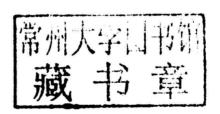
## **Materials Research and Application**



Edited by Mihai Branzei and Iulian Vasile Antoniac

# Materials Research and Application

Selected, peer reviewed papers from the 5<sup>th</sup> International Conference on Materials Science and Technologies (RoMat 2014),
October 15-17, 2014, Busharest, Romania



Edited by

Mihai Branzei and Iulian Vasile Antoniac



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### **Preface**

The aim of RoMat 2014 conference is to provide a platform for researchers, engineering, academicians as well as industrial professional from all over the world to present the research results and development activities in Materials Science and Engineering Technology. Held every two years, the conference provides opportunities for the delegates to exchange new ideas and application experiences face to face, to establish business or research relations and to find global partners for future collaboration. Young scientists are welcome to actively contribute to the conference. RoMat 2014 was the 5<sup>th</sup> in a series of successful conferences on Materials Science and Technologies, which takes place every two years. During the conference many plenary and invited lectures were presented, as well as oral presentations and poster sessions. RoMat offers a opportunity for the worldwide Materials Science Community to share their recent achievements and to provide a forum for academic scientists and industrial researchers to meet and exchange valuable experiences.

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## **CHAPTER 1:**

**Materials Properties and Application** 

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## Microstructural Refinement in a Commercial Aluminum Alloy Processed by ECAP using a Die Channel Angle of 110 Degrees

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**Keywords:** aluminum alloys, equal channel angular pressing, severe plastic deformation, ultrafine grained materials, metallographic analysis.

Abstract. The properties of ultra-fine grained materials are superior to those of corresponding conventional coarse grained materials, being significantly improved as a result of grain refinement. Equal channel angular pressing (ECAP) is an efficient method for modifying the microstructure by refining grain size via severe plastic deformation (SPD) in producing ultra-fine grained materials (UFG) and nanomaterials (NM). The grain sizes produced by ECAP processing are typically in the submicrometer range and this leads to high strength at ambient temperatures. ECAP is performed by pressing test samples through a die containing two channels, equal in cross-section and intersecting at a certain angle. The billet experiences simple shear deformation at the intersection, without any precipitous change in the cross-section area because the die prevents lateral expansion and therefore the billet can be pressed more than once and it can be rotated around its pressing axis during subsequent passes. After ECAP significant grain refinement occurs together with dislocation strengthening, resulting in a considerable enhancement in the strength of the alloys. A commercial AlMgSi alloy (AA6063) was investigated in this study. The specimens were processed for a number of passes up to nine, using a die channel angle of 110°, applying the ECAP route B<sub>C</sub>. After ECAP, samples were cut from each specimen and prepared for metallographic analysis. The microstructure of the ECAP-ed and as-received material was investigated using optical (OLYMPUS - BX60M) and SEM microscopy (TESCAN VEGA II - XMU). It was determined that for the as-received material the microstructure shows a rough appearance, with large grains of dendritic or seaweed aspect and with a secondary phase at grain boundaries (continuous casting structure). For the ECAP processed samples, the microstructure shows a finished aspect, with refined, elongated grains, also with crumbled and uniformly distributed second phase particles after a typical ECAP texture.

#### Introduction

Over the last decades, metals and alloys with submicrometer grain sizes were in the spotlight of researchers, academics and scientists from around the world. In most cases, the properties of an ultrafine grained (UFG) material are superior to those of the corresponding conventional coarse grained material; the strength, hardness and ductility of UFG materials being significantly improved as a result of grain refinement. A particular set of techniques that can lead to a reduction of the material grain size up to submicrometer or nanometer range are based on severe plastic deformation (SPD), a field of materials science recording a continuous development and striking innovations. Formally, processing by SPD may be defined as those metal forming procedures in which a very high strain is imposed on a bulk solid without the introduction of any significant change in the overall dimensions of the solid and leading to the production of exceptional grain refinement, with grain sizes mostly in the submicrometer or even in the nanometer range [1]. This significant grain refinement obtained by SPD leads to the improvement of mechanical, microstructural and physical properties for the processed material [2].

ECAP processing consists mainly of pressing test samples (generally square or round) through a die containing two channels that are equal in cross section (and also identical to the workpiece cross-section) and intersect at a certain angle. Grain refinement is obtained as a result of the imposed deformation by simple shear which occurs throughout the theoretical shear plane. Despite the introduction of a very intense strain as the sample passes through the shear plane, the sample ultimately emerges from the die without experiencing any change in the cross-sectional dimensions. Since the cross-sectional area remains unchanged, the same sample may be pressed repetitively to attain exceptionally high strains and an advanced microstructure refinement. In ECAP processing a sample is pressed through a die in which two channels of equal cross section intersect at an angle of  $\phi$ . An additional angle of  $\psi$  defines the arc of curvature at the outer point of intersection of the two channels, as shown in Fig. 1, a. In this figure, ABC is the plastic deformation zone (PDZ) and the x, y and z planes denote the transverse plane, the flow plane and the longitudinal plane, respectively. Considering the billet rotation, different processing routes are possible: route A with no rotation of the billet between consecutive passes; route BA when the billet is rotated counter clockwise 90° on even number of passes and clockwise 90° on odd number of passes; route B<sub>C</sub> when the billet is rotated counter clockwise 90° after every pass (Fig. 1, b); and route C with the billet rotated 180° after every pass [3]. It was shown that in ECAP processing, from all the possible deformation routes, the optimum solution is represented by route  $B_C$  [4-8].

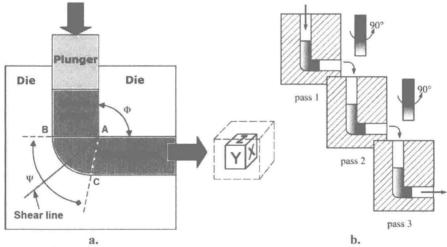


Fig. 1. Illustration of ECAP: a. the principle of ECAP processing; b. ECAP route B<sub>C</sub>.

#### Materials and methods

A commercial aluminum alloy (AA6063) was investigated in this study. The chemical composition (wt%) of the AA6063 alloy used in the experiments was: Si 0.467, Mg 0.488, Fe 0.602, Cu 0.103, Mn 0.086, Zn 0.133, Ti 0.012, Pb 0.012, Ni < 0.003, Cr < 0.009, balance Al. The Al-6063 specimens were obtained from 100 cm round billets stock, obtained by continuous casting and heat treated. The specimens were machined such that the specimen axis was perpendicular to the continuous casting direction of the billets. The specimens machining for the ECAP processing were performed using an abrasive cutter Metkon SERVOCUT M 300 to cut specimens from the initial continuous casted billets. The final specimens shape for the ECAP process was 60 x 9.6 x 9.6 mm. The ECAP die [9] (see Fig. 2, a and b) had a channel angle of  $\phi = 110^{\circ}$  and a corner angle of approximately  $\psi = 20^{\circ}$ . As lubricant graphite powder was used. The specimens were pressed at room temperature for up to nine passes using a hydraulic press (200 tf) and a pressing speed of 10 mm/s (see Fig. 2, c). The processing route B<sub>C</sub> was applied, meaning that the sample was rotated counter clockwise 90° after every pass (see Fig. 1, b).

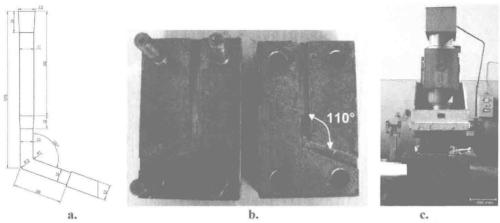


Fig. 2. ECAP experimental setup: a. die channel geometry; b. the ECAP 110° die-set; c. the hydraulic press used in the experiments.

From the as-received and the ECAP-ed specimens, samples for metallographic analysis were cut. For the samples subject to ECAP, the cutting direction was parallel to the ECAP direction, in such a manner that the microstructure of the specimen can be examined for the longitudinal plane (direction). A precision cutter Metkon MICRACUT 200 was used to obtain samples with approximately 10 x 7 x 5 mm dimensions and with a very good surface finish. All samples were hot mounted using a BUEHLER SIMPLIMET 1000 automatic mounting press and each of them was subject to grinding and polishing, using a BUEHLER PHOENIX 4000 – BETA/1 SINGLE semi automatic grinder/polisher. All specimens were etched for 20 seconds using Keller's reagent. The microstructure of the ECAP processed and also for the as-cast material was investigated using both optical (OLYMPUS – BX60M) and SEM microscopy (TESCAN VEGA II – XMU).

#### Results and discussion

The accumulated equivalent strain values were calculated using the die channel and relief angles in Eq. 1 [10] were N is the number of passes,  $\phi$  is the channel angle and  $\psi$  is the corner angle.

$$\varepsilon_N = N \cdot \frac{1}{\sqrt{3}} \left[ 2 \operatorname{ctg} \left( \frac{\phi}{2} + \frac{\psi}{2} \right) + \psi \operatorname{csc} \left( \frac{\phi}{2} + \frac{\psi}{2} \right) \right]. \tag{1}$$

According to Eq. 1 the equivalent strain depends on both  $\phi$  and  $\psi$  angles. It decreases when  $\psi$  increases and the maximum ( $\varepsilon_N \sim 1.15$ ) is obtained for  $\phi = 90^\circ$  and  $\psi$  close to zero [10-12]. Eq. 1, proposed by Iwahashi et al. [10], is an analytical expression for calculating the equivalent strain imposed in each ECAP pass only in terms of die geometric parameters. The assumptions in this geometric analysis include simple shear, a frictionless die surface, an uniform plastic flow on a plane, a complete filling of the die channel by the workpiece and a rigid perfectly plastic material (no strain hardening behaviour is included). With this assumptions, Eq. 1 doesn't take into account for the effect of friction, strain hardening, strain distribution and deformation gradient, providing a homogeneous value of strain in the whole workpiece.

For a channel angle of  $\phi = 110^\circ$  and a corner angle of approximately  $\psi = 20^\circ$ , the equivalent strain for each pass subjected to each specimen is about 0.76. Using Eq. 1 the variation of accumulated equivalent strain was calculated for 1, 3, 6 and 9 ECAP processing passes, Fig. 3 showing that the variation of accumulated equivalent strain is linear dependent to the number of passes, the slop of variation is a function of die geometry, defined by  $\phi$  the channel angle and  $\psi$  the corner angle.

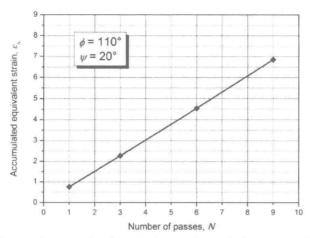


Fig. 3. Calculated accumulated equivalent strain evolution vs. number of passes.

The micrographs obtained by means of optical microscopy and also via scanning electron microscopy (SEM) for all investigated samples (as-cast, one pass, three, six and respectively nine passes ECAP processed material) are shown in Fig. 4 to Fig. 8, respectively. All results for all experiments are based on the current configuration of the specimen.

The microstructure of the as-cast AA6063 aluminum alloy is given in Fig. 4. From this figure one can observe that the microstructure consists of large grains in which the dendritic or seaweed segregation structure is clearly seen (specific continuous casting structure). Also, it can be observed that some compounds are present in the alloy at grain boundaries as secondary phase.

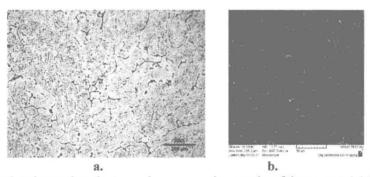


Fig. 4. Optical and scanning electron microscopy micrographs of the as-cast AA6063 aluminum alloy: a. optical microscopy; b. scanning electron microscopy.

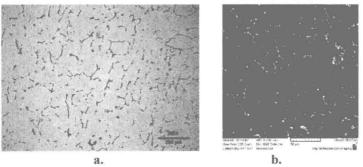


Fig. 5. Optical and scanning electron microscopy micrographs of the ECAP-ed material with one pass: a. optical microscopy; b. scanning electron microscopy.

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