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Ultrafine-grained Al–0.2Sc–0.1Zr alloy: The mechanistic contribution of nano-sized precipitates on grain refinement during the novel process of accumulative continuous extrusion

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ABSTRACT

We elucidate the potential significance of nanosized, $Al_3(Sc, Zr)$, precipitates in obtaining ultrafine-grained structure in an Al–0.2Sc–0.1Zr alloy during the novel process, referred as accumulative continuous extrusion forming (ACEF). The grain size of the alloy was dramatically refined from 100 μm to 800 nm through continuous dynamic recrystallization (CDRX). The effectiveness of nanosized precipitates on CDRX was pronounced with increase in the ratio of the volume fraction (F_v) to the diameter (d) of the $Al_3(Sc, Zr)$ precipitates. Nanosized $Al_3(Sc, Zr)$, precipitates promoted grain refinement through three mechanisms: (i) the precipitates facilitated retention of high dislocation density in the alloy by promoting the generation of dislocation and pinning dislocation slip, which increased the driving force for CDRX, (ii) promoted the formation of deformation bands, providing sites for activation of CDRX, and (iii) activated CDRX near the grain boundary.

1. Introduction

Al–Sc–Zr alloys are new generation of metallic materials that provide high temperature creep resistance [1]. They are excellent candidates to replace high density iron-based alloys and titanium alloys for automotive and aerospace components that are exposed to intermediate temperatures (250–400 °C) and low pressure (<20 MPa) [2]. Scandium contributes significantly to strength through nanoscale coherent, Al_3Sc, precipitates [1]. The addition of ternary element, Zr, is added to Al–Sc alloy to further improve the mechanical properties and nanostructural stability [1,3].

Controlling grain size has long been recognized as a viable approach to design materials with desired properties [4]. For instance, grain refinement is the only approach that effectively increases both strength and impact toughness [5,6]. Severe plastic deformation (SPD) has attracted significant interest for obtaining ultrafine or nanograined structure [4,7–12]. Equal channel angular pressing (ECAP) is one of the most important SPD process [13–17]. However, ECAP process is not continuous or suitable for fabrication of large components [18]. Thus, conventional extrusion and ECAP were combined to produce long ZK60 magnesium alloy bars with an excellent combination of strength and ductility. However, the integration of the two methods is not continuous [19]. To make the process continuous, ECAP and continuous extrusion forming (CEF), referred as ECAP-CEF, were combined. In the ECAP-CEF process, a rod of the starting material is fed into the rotating roll, pushed forward, and sheared at an angle of 90°. After several passes, the grain size of the alloy is refined to several microns, and a material with ultrafine-grained (UFG) structure is generated [18,20]. UFG pure titanium- and aluminum alloys were prepared using the ECAP-CEF process [18–20]. Although ECAP-CEF is capable of producing long components with ultrafine grains, the approach cannot produce UFG material with different cross-sectional shape. Thus, we have developed an innovative process, referred as accumulative continuous extrusion forming (ACEF) that integrates and combines the advantages of ECAP-CEF and conventional extrusion. In this approach, an extrusion mold is connected in the same manner as in the ECAP-CEF to generate high strain in a single pass, replacing the extrusion mold in the final pass to fabricate long components with ultrafine-grained structure and of varied cross-section.

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Grain refinement during SPD processing is normally determined by the formation and migration of low angle grain boundaries (LAGBs) and high angle grain boundaries (HAGBs). Precipitates are known to affect the transformation from LAGBs to HAGBs and play an important role in grain refinement because they can pin dislocations and grain boundaries [7,21]. For example, the nanosized precipitates in Al alloy can accelerate grain refinement during SPD processing [22,23]. It is reported [24] that fine Al_6Mn precipitates in Al–Mg–Mn alloys promote the generation of dislocations, accelerating CDRX and grain refinement at 300 °C during ECAP. Barlow et al. reported that nanosized dispersoids of alumina platelets encouraged increase in dislocation density, restricted slip, and accelerated grain refinement during cold rolling [22]. While Troeger and Robson observed that the large particles (>1 μm diameter) stimulated recrystallization during SPD processing [25,26]. On the other hand, the large volume fraction of nanosized precipitates promoted the formation of shear bands and provided potential sites for recrystallization [27]. But low volume fraction of nanosized precipitates had insignificant effect on the formation of deformation bands [28]. In contrast, some researchers believe that precipitates may inhibit the transformation of LAGBs to HAGBs, thus retarding the formation of a UFG microstructure during SPD processing [29]. For example, Apps et al. [29] suggested that Al_3Sc precipitates with an average diameter of 20 nm in an Al–0.2Sc alloy homogenizes dislocation slip, reduces the rate of HAGBs at moderate strain, and retards grain refinement. On the other hand, Humphreys et al. [27] noted that the effect of precipitates on grain refinement during SPD depends on the ratio of the volume fraction (F_v) to the diameter (d) of the precipitates, i.e., F_v/d. However, evidence for such an observation is rare [30].

To date, the effect of the dispersoid on the formation of UFG structure has not been satisfactorily elucidated. In this study, Al–0.2Sc–0.1Zr (wt.%) alloys with different F_v/d values of Al_3(Sc, Zr) precipitates were prepared through a continuous semisolid extrusion process [31,32] followed by heat treatment. These alloys were then deformed through the novel ACEF process. The impact of Al_3(Sc, Zr) precipitates on grain refinement of Al–0.2Sc–0.1Zr (wt.%) alloy were systematically studied using electron-backscattered diffraction (EBSD) and transmission electron microscopy (TEM) and compared with pure Al processed in an identical manner using ACEF.

2. Experimental

2.1. Material preparation

Al–0.2Sc–0.1Zr alloy and pure Al (99.99 wt.%) wires were prepared by the continuous semisolid extrusion process, as previously described in detail [31]. After continuous semisolid extrusion processing, the Al–0.2Sc–0.1Zr alloy and pure Al were heat-treated at 300 °C for 0–10 h to obtain similar initial grain size. The annealed Al–0.2Sc–0.1Zr alloy was further heat-treated at 600 °C for 36 h, quenched in water at 15 °C, and then aged at 300 °C for 0–70 h [33,34] to obtain Al_3(Sc, Zr) precipitates with different F_v/d values.

Subsequently, the resulting Al–0.2Sc–0.1Zr alloy and pure Al, which had an average grain size of 100 μm (Fig. 1a and b), were fed into the entrance of the roll-shoe gap as starting materials and were processed by ACEF involving four passes. In the final pass, the extrusion molds for a round bar with a diameter of 10 mm was used to produce UFG material, as shown in Fig. 1c. During ACEF processing, the rotating speed of the extrusion roll was 7.5 m/min and the flow rate of the cooling water was 14 L/min. After each pass, the temperature was ∼300 °C at point 8 in Fig. 1c. During the ACEF processing, the wire was fed in the same direction after each pass.

2.2. Microstructure

To study the microstructure of Al–0.2Sc–0.1Zr alloy, specimens were collected from different positions (positions I–V) along the shear direction (SD) in the roll-shoe gap and extrusion mold, as shown in Fig. 1c. Additionally, Al–0.2Sc–0.1Zr alloy processed by ACEF process involving a numbers of passes was collected from the central area of the wires parallel to the extrusion direction (ED) after quenching. For electron back scattering diffraction (EBSD) studies, the collected specimens were ground and electropolished in an electrolyte consisting of 11% $HClO_4$ and 89% CH_3OH to remove the damage to the surface caused by grinding and mechanical polishing. Subsequently, EBSD measurements were carried out in the plane perpendicular to the transverse direction of the sheet using field emission scanning electron microscope (FE-SEM) (Zeiss Ultra 55, Carl Zeiss Microscopy, Jena, Germany). The operating voltage was 15 kV. The EBSD data was evaluated by orientation imaging microscopy (OIM, HKL-Channel 5) software. The misorientation angle quantification was used to identify LAGBs (between 2 and 15°) and HAGBs (>15°), as indicated by black and white lines, respectively, in the EBSD maps. The boundaries with an orientation difference <2° were excluded, and values above this threshold were counted as adjacent grains. The grains surrounded by HAGBs were referred as "grains", while the grains surrounded by LAGBs were referred to as "subgrains". The mean linear intercept method (ASTM E112) was used to measure the average diameter of the grains, excluding the subgrains. The fraction of recrystallized grains was calculated according to the following assumptions [35]. (1) If the angle of the boundaries in a grain exceeded 2°, the grain was classified as "deformed." (2) If the grains consisted of subgrains that had an internal misorientation that was lower than 2°, but the misorientation between subgrains was over 2°, the grain was classified as "substructure." (3) The remaining grains were considered to be recrystallized grains.

Specimens for TEM observation were cut from the annealed samples and the homogeneously deformed region of the samples after tensile straining and mechanically polishing from both the sides to a final thickness of 50 μm. Subsequently, foils of 3 mm diameter were punched and thinned using an ion beam thinner (Gatan, Inc., Pleasanton, USA). The TEM observations were performed using field-emission-gun (FEG) Tecnai G2 20 microscope (FEI, Oregon, USA) operating at an accelerating voltage of 200 kV. The dislocation density was estimated by counting individual dislocations crossing the thin foil surface [24].

3. Results

3.1. Characterization of Al–0.2Sc–0.1Zr alloy prior to ACEF processing

Bright-field TEM micrographs and selected area diffraction (SAD) patterns of Al–0.2Sc–0.1Zr alloy after different heat treatments are presented in Fig. 2. The significant aspect in Fig. 2 is the presence of a number of spherical precipitates in the matrix. With increase in aging time, the average diameter, volume fraction and F_v/d of the precipitates was gradually increased (Table 1). For example, the average particle size of the precipitates was ∼50 nm on aging at 300 °C for 2 h and was increased to ∼100 nm, after aging at the same temperature for 10 h. The corresponding ratio of F_v/d increased from $1.59 \times 10^{-5}\ nm^{-1}$ to $1.85 \times 10^{-5}\ nm^{-1}$ (Table 1). EDX analysis suggested that the precipitates mainly contained Al, Sc, and Zr with chemical composition in weight% of approximately 96 Al, 3.5 Sc, and 0.5 Zr at position A and ∼75.5 Al, 15 Sc, and 9.55 Zr at position B. The precipitates of these compositions were referred as Al_3(Sc, Zr) with a $L1_2$ structure similar to Al_3Sc [36].