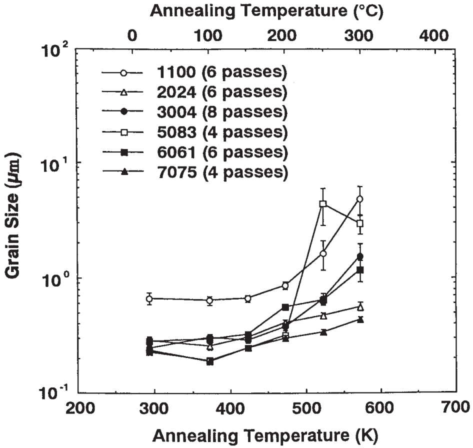

Fig.4. Variation of grain size with respect to annealing temperature.

increases. It should be noted that a comparison beyond an equivalent strain of 3 was not possible in this study because of the limitation in the initial thickness of ingots: since the final thickness should be kept to 2 mm for the tensile specimen, an initial thickness of ~65 mm is required to achieve an equivalent strain of 4. The results suggest the importance of ECA pressing for the improvement of ductility. It is considered that this improvement is due to the formation of fine-grained structures having high angle boundaries.

The grain sizes after static annealing of the ECA-pressed samples are plotted in Fig 4 against the annealing temperature. For the alloys of 1100, 3004, 5083 and 6061, the grain size remains essentially unchanged up to a temperature of 200°C. Observations by TEM shows that large grains with sizes greater than 1 μm formed in some part of the samples at 250°C and they extended throughout the samples at 300°C. For the alloys of 2024 and 7075, however, the fine-grained structures of ECA pressing were retained even at 300°C.

4. SUMMARY AND CONCLUSIONS

(1) Grain refinement down to a submicrometer level was achieved in six different commercial Al alloys using ECA pressing.

(2) Tensile tests showed that the proof stress significantly increases after the first ECA pressing followed by a gradual increase with a further increase in strain. The elongation to failure exhibits a large decrease after the first pressing but it remains the same or even increases with a further increase in imposed strain. These results suggest a potential enhancement of toughness in the

alloys used in this study.

(3) The level of the proof stress was highest in 5083 and lowest in 1100 and the order follows the Mg contents of the alloys.

(4) There was no appreciable difference between the proof stresses of the ECA-pressed and cold-rolled specimens. However, the total elongation was appreciably lower in the cold-rolled specimens than in the ECA-pressed specimens especially as the equivalent strain increases. It is suggested that this improvement is due to the formation of fine-grained structures having high angle boundaries during ECA pressing.

(5) Static annealing of the ECA-pressed samples showed that extensive grain growth occurs above ~200°C in 1100, 3004, 5083 and 6061 but the fine-grained structures are retained in 2024 and 7075 even at 300°C.

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