Microstructure features and mechanical properties of a UFG Al-Mg-Si alloy produced via SPD

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Abstract. The effect of equal channel angular pressing in parallel channels (ECAP-PC) and subsequent artificial ageing on the microstructure and room temperature mechanical properties of the commercial aluminum alloys 6063 (Al-0.6Mg-0.5Si, wt.%) and 6010 (Al-0.8Mg-1.0Si-0.15Cu-0.25Mn, wt.%) was investigated. It was shown that mechanical strength of the ECAP-PC processed Al alloys is higher compared to that achieved in these alloys after conventional thermo-mechanical processing. Prior ECAP-PC solution treatment and post-ECAP-PC artificial aging can additionally increase the mechanical strength of both Al alloys. Under optimal artificial ageing conditions, the yield strength (YS) of 299 MPa and ultimate tensile strength (UTS) of 308 MPa was achieved in the 6063 alloy, whereas YS of 423 MPa and UTS of 436 MPa was achieved in the 6010 alloy.

1. Introduction

Metallic materials with nanocrystalline (NC) and ultrafine-grained (UFG) microstructures processed by severe plastic deformation (SPD) have been widely studied over the last two decades [1-3]. It is well known now that such materials can possess ultra-high mechanical strength at room and cryogenic temperatures [1-15]. Recent investigations have also revealed that their ductility [5, 6], crack resistance [8, 9], fatigue strength [10-12], etc. can also be simultaneously improved.

The most advanced SPD technique for fabrication of NC and UFG metallic materials is equal channel angular pressing (ECAP), where high shear deformation is induced into bulk billets, leading to microstructure refinement [2-4, 16]. Very high potential of ECAP technique for fabrication of aluminum alloys with UFG microstructures showing unique properties was demonstrated for the first time in [13]. In later investigations [9, 10, 14, 15], it was shown that the combination of ECAP processing with conventional heat treatments can significantly enhance mechanical strength and ductility of Al alloys.

Solid solution hardening (via alloying), precipitation hardening (via ageing), and dislocation hardening (via thermo-mechanical processing) were considered as the main approaches to improve mechanical strength of Al. Rapid development of SPD processing showed that grain refinement down to ultra-fine scale can be the most efficient strategy to improve mechanical strength of the Al alloys [17]. The main objective of the present investigation is to fabricate...
high strength commercial aluminum alloys 6063 and 6010 via equal channel angular pressing with parallel channels (ECAP-PC) followed by artificial ageing. The ECAP-PC technique is a modification of standard ECAP. It is characterized by higher efficiency and lower wastage of material compared to the standard ECAP [18].

2. Experimental procedures

The heat-treatable 6063 (Al-0.6Mg-0.5Si, wt.%) and 6010 (Al-0.8Mg-1.0Si-0.15Cu-0.25Mn, wt.%) alloys were chosen as materials for this investigation. Both materials have been widely used in various sectors of engineering. Billets with a cross section of 18 mm x 18 mm and a length of 100 mm were machined from both alloys. They were subjected to solution treatment at 535°C for 2h and quenched in water. As-quenched billets were subjected to ECAP-PC processing at 100°C for 4 passes. One ECAP-PC pass corresponds to two conventional ECAP passes, since sample undergoes two shear events in one ECAP-PC pass. The angle of ECAP-PC die was 100° and the distance between axes of parallel channels, K, was 18 mm (Fig. 1). More detailed information about ECAP-PC processing technique can be found in [18-20]. Billets after ECAP-PC processing were artificially aged at 130°C up for 24 hours.

Transmission electron microscopy (TEM) studies of the microstructure were carried out using JEM-2000 EX microscope operating at 200 kV. The microscope is equipped with a digital camera OLYMPUS D71. The specimens for TEM analysis were cut from longitudinal and transversal sections. Observations were made in both bright and dark field modes, and selected area electron diffraction (SAED) patterns were taken from the areas of interest using an aperture of 1 mm nominal diameter. The linear intercept method was used to estimate the size of grains/subgrains and the size of least 300 grains was measured.

Tensile specimens with a gauge diameter of 3 mm and a gauge length of 15 mm were machined from the ECAP-PC processed billets. Tensile tests were conducted at room temperature at an initial strain rate of 5.0 x 10^-4 s^-1 using an INSTRON 1185 testing system. Yield strength (σ0.2), ultimate tensile strength (σUTS), elongation to failure (δ), and area reduction (ψ) were determined. At least three specimens were tested for each material's condition and the results were found to be reproducible.

Figure 1. a) Schematic presentation of ECAP-PC processing die: d – diameter of channels, K – distance between axes of parallel channels, Φ – intersection angle between parallel channels and the connecting channel; b) ECAP-PC processing.
3. Results and discussions

**Microstructure of the ECAP-PC processed materials**

Figure 2 illustrates typical microstructures of the 6063 alloy after ECAP-PC processing at 100°C for 4 passes. Formation of microstructure consisting mainly of ultra-fine grains is observed. This is also confirmed by SAED patterns. Elongated grains having a length of 560 nm and aspect ratio of ~2 are seen on longitudinal section (Fig. 2a). The grains are elongated along the shear plane in last ECAP-PC pass. On transversal cross section, ultra-fine grains having an average size of 515 nm and aspect ratio of ~1.5 are observed. Nanosized spherical second phase precipitates having an average size of 10 nm are observed in the interior of ultra-fine grains (Fig. 2a).

![Figure 2. Microstructure of the 6063 alloy after ECAP-PC processing at 100°C for 4 passes: a) longitudinal section, b) transversal section.](image)

![Figure 3. The needle-like β′-Mg2Si precipitates in the grain interior of the 6063 alloy after ECAP-PC processing at 100°C for 4 passes followed by artificial aging.](image)
According to the earlier studies on the SPD processed Al-Mg-Si alloys [21-23], these nanosized precipitates can be identified as $\beta'$-Mg$_2$Si precipitates. They are formed during ECAP-PC processing due to dynamic aging resulting in decomposition of supersaturated solid solution [21-24].

**Mechanical properties of the ultra-fine grained Al alloys**

**Mechanical strength and ductility**

The results of mechanical tensile testing of the studied Al alloys are listed in Table 1. It is seen that ECAP-PC processing of the studied materials has increased their mechanical strength. In the 6063 alloy, yield strength has increased by 15 % and ultimate tensile strength by 20 %, whereas in the 6010 alloy, these properties have increased by 30 and 40 %, respectively (Table 1). The artificial aging leads to further improvement of mechanical strength. For example, in the 6010 alloy, yield strength has increased by 65 % compared to that of the coarse-grained material. Such an increase of mechanical strength can be related to combination of solid solution hardening with grain size hardening, as well as precipitation hardening.

**Table 1. Mechanical properties of the studied Al alloys.**

| Processing                                | $\sigma_{\text{UTS}}$, MPa | $\sigma_{0.2}$, MPa | $\delta$, % |
|-------------------------------------------|-----------------------------|---------------------|-------------|
| 6063 alloy                                |                             |                     |             |
| ECAP-PC at 100 °C for 4 passes            | 264 ± 4                     | 256 ± 5             | 12.5 ± 0.5  |
| ECAP-PC at 100 °C for 4 passes + AA       | 308 ± 7                     | 299 ± 6             | 15.5 ± 0.5  |
| Coarse-grained material, T6$^1$           | 200 ± 5                     | 170 ± 6             | 14.2 ± 0.3  |
| 6010 alloy                                |                             |                     |             |
| ECAP-PC at 100 °C for 4 passes            | 408 ± 6                     | 386 ± 9             | 11.0 ± 0.3  |
| ECAP-PC at 100 °C for 4 passes + AA       | 436 ± 1                     | 423 ± 3             | 12.1 ± 1.3  |
| Coarse-grained material, T6$^1$           | 295 ± 5                     | 226 ± 5             | 12.0 ± 1    |

$^1$– T6 is a standard thermal treatment: annealing at 540°C, quenching in water, and artificial aging at 160°C for 12 h.

**Effect of chemical composition on the mechanical properties of the UFG Al-Mg-Si alloys**

As it was mentioned above, there are a few strengthening mechanisms acting in the studied UFG Al alloys. Total strength of the UFG Al alloys can be determined as

$$\sigma = \sigma_o + \Delta\sigma_{\text{ss}} + \Delta\sigma_p + \Delta\sigma_{\text{gs}} + \Delta\sigma_d,$$

(1)

where $\sigma_o$ is the Peierls-Nabarro stress, $\Delta\sigma_{\text{ss}}$ contribution of solid solution hardening, $\Delta\sigma_{\text{gs}}$ contribution of grain size hardening, and $\Delta\sigma_d$ contribution of dislocation hardening.

There are theoretical approaches to estimate contribution of each strengthening mechanism based on chemical composition or microstructural parameters [17]. In this work, we employed another approach to estimate contribution of individual strengthening mechanisms. Additional
tensile testing of coarse-grained annealed pure Al, coarse-grained solution treated Al alloys, and coarse-grained solution treated and artificially aged Al alloys was performed. For each Al alloy, the $\Delta\sigma_{ss}$-parameter was calculated as a difference in yield strength of this coarse-grained Al alloy and coarse-grained pure Al. The $\Delta\sigma_p$-parameter was calculated as a difference in yield strength of ECAP-PC processed Al alloy and ECAP-PC processed + artificially aged Al alloy. The $\Delta\sigma_{gs} + \Delta\sigma_d$ was calculated as a difference in yield strength of ECAP-PC processed Al alloy and coarse-grained solution treated Al alloy. The obtained results are listed in Table 2.

Table 2. Contribution of strengthening mechanisms into total yield strength of the Al-Mg-Si alloys [MPa].

| Strengthening mechanisms                                      | 6063 | 6010 |
|---------------------------------------------------------------|------|------|
| Solid solution hardening, $\Delta\sigma_{ss}$                | 30   | 45   |
| Precipitation hardening, $\Delta\sigma_p$                    | 43   | 37   |
| Grain size hardening + dislocation strengthening, $\Delta\sigma_{gs} + \Delta\sigma_d$ | 206  | 266  |

It is clearly seen that grain refinement via ECAP-PC processing provides the highest strengthening in both studied Al alloys (206 MPa in the 6063 alloy and 266 MPa in the 6010 alloy) followed by precipitation strengthening and solid solution hardening (Table 2).

4. Summary

The 6063 and 6010 alloys were subjected to solution treatment followed by ECAP-PC processing and artificial aging. ECAP-PC processing of both alloys resulted in formation of homogeneous ultra-fine grained microstructure containing nanoscale $\beta'$-Mg$_2$Si precipitates formed due to dynamic aging. Artificial aging of both alloys led to further decomposition of solid solution resulting in formation of needle-like nanoscale $\beta''$-Mg$_2$Si precipitates. It was demonstrated that mechanical strength of the ECAP-PC processed Al alloys is much higher compared to that achieved in these alloys after conventional thermo-mechanical processing. Analysis of strengthening mechanisms acting in the UFG Al-Mg-Si alloys showed that grain size hardening and dislocation strengthening provide the highest contribution into total strength.

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