Fabrication of Superplastic Aluminum Sheets by Equal-Channel Angular Pressing Followed by Isothermal Rolling

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A two-step technique consisting of equal channel angular pressing (ECAP) with rectangular shape of channels followed by isothermal rolling (IR) was developed to produce superplastic sheets from high-strength aluminum alloys. To demonstrate the feasibility of this technique an Al-Li-Mg-Sc alloy was subjected to ECAP up to a true total strain of ~4 at a temperature of 325° C. This material subjected to ECAP was additionally processed via isothermal rolling at the similar temperature to a true strain of ~2. Sheets produced from this alloy demonstrate a high superplastic ductility of 1481% at 350° C and $\epsilon = 1.4 \times 10^{-2} \text{ s}^{-1}$. It was shown that superior superplastic properties are attributed to very uniform ultra-fine grained (UFG) structure with an average grain size of ~1.3 µm that evolved in this alloy under intense plastic straining. Thus it was demonstrated that the two-step processing is a simple technique that is best suited to produce superplastic sheets from aluminum alloy.

Keywords: equal-channel angular extrusion, aluminum alloy, superplasticity, isothermal rolling, microstructure.

1. Introduction

Equal channel angular pressing (ECAP) is a widely-known processing procedure for the fabrication of aluminum alloys with the ultra-fine grained (UFG) structure, in which a sample is pressed through a die comprising two channel portions having an L shaped configuration [1]. Typically, samples are pressed for several consecutive passes through the die to impose very high strains [1]. This technique is especially attractive for the producing of semi-finished products from aluminum alloys for several reasons. First, conventional tool steels, used as a structural material for the ECAP die, make feasible the ECAP of aluminum allovs in a wide temperature range. Second, ECAP can be applied to process fairly large billets due to the fact that this processing requires a reasonable low load capacity. Third, ECAP is a relatively simple procedure where high strains are introduced into a billet by a simple shear providing sufficient homogeneity of the deformation microstructure. Fourth, the simple shear nature of the plastic deformation under ECAP is highly effective for extensive grain refinement in aluminum alloys in comparison with the other deformation methods. However, ECAP usually provides the fabrication of relatively short billets in the form of bars or rods with a length being higher than the thickness by a factor of four or even less. These billets cannot be used directly for the commercial fabrication of thin sheets. Billets in the form of plates having rectangular cross-section are best suited to roll thin sheets at ambient temperature. Thin sheets can be easily rolled from a plate subjected to ECAP. Design of ECAP die for processing of plate samples was developed [1] to produce thin sheets with UFG structure from low alloy aluminum.

It was shown [2] that high alloy aluminum with UFG structure can be produced as sheets through combination of ECAP with a rectangular shape of the intersecting channels and subsequent rolling at a similar temperature. This two-step processing was recently modified [3] by the application of back pressure in ECAP. The role of the back-pressure is threefold. First, processing by ECAP with back pressure provides high uniformity of the metal flow and, therefore, the uniformity of the deformation microstructure developed along the billet cross-section due to removal of the dead zone and attaining a pure shear. Second, back pressure improves the ductility by elimination of any perceptible cracking on billet surfaces. Third, the application of back pressure improves billet quality due to elimination of outer corner defect. The last two issues are very important as the grain refinement using ECAP processing required multiple passes, and a billet has to withstand intense plastic straining without

additional machining that is widely used to eliminate pressing defects in ECAP processing without back pressure. In addition, the back pressure system provides a back pressure under the working stroke, and knocks out the billet from the die under the back stroke (Fig.1).



Fig.1. Schematic illustration of the ECAP die

Combination of the modified ECAP technique and subsequent IR gives a synergetic effect on economic efficiency of this technique due to a decrease in total operation time and enhancement of sheet quality [3]. It was shown in previous works [2,3] that both IR following ECAP and imposing a back pressure as well provide increased the uniformity of UFG structure developed under ECAP, allowing a reduction in a total strain being necessary for the formation of a fully recrystallized structure with an average grain size of ~1 μ m. As a result, the two-step technique consisting of modified ECAP followed by IR is highly suitable for commercial application. The aim of the present work is to demonstrate the feasibility of this technique for the manufacturing of thin sheets from an Al-Li-Mg-Sc alloy. It will be shown that this technique provides extensive grain refinement and attaining superior superplastic properties by imposing essentially moderate strain into this alloy by ECAP with rectangular shape of channels followed by rolling under isothermal conditions.

2. Experimental methods and material

The experiments were undertaken using extruded rods of an aluminum alloy with a chemical composition of Al-5.1%Mg-2.1%Li-0.17%Sc-0.08%Zr (weight pct). This alloy was designated in the former Soviet Union as 1421 aluminum alloy and denoted as 1421 Al herein. Details of extrusion process of this alloy were reported previously [5-7]. Plates with a rectangular cross-section of 152×34 mm² and a length of 152 mm were machined from the central part of the extruded rods parallel to the major axis. These plates were deformed by ECAP at 325°C. An isothermal die with a rectangular cross-section of 152×34 mm² and a channel angle, Φ , of 90° had a horizontal L-shaped configuration. It was set up on a computer controlled hydraulic press with a 400 ton force with a separate hydraulic line to provide back pressure (Fig.2). The angle Ψ , which represents the outer arc of the curvature where the two parts of the channel intersect, was equal to ~1°. The deformation through this die produced an imposed strain of ~1 in each passage [1]. The plates were pressed 4 times with an approximate total accumulated true strain of ~4; the samples were rotated by 180° around the X-axis (Fig. 4a), i.e. route C_x was used [1]. The pressing speed was approximately 3 mm/s.

The die for the modified ECAP (Fig.1) has a well-adopted [1-4] horizontal configuration. A heating system was used to carry out ECAP in isothermal conditions up to 450°C. The sample with the rectangular cross-section is inserted into the inlet channel. The flat billet with the dimensions depicted in Fig.2 is extruded through two intersecting channels of identical cross areas by the plunger 1 which forces the flat billet under an appropriate ram pressure P (Fig.1). Concurrently, under a working stroke of the ram 1, the plunger 2 provides a back pressure of 0.2P (Fig.1); the ram 2 forces the billet from the opposite side by a ram pressure being 20 pct. from the extruded pressure P. The extrusion process terminates when the plunger 1 attains the upper wall of the outlet channel. The slider (Fig.1) is secured (under the extrusion). To extract the billet the slider is lifted up by the ejector and fixed by clamps which are not depicted in Fig.1. To eject the billet the plunger 2 plays a role of the ejector moving the extruding billet to the left by a long retreating stroke. Finally, the billet is removed from the die; the slider and the plunger 2 return to their original position. This type of ECAP processing produces the defectless flat billets. As a result, these billets are suitable for immediate repetitive pressing; the total operation time of ECAP processing up to a total strain of ~4 did not exceed 8 minutes.

The as-pressed plates were ground parallel to the extrusion direction; 5 mm from each side were removed to make plates with flat parallel surfaces without defective layers. The X, Y and Z planes correspond to the planes perpendicular to the rolling (RD), transverse (TD) and normal (ND) directions, respectively, as shown in Fig.4. These plates with dimensions of $115 \times 115 \times 15$ mm were heated to 325° C and then rolled to a final thickness of 1.8 mm, giving a total reduction of 88% in 8 passes. A 6-high roll mill with isothermal internal rollers being 70 mm in diameter and 300 mm in length was used (Fig.3). The inner rollers were heated and kept at 325° C during rolling.



Fig. 2. ECAP die with a back pressure with 400 tons press capacity

Fig. 3. 6-high roll mill with isothermal internal rollers

Tensile specimens of 6 mm gauge length and $1.4 \times 3 \text{ mm}^2$ cross-section were machined from the resulting thin sheets; the gauge lengths were parallel to the RD. These samples were tensioned to

failure at a temperature of 350°C at strain rates ranging from 1.4×10^{-4} to 1.4×10^{-1} s⁻¹. Other details of mechanical tests were reported [5, 6] previously.

Structural characterization was carried out in parallel sections of the Z-plane, i.e. (RD) - (TD) sections of the rolled plates (Fig.4b). Microstructural changes and cavitation during superplastic deformation were examined in the similar parallel sections. The areas within 5 mm of the fracture surface were analyzed to examine cavitation. The methods of optical metallography (OM), transmission electron microscopy (TEM), cavitation studies and electron backscattering diffraction (EBSD) analysis were described in previous works [2,3,5,6]. The misorientations of (sub)grain boundaries were determined using a JEOL JSM-840 SEM fitted with an automated EBSD pattern collection system provided by Oxford Instruments, Ltd. Notably, thick and thin lines on the EBSD maps indicate the high-angle boundaries (HAGB) (\geq 15°) and low-angle boundaries (LAGB) (3-15°), respectively. Thin foils were examined using a Jeol-2000EX TEM with a double-tilt stage at an accelerating potential of 200 kV.



Fig. 4 Schematic illustration of (a) a rectangular plate for ECAP and (b) subsequent rolling

3. Results and discussion

ECAP up to a total strain of ~4 provides the formation of a partially recrystallized structure (. 5a). HAGBs dominate in the deformed structure; the population of HAGBs is ~79 pct; the average misorientation is 33.1°. The fraction of recrystallized grains with an average size of ~1.6 μ m is very high ~75 pct. It seems that the use of ECAP die with rectangular shape of billets provided an increase in the recrystallized fraction from ~50 to ~75 pct at a temperature of 325°C and similar total strain [7]. In addition, ~75 pct recrystallized fraction was attained at significantly low total strain in comparison with ECAP with rod samples [7].

Subsequent IR leads to the formation of the essentially uniform recrystallized structure (Fig.5b). The volume fraction of recrystallized grains is 93pct., the average grain size is ~1.3 μ m; the portion of HAGBs is 78 pct., the volume fraction of true grains entirely delimited by HAGBs is 50 pct.; the average misorientation is 32.4°. Thus, the combination of ECAP and IR provides attaining an almost fully recrystallized structure at a total strain twice less than that imposed into the 1421 Al by conventional ECAP with rods [1] for the similar grain refinement [7].

True strain – true stress curve of the 1421 Al subjected to IR following ECAP is shown in Fig.6a. It is seen that extensive strain hardening takes place initially up to a true strain of ~0.4. After reaching a maximum stress, the flow stress continuously decreases until fracture. A well defined peak in flow stress can be observed, and no steady-state flow occurs (Fig. 6a). A progressive strain softening takes

place with strain at $\varepsilon \ge 0.4$. Two stages of strain softening could be distinguished. In the strain interval 0.4-1.5 the strain softening occurs with low rate. At higher strain, extensive strain softening takes place. However, an exceptionally high elongation-to-failure of 1481% was attained at a temperature of 350° C and an initial strain rate of 1.4×10^{-2} s⁻¹ despite the strain softening. It seems that high total elongation is attributed to unusual strain dependence [8] of the coefficient of strain rate sensitivity, m (Fig.6b). The m value tends to increase with increasing strain. At high strains, the m value attains ~0.65 that compensates extensive softening. As a result, a uniform plastic flow takes place in the 1421Al up to failure; pseudo-brittle fracture [8] restricts ductility of the 1421 Al.



Fig.5. Typical microstructures of the 1421 Al subjected to ECAP (a) and to ECAP followed by IR (b)

The coefficient of strain rate sensitivity, *m*, is plotted on a semi-logarithmic scale as a function of strain rate at 350°C in Fig.6c. It is seen that the maximum m values are attained at an initial strain rate of 1.4×10^{-2} s⁻¹. Therefore, the 1421 Al subjected IR following ECAP exhibits high strain rate superplasticity.

Thus, the two-step process is feasible to fabricate thin sheets of 1421Al with the UFG structure. IR with a reduction of 88 pct. following ECAP with 4 passes provides the formation of the almost fully recrystallized structure. Therefore, the use of IR following ECAP with rectangular shape of channels allows strongly decreasing the total strain which is necessary to be imposed into the 1421Al [7] to provide extensive grain refinement. In other words the combination of ECAE with rectangular shape of channels and extensive IR provides a capability for achieving substantial grain refinement in this alloys and thereby producing thin sheets with UFG structure. It is worth noting that a moderate total strain of ~6 was imposed into the 1421 Al at 325°C by this two-step processing. Therefore, the combination of the ECAP and subsequent IR allows reducing the number of ECAP passages; the fully recrystallized structure is evolved at a total true strain (ϵ ~6) which is twice less comparing with conventional ECAP (ϵ ~12-16) [5-7].

Thus, IR following ECAP with rectangular shape of channels is suitable for the fabrication of sheets with superior superplastic properties from the high strength aluminum-lithium alloy containing Sc. The sheets of the 1421 Al can be used for industrial superplastic forming; high stability of superplastic flow taking place due to sufficient uniformity of UFG structure provides the fabrication of articles with a complex shape for airspace industry [8]. The present work clearly demonstrated that the heavily alloyed Al material can be easily processed into the UFG state via this technique.



Fig.6. Mechanical properties of the 1421 Al subjected to ECAP followed by IR. (a) effect of temperature on the true stress-true strain curves at an initial strain rate of $1.4 \times 10^{-2} \text{ s}^{-1}$; (b) the variation of the coefficient of strain rate sensitivity, *m*, with strain at 350°C and $\dot{\epsilon} \sim 1.4 \times 10^{-2} \text{ s}^{-1}$; (c) the variation of the coefficient of strain rate sensitivity, *m*, with strain rate at 350°C.

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