Proceedings of the 12th International Conference on
Aluminium Alloys, September 5-9, 2010, Yokohama, Japan
©2010 The Japan Institute of Light Metals
pp. 1720-1725

Microstructure Behavior of Al-Mg-Si Alloy Processed by ECAP And Its Thermal Stability

Tomáš Kovářík[1], Jozef Zrník[2]

[1] ZČU, FST, Department of Material Science and Engineering, Univerzitní 22, Plzeň, Czech Republic
[2] Comtes FHT Ltd., Průmyslová 995, Dobřany, 334 41, Czech Republic

Abstract

Methods of severe plastic deformation of ductile metals and alloys offer the possibility of processing engineering materials to very high strength with good ductility. After typical amount of processing strain a material with submicron grain size is obtained with boundaries of rather low misorientation angles and grains containing a high density of dislocations. In the present study an Al–Mg–Si (EN AW 6082) alloy was severely plastically deformed by equal channel angular pressing (ECAP) to produce such a material. Aluminium alloy prepared in three different initial states of structure was processed by the ECAP deformation technique at room and increased temperature in two different modes. Substructure and grain refinement of specimens were investigated by TEM of thin foils after successive application of different passes corresponding to a different effective true strain. Hardness and tensile measurements were used to observe the evaluation of mechanical properties. Structure stability and changes in mechanical properties related to the applied strain were studied at two elevated temperatures 270 and 350 °C. The effect of annealing was monitored by structural changes and hardness measurements at different time holds (hours) and even at shorter exposition time of 10 and 20 minutes.

Keywords: *Severe plastic deformation, Ultrafine structure, ECAP method.*

1. Introduction

There is the current major commercial interest in the development of aluminium alloys with ultra-fine grain (UFG) structures for structural applications [1, 2]. Prangnell et al. [3] proved the use of high strain deformation to improve the properties of materials through grain refinement. It can be applied for simple non-heat treatable alloys to obtain strength levels equivalent to much more highly alloyed materials [4]. Such high strains can be currently obtained using specialized process like the Equal Channel Angular Pressing (ECAP). ECAP is a severe plastic deformation (SPD) processing technique first described by Segal et al. [5]. It has been proposed by Humphreys et al. [6] that during deformation at conventional strains second-phase particles can increase the rate of dislocation generation and develop surrounding deformation zones in the matrix that contain large local misorientation gradients. New high angle boundaries can be formed within such deformation zones at relatively low strains [7]. The previous research has also shown that at strains approaching the limits $\varepsilon_{vm} \sim 4$ submicron grain structures started to form in aluminium alloys containing significant volume fractions of micron-scale second-phase particles [8]. Pragnell [3] has published that for a severely deformed material a true submicron grain structure (the average spacing of boundaries misorientated by >15° in all directions), should be < 1μm and the fraction of the high angle boundary (HAB) area, relative to the total boundary area, should be >70%. The interest of this work is focused on substructure development in dependence on the introduced strain by ECAP (details vide paragraph 2).

2. Experimental proceduress

The investigation was conducted by using commercial aluminium alloy EN AW 6082. The chemical composition was corresponded to the norm EN 573 – 3 (noted in *Table 1*). The rod of 6082 with a diameter of 40 mm was received after continual hot extrusion. The initial state is according to the norm ČSN EN 515, F.

Table 1. The chemical composition of material (according to the norm EN 573 – 3).

Elem.	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
(%)	0.70 – 1.30	max 0.50	max 0.10	0.40 – 1.0	0.60 – 1.20	max 0.25	max 0.20	0.1	rest

The whole experimental programme was divided into two regimes. The first regime was titled 4P by virtue of four passes of aluminium alloy specimen going through the ECAP die. The second one was called 6P on grounds of six passes (detailed descriptions see below).
The billets for regime 4P were cut from the center part of aluminium rod with dimension of 8 × 8 × 30 mm. Samples for regime 6P were prepared in the same way with a geometry l = 50 mm, Ø 8 mm (circular cross-section).

2.1 Specifications of regime 4P

There were determined three states of pre-deformation heat treatments for experimental samples from aluminium alloy:

S1 – initial state (represents structural conditions corresponding to mechanical fabrication of rods),
S2 – annealed state (at 540 °C for 1.5 h followed by water quenching),
S3 – quenched and aged state (by temper hardening at 160 °C for 12 h).

The deformation process was realized after heat treatment. The experimental samples in the state of S1, S2, S3 were subsequently extruded 1 – 4 times by ECAP die applying route B_C at room temperature. The ECAP geometry was defined by intersection angle Φ equal to 90° and inner addition angle Ψ equal to 20°. *Fig. 1* shows a schematic illustration of ECAP deformation procedure. The effective strain corresponding to one pass was $\varepsilon \sim 1.05$. Four passes corresponded to the total strain of $\varepsilon_{ef} \sim 4.2$.

The experimental samples were consequently exposed to heat treatment at 270 °C and 350 °C for 0.5 h, 2 h, 4 h, 6 h (all states, 1 – 4 passes). The effect of annealing was monitored by structural changes and hardness measurements even at shorter exposition times of 10 and 20 minutes.

2.2 Specifications of regime 6P

The experimental samples in states $S1_{6P}$, $S2_{6P}$ and $S3_{6P}$ (equivalent marking to 1.1) were subsequently extruded 1 – 6 times by ECAP die applying route B_C at elevated temperature of 150 °C. The ECAP die used for the experiment was heated to pressing temperature and held for 30 minutes. The sample was tempered for 300 s prior to pressing, which was done inside the pre-heated die until samples reached the pressing temperature.

The ECAP geometry was defined by intersection angle Φ equal to 120° and inner addition angle Ψ equal to 20°. The effective strain corresponding to one pass was $\varepsilon \sim 0.67$. The six passes corresponded to the total strain of $\varepsilon_{ef} \sim 4.02$.

The experimental samples were consequently exposed to heat treatment at 270 °C for 1, 2, 5, 10 and 20 minutes.

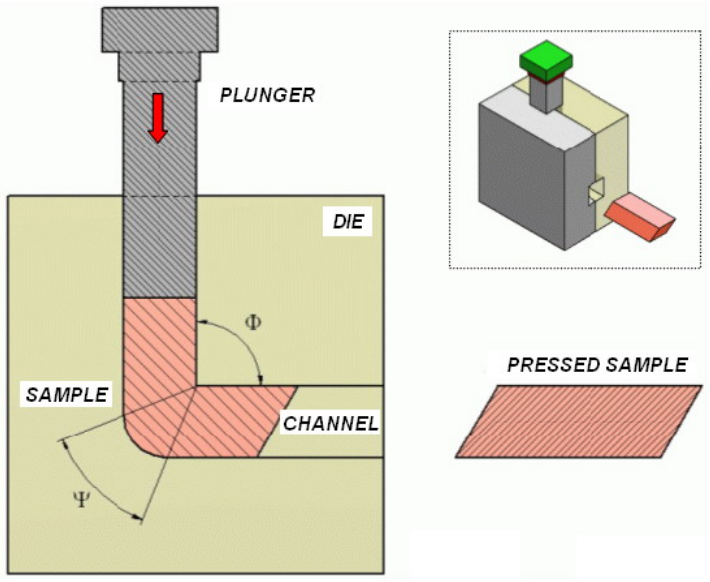

Fig. 1. Illustration of ECAP deformation procedure.

Microstructures of ECAP deformed samples were examined by light microscopy (LM) and transmission electron microscopy (TEM). Thin foils for TEM observation were prepared normal to the longitudinal axis of ECAP pressed billets. The microstructures were obtained by using JEOL JEM 200FX TEM operating at 200 kV. Selected area electron diffraction (SAED) was used to investigate the ultra-fine grain structure evolution depending on introduced strain. The hardness and tensile tests were carried out using MTS universal testing machine equipped with Multisens extensometer. Tensile specimens with gauge length of l_0= 25 mm were tested at a constant cross-head speed of 0.016 mm.s^{-1} until failure. The engineering stress-strain curves were constructed.

3. Experimental results

3.1 Microstructure after ECA pressing

The initial grain size of the specimen in the as-received conditions was determined as 150 μm ± 5 μm. Transmission electron microscopy observation of the material after ECAP revealed multifarious deformed microstructures which involved areas of dislocation network, substructure formations and dislocation cells, polygonized subgrains and new locally UFG structure with high angle boundary orientation.

Coarse original grains were rapidly subdivided at low strains by the formation of new high-angle boundaries at the boarders of primary deformation bands. The state after 2 passes was represented as combination of nascent oriented deformation bands and dense dislocation network (*Fig. 2*). New grain evolution with a mixture of high-angle and low-angle boundaries was observed in the centre of extruded billet. At a finer scale there were examined a rare small grains within minimum size 70 nm (*Fig. 2, detail*).

As the strain was increasing a progressively more fibrous microstructure was developing. High-angle boundaries orientation became predominantly aligned into the deformation direction. Continued subdivision (which can generate further lamella boundaries) of some deformation bands was appeared. Structure development after four passes in regime 4P (state S3) was assessed as heterogeneous microstructure with forming ultra-fine grained structure and high rate of submicron grains (*Fig. 3*). Deformation substructure had at low to medium strains conditions still heterogeneous character.

Heterogeneous deformation structure was observed with an equiaxed dislocation cell structure and fine substructure formations in initial state S1 after six passes at elevated temperature. A new locally high-angle boundary grains were appeared within the range of 100 – 200 nm (*Fig. 4*). The effect of the elevated deformation temperature had the main influence (in all states) on initiation and progress of polygonization process and also nucleation of new sub-grains (*Fig. 5*).

The increased temperature of ECAP regime 6P contributed in transformation deformed structure to a dynamic recovery process.

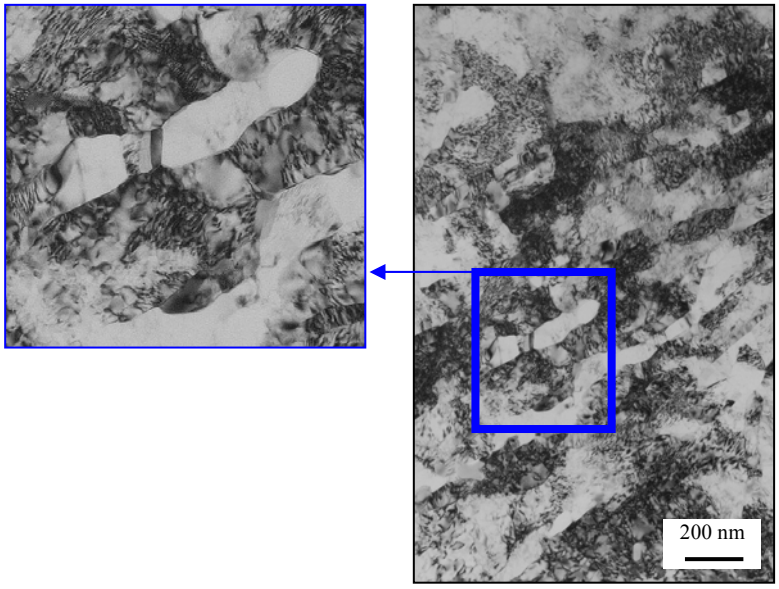

Fig. 2. Bounded deformation structure (S2, 2P - detail).

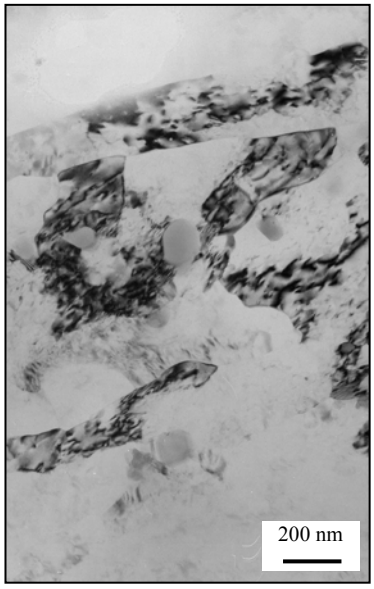

Fig. 3. New local UFG structure (S3, 4P).

It was also found that dissimilar regions of the material displayed different rates of recovery. The results of analysis confirmed that the microstructure development was not influenced by the initial microstructure conditions resulting from preliminary pre-deformation heat treatments. TEM provided the evidence that applied effective strain at regime 6P was not sufficient to deform structure uniformly and to produce grains having higher fraction of high-angle boundary.

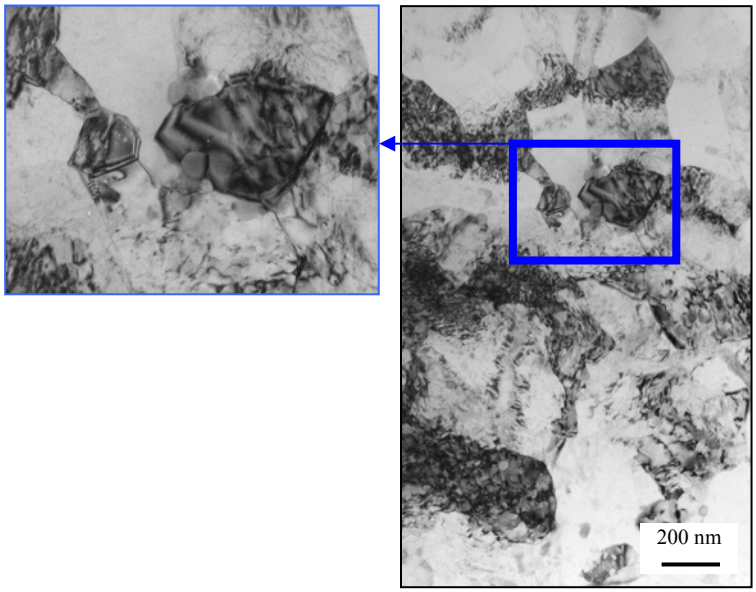

Fig. 4. Small grains with high density of dislocations

Fig. 5. Evolvement of sub-grain structure ($S3_{6P}$, 6P).

This was later confirmed by EBSD analysis. In the EN AW 6082 alloy approximately *28%* of the total boundary area was classed as being high angle (i.e. >15° misorientation) after just six ECAP passes at elevated temperature ($S3_{6P}$). In comparison, the rate of formation of HAGB in the state after four passes ($S3_{4P}$) is very similar about *27%* (total boundary area).

3.2 Tensile properties after ECA pressing

The results of tensile testing performed at room temperature are shown in *Table 2* (next page), which represents all three states in two deformation regimes. It means results after four and six passes of samples through ECAP die (detailed description s. 2.1). Similar course of stress–strain curves is monitored in states S1 and S2 (*Fig. 6*). Very specific strengthening and enhancement of tensile strength value compared to non-deformed alloy state about 175% is observed (*Table 2*, green marks). Distinct shortening of deformation section in the case of state S3 is attributed to influence of secondary phase which is precipitating during pre-deformation heat treatment. The yield strength value is as compared to initial state higher about 320 % (*Table 2*, blue mark).

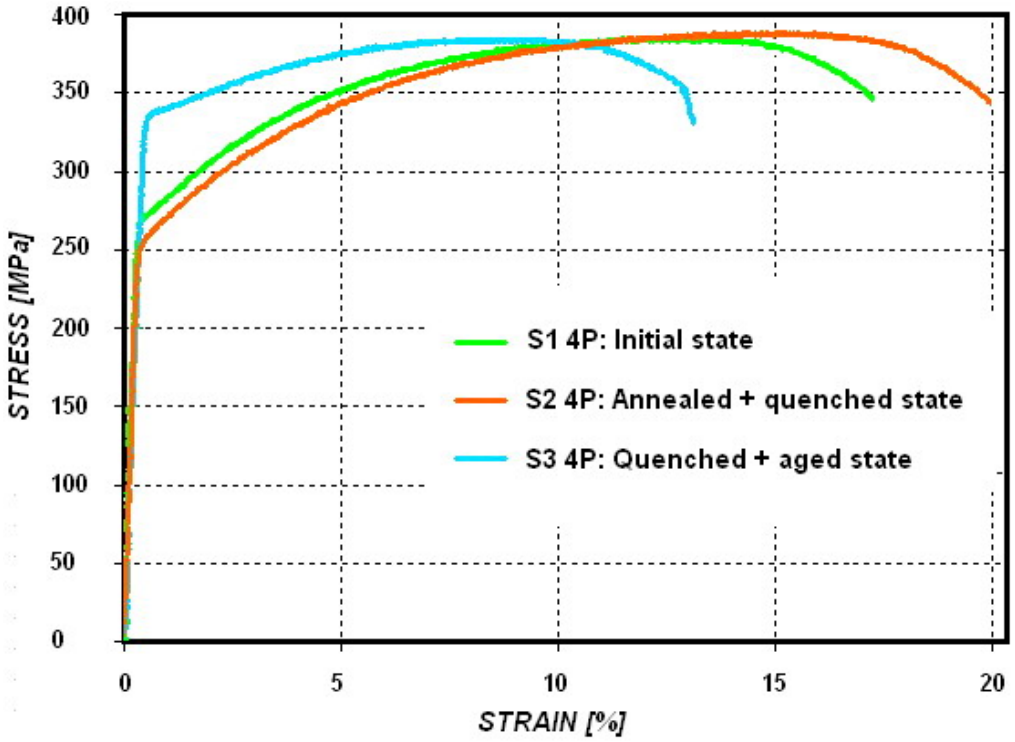

Fig. 6. Engineering stress-strain curves for regime 4P.

Significant strengthening is evident in the state S3 after six passes (regime 6P). The yield strength of the third state (subsequent aged) ECAPed alloy sample is equal to 370 MPa (*Table 2*, yellow mark). The YS is higher round 430 % than value YS data of the un-ECAPed material (70 MPa). The tensile strength of state S3 (6P) is significantly higher than values in the other stages (*Table 2*, orange mark). There is a possibility of the presence of additional mechanisms as solution hardening, deformation hardening (the strengthening effect from ECAP processing) and the precipitation hardening. This is due to the fact that the amount of Mg and Si is presented in supersaturated solution. These elements are essential to form the GP zones and hardening particles of β-Mg_2Si phase precipitated during ageing process [9]. Increased value of TS can be also caused by inception of secondary precipitation during deformation process ECAP at elevated temperature. These mechanisms are probably responsible for lower value of residual ductility.

Table 2. Mechanical properties of commercial 6082 aluminium alloy after ECAP;
IC – initial conditions; *SX* – pre-deformation heat treatment state;
4P, *6P* – number of passes depending on deformation regime (vide 2.1).

spec. AlMgSi1	D_0 *[mm]*	D_U *[mm]*	S_0 *[mm²]*	S_u *[mm²]*	L_0 *[mm]*	L_U *[mm]*	*E [GPa]*	$Rp_{0.2}$ *[MPa]*	*Rm [MPa]*	A_5 *[%]*
IC state	5.02	-	19.78	-	25.00	-	-	80	140	18
$S1_{4P}$ *state*	5.03	4.18	19.87	13.72	25.00	29.81	89.3	271.5	385.8	19.2
$S2_{4P}$ *state*	5.04	4.16	19.95	13.59	25.00	30.15	85.4	257.8	388.2	20.6
$S3_{4P}$ *state*	5.02	4.27	19.79	14.32	25.00	28.54	73.7	336.82	384.36	14.16
$S1_{6P}$ *state*	5.03	3.95	19.87	12.25	25.00	27.95	58.64	302.01	321.90	11.80
$S2_{6P}$ *state*	4.95	3.61	19.24	10.24	25.00	28.74	73.33	178.93	208.06	14.96
$S3_{6P}$ *state*	5.04	3.75	19.94	11.04	25.00	27.57	64.56	369.92	394.04	4.18

4. Summary and conclusions

a) TEM investigation demonstrated heterogeneous multifarious deformed microstructure after four and six passes in both regimes.
b) It was proved that total introduced deformation $\varepsilon_{vm} \sim 4$ (in both regimes) wasn't enough for formation of uniform UFG structure with a higher fraction of HAB.
c) Initial states of microstructures in both regimes (vide S1, S2, S3 heat treatments) had no effect on structure development during and after deformation process.
d) Expressive enhancement of strength properties in states $S3_{4P}$ and $S3_{6P}$ is attributed to precipitation of phase β´´ - β´ Mg_2Si.
e) Degradation of deformation structure was started after 5 minutes at 270 °C. Essential influence of β´´ - β´ Mg_2Si to higher thermal stability wasn't observed.

References

[1] R. Z. Valiev, I. V. Islamgaliev: Progress in Materials Science 45 (2000) 103.
[2] R. Z. Valiev, T. G. Langdon: Principles of equal-channel angular pressing as a processing tool for grain refinement, Progress in Materials Science 51 (2006) 881–981.
[3] P. B. Prangnell, J. R. Bowen, P. J. Apps: Ultra-fine grain structures in aluminium alloys by severe deformation processing, Materials Science and Engineering A 375–377 (2004) 178–185.
[4] J. S. Hayes, R. Keyte, P. B. Prangnell: Mater Sci and Tech 16 (2000) 1259.
[5] V. M. Segal, et al.: Russ. Metall. 1 (1981) 99-105.
[6] F. J. Humphreys: Recrystallization and related annealing phenomena, Pergamon, (1995).
[7] F. J. Humphreys: Acta Metal 27 (1979) 1801.
[8] H. Jazeri, et al., *Proceedings of 5th Int. conf. on Recrystallization and Related Annealing Phenomena*, (Achen, Belgium, 2001), pp. 549.
[9] G. Mrowka, et al.: Influence of heat treatment on the microstructure and mechanical properties of 6005 and 6082 aluminium alloys, Journal of Materials Processing Technology 162–163 (2005) 367–372.