

## Effects of ECAP on the mechanical properties of Mg-Al<sub>2</sub>O<sub>3</sub> nanocomposites

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### ABSTRACT

**Purpose:** The purpose of this paper is the study of the effect of equal channel angular pressing (ECAP) on the mechanical properties of the Mg-Al<sub>2</sub>O<sub>3</sub> nanocomposites. Magnesium and its alloys have excellent physical and mechanical properties for a number of applications. In particular its high strength: weight ratio makes it an ideal metal for automotive and aerospace applications, where weight reduction is of significant concern.

**Design/methodology/approach:** Severe plastic deformation is a useful methodology to refine the grain size to the submicron or even nanometer size

**Findings:** In the present work the influence of number of passes of ECAP by grain size, evolution of microstructure, mechanical properties and fracture of magnesium composites with different volume fraction of Al<sub>2</sub>O<sub>3</sub> particles has been investigated by means of optical microscopy, tensile tests and scanning electron microscopy.

**Research limitations/implications:** It has been found, that the grain size decreases with increasing number of passes. The mechanical properties of magnesium alloys are significantly influenced by the testing temperature leading to a decrease in the strength, by reinforcement and/or grain reinforcement leading to an increase in the strength.

**Originality/value:** From previous studies, it was found that the MMCs using different size particles and different ECAP passes can improved the mechanical properties. But the research of Mg MMCs reinforcement with different wt.% nanoscale Al<sub>2</sub>O<sub>3</sub> particles is not adequate.

**Keywords:** Material properties; ECAP; Microstructure; Tensile testing; UFG composites

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### PROPERTIES

## 1. Introduction

Magnesium alloys are attractive materials for such structural applications, because of its high strength to the weight ratio, which is better than of aluminium and many other metals and alloys, low density, excellent damping capacity, good recycling capacity and machinability [1]. For many applications, particularly in the automotive industry, a demand for greater strength and also wears resistance has to be met. Despite these advantages, the formability of wrought magnesium alloys at room temperature is rather poor due to their hexagonal closed packed (hcp) structure with limited slip systems [2]. The mechanical properties of magnesium alloys are significantly influenced by the testing temperature leading to a decrease in the strength by reinforcement and /or grain reinforcement leading to an increase in the strength. [3,4].

There are three primary approaches depending on the reinforcement material types and sizes to improve the mechanical properties of Mg alloys/composites including modulus, strength, and/or elongation: (a) addition of micron-size ceramic particulates [5], (b) addition of various oxide/carbide nanoparticles (NPs) or carbon nanotubes (CNTs) [6,7], and (c) addition of metallic particles such as Ti [8]. In recent years ceramic particle-reinforced metal matrix composites (MMCs) have gained wide acceptance because of their attractive properties [9].

Further improvement of mechanical properties of Mg alloys is possible using severe plastic deformation (SPD) methods. SPD is a useful processing tool to refine the grain size to the submicron or even nanometer size [10].

Although several severe plastic deformation techniques are available, equal channel angular pressing is an attractive process because it has potential to produce large samples [11]. Most investigations on ECAP have concentrated on pure metals and metallic alloys. Some limited reports are available on application of ECAP to metal matrix composites [12,13].

The present study is aiming to fabricate the fine-grained AZ61 alloys reinforcements with  $\text{Al}_2\text{O}_3$  nanoparticles and investigate the effect of ECAP on the microstructure and mechanical properties of the as-cast AZ61 alloys.

## 2. Experimental materials and methods

### 2.1. Materials preparation

The matrix used in this work is magnesium alloy AZ61 with ~6.0% aluminium and ~1.0% Zn. Its chemical composition is shown in Table 1.

Table 1.

Chemical composition of AZ61

Elements	Al	Mn	Zn	Si	Fe	Cu	Ni
Wt%	5.83	0.549	0.794	0.013	0.005	0.01	0.008
Mg.....Balance							

$\text{Al}_2\text{O}_3$  particles with weight fraction of 1, 2, and 5% within MMCs are used as the reinforcement phase. The commercially-available  $\text{Al}_2\text{O}_3$  powder with a particle diameter about 50 nm, purity of ~ 99.8%, is added into AZ61 to form Mg-based metal-matrix composites.

The melt-stirring technique is used to fabricate the present Mg MMCs. The AZ61 is initially placed inside a graphite crucible and heated to 760°C in a resistance-heated furnace. The molten alloy is stirred with a vane operated at 450 rev/min for 10 minutes. Preheated  $\text{Al}_2\text{O}_3$  particles are simultaneously added to the stirred alloy. Then the composite melt is finally poured into a metallic mold. The AZ61 MMCs containing  $\text{Al}_2\text{O}_3$  with different weight fraction of 1, 2, and 5 wt% are prepared for further mechanical testing. Subsequently the AZ61 MMCs were homogenized at 400°C for 10 h and water quenched.

### 2.2. Equal channel angular pressing (ECAP)

Three different composite materials AZ61+1, 2 and 5 vol.% of  $\text{Al}_2\text{O}_3$  were deformed by means of equal channel angular pressing (ECAP). ECAP tool with channel intersection angle  $\Phi = 90^\circ$  and outer radius angle  $\Psi = 0^\circ$ , with straining of 1.15 introduced during one pass, was used (Fig. 1).

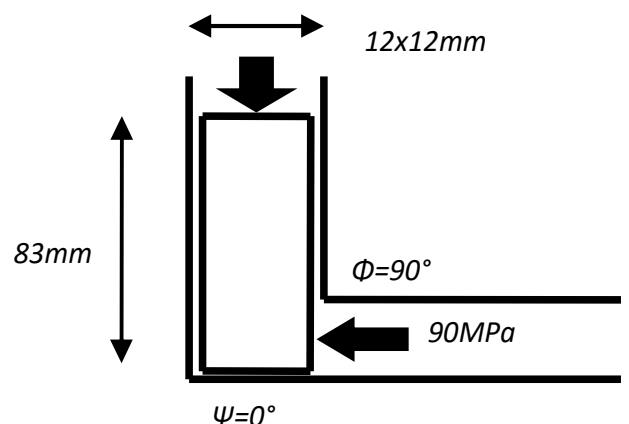


Fig. 1. Schematic drawings of ECAP process

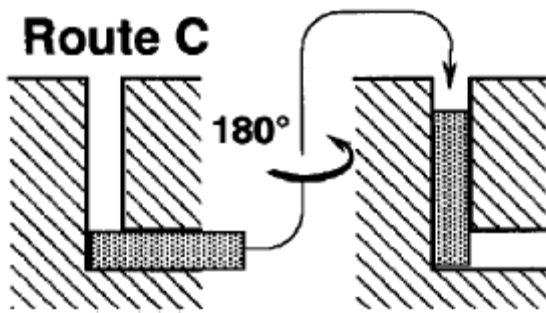


Fig. 2 Schematic illustration of via Route C

The cross section of ECAP channels was 12 x 12mm<sup>2</sup>. In order to homogenize the material flow (i.e. strain, stress, strain rate) within the outlet ECAP channel, defined backpressures of 90 MPa was applied by means of plunger of hydraulic valve. Composite material lubricated with graphite water solution was filled into the preheated die and heated up to pressing temperatures of 270°C for 7 min before consolidation. The ram speed during pressing was set up to 0.7 mm.s<sup>-1</sup>. The overall time of a temperature exposure of composite material during one ECAP pass was ~10 min. After a withdrawal of pressed composite material from ECAP die, material was immersed into the cold water. Either two or four ECAP passes via Route C were applied (Fig. 2).

### 2.3. Materials testing

The samples for the microstructure, mechanical properties investigations and fracture characterization were gradually removed from samples after all passes.

In order to evaluate homogeneity of microstructure and to determine the average size of matrix grains, pores and inclusions, metallographic and EDX analyses were performed. Tensile tests were carried out at room temperature, 100°C and 200°C. Hardness was measured using the TUKON™ 1102, micro hardness indentation (HV0.3) using a Vickers indenter.

The fracture analyses of the fracture surfaces after the tensile tests were carried out using scanning electron microscopy, JSM – JEOL 7000F.

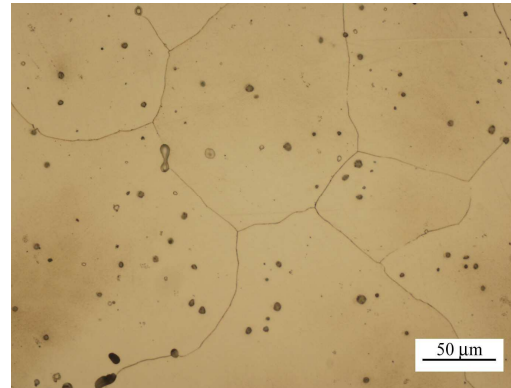
## 3. Results and discussion

### 3.1. Microstructure

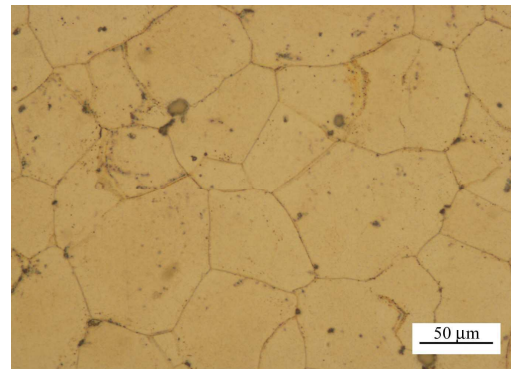
The typical optical microstructures of the investigated material AZ61 MMCs with different weight percentage of Al<sub>2</sub>O<sub>3</sub> before ECAP are shown in Figs. 3a-c.

Microstructure parameters of the experimental materials, in particular the matrix grain size, average size of the dispersed particles and their distribution were evaluated. The grains of the as-cast material were heterogeneous and had grain size from 50 to 200 μm. There is a slight tendency to smaller grains in materials with a higher volume fraction of secondary particles. The grain size of Al<sub>2</sub>O<sub>3</sub> particles was 50 nm [14].

a)



b)



c)

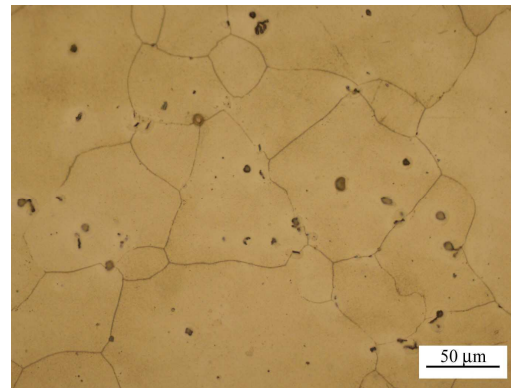


Fig. 3. Microstructure of as-cast materials: a) AZ61+1wt%Al<sub>2</sub>O<sub>3</sub>, b) AZ61+2wt%Al<sub>2</sub>O<sub>3</sub>, c) AZ61+5wt%Al<sub>2</sub>O<sub>3</sub>



After ECAP important microstructure changes were observed. The grains size after 2 passes of ECAP treatment was from 10 to 25  $\mu\text{m}$  and the grains size after 4 passes of ECAP treatment was from 5 to 8  $\mu\text{m}$ . This tendency was reported previously in [14], that the heat deformation process of such materials, besides the formation of incorporated  $\text{Al}_2\text{O}_3$  particles, also leads to the creation of intermetallic compound  $\text{Mg}_{17}\text{Al}_{12}$  [15]. These particles are distributed previously on the grain boundaries and avoid the grains growth [16]. Deformation takes place through slip systems which mutually interact by forming

microcracks. The average grain sizes of MMCs decreased evidently with the increase of the weight percentage of  $\text{Al}_2\text{O}_3$  particles additions and ECAP passes.

The typical optical microstructures of the materials after 2 and 4 passes of ECAP are shown in Figs. 4a-c.

The grain size of the material with 2 wt%  $\text{Al}_2\text{O}_3$  after 2 and 4 ECAP passess, was the most heterogeneous in comparison with other studying materials, Fig. 4b. Heterogeneity of microstructure was caused by the preparation of materials and compaction processes.

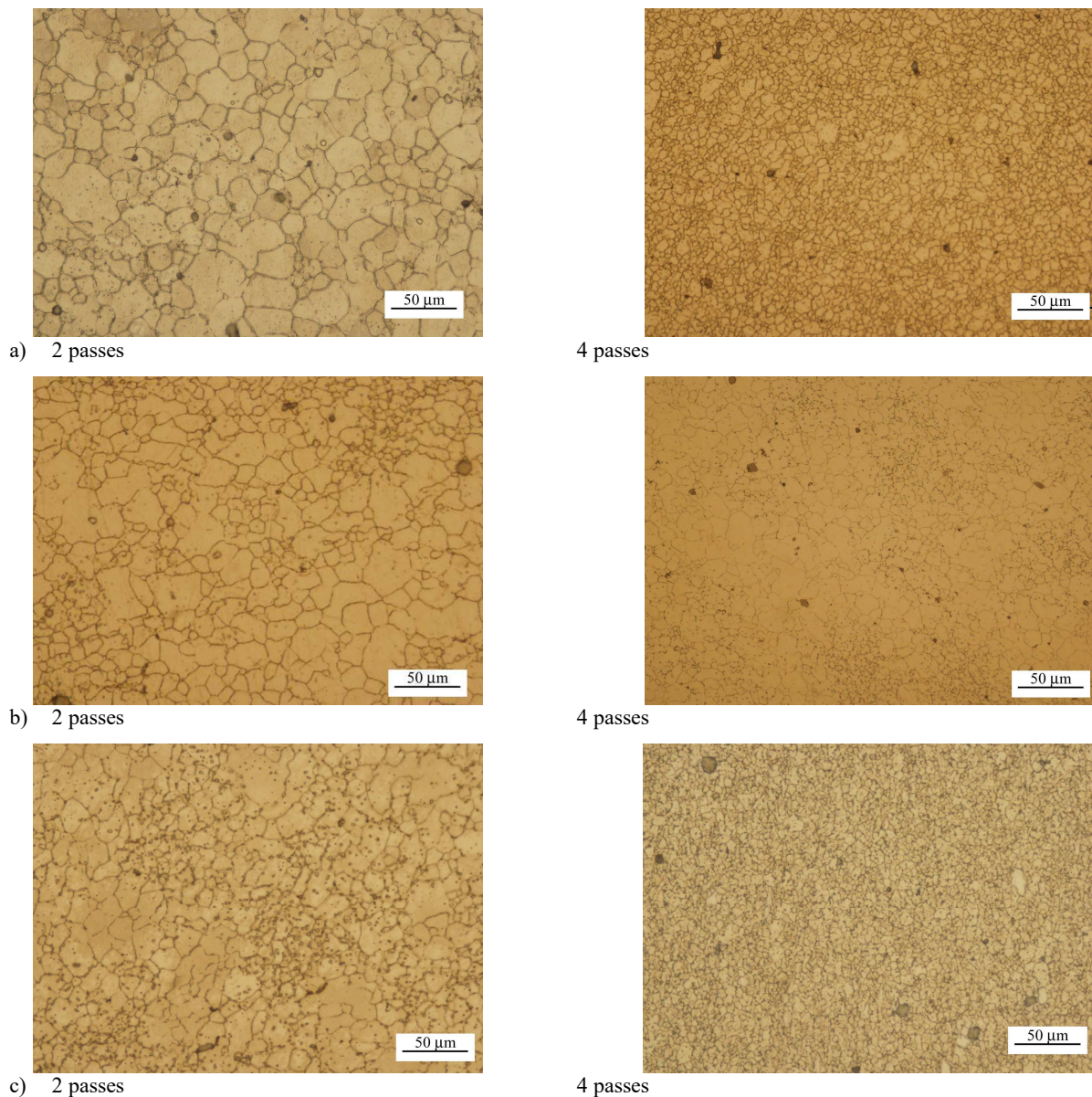


Fig. 4. Microstructure of the materials after 2 and 4 passes of ECAP (a) AZ61+1wt%  $\text{Al}_2\text{O}_3$ , (b) AZ61+2wt%  $\text{Al}_2\text{O}_3$  (c) AZ61+5wt%  $\text{Al}_2\text{O}_3$

A more uniform microstructure consisting of grains with an average grain size of 5  $\mu\text{m}$  is observed in the AZ61-5 wt%  $\text{Al}_2\text{O}_3$  particles MMC after 4 passes (Fig. 4c). In the case of ECAP-ed AZ61 MMCs, grain growth restriction might have been preferred by the presence of nano- $\text{Al}_2\text{O}_3$  particles. The grain refinement is caused by capability of nano- $\text{Al}_2\text{O}_3$  particles nucleating MMC grains during recrystallization, and the nano- $\text{Al}_2\text{O}_3$  particles also hinder the movement of the matrix and retard the crystalline grains growth when the melt is solidified to result the grain refinement.

### 3.2. Mechanical properties

The effect of the different manufacturing processes on the yield strength at elevated temperatures is shown in the Figs. 5a-c and on the tensile strength and elongation in the Figs. 6a-c. The yield strength and tensile strength at room temperature increased with the ECAP passes number, and also with the volume fraction of  $\text{Al}_2\text{O}_3$  particles, Fig. 5 and Fig. 6a, respectively. The tensile strength of the as-cast material was from 240 to 252 MPa at room temperature depending of the volume fraction of the secondary phase and increased to 260 – 270 MPa after 2 ECAP passes and to 300-350 MPa after 4 ECAP passes.

The yield strength and the ultimate tensile strength at 100°C show a similar trend as at room temperature, Figs. 5b and 6b.

The yield strength and ultimate tensile strength decreased at 100°C approximately by half compared to the strength characteristics at room temperature. The ultimate tensile strength of as-cast material was from 117 to 141 MPa depending on the volume fraction of the secondary phase and increased to 208 – 220 MPa after 2 and 4 passes. Significant changes of mechanical properties are occurred at 200°C when the the yield stress and the ultimate tensile strength decreased in ECAPed materials in contrast to the materials without ECAP, Fig. 5c and Fig. 6c, respectively.

Plastic properties and their changes depending on the volume fraction of secondary  $\text{Al}_2\text{O}_3$  particles and ECAP passes number are shown in Figs. 6a,b,c.

Elongation decreases at all test conditions (room temperature, 100 and 200°C) with increasing fraction of reinforced  $\text{Al}_2\text{O}_3$  particles in composites. Further, the dissolving lamellar  $\beta$  phase ( $\text{Mg}_{17}\text{Al}_{12}$ ) affects the tensile strength. With the amount of  $\beta$  phase ( $\text{Mg}_{17}\text{Al}_{12}$ ) hardness increases but ductility decreases.

Simultaneously elongation decreases with higher number of ECAP passes at RT and 200°C. This tendency was not confirmed at mechanical testing at temperature of 100°C.

The material after 2 ECAP passes had a highest elongation, what can be related with higher heterogeneity of the grain size that was confirmed by the microstructure analysis.

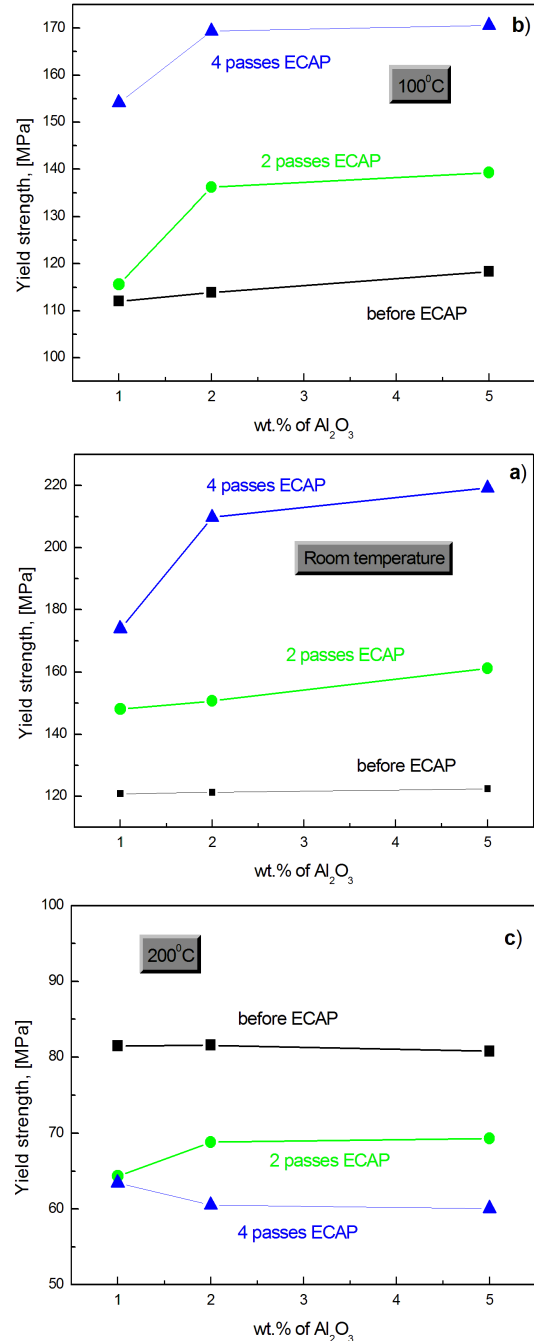


Fig. 5. The effect of different manufacturing processes on the yield strength at a) room temperature, b) 100°C, c) 200°C

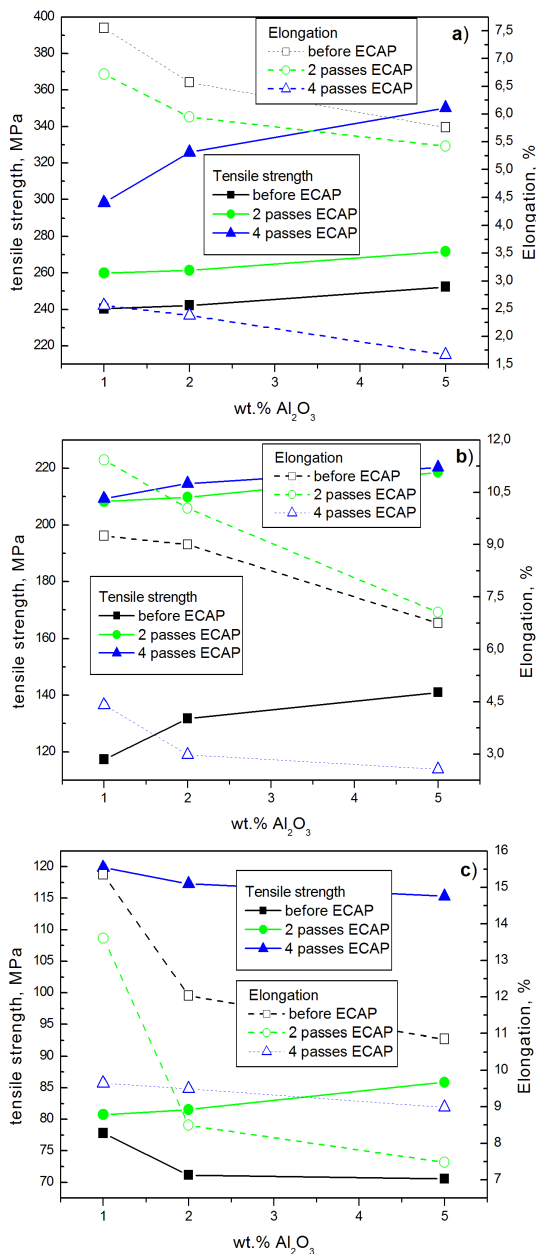


Fig. 6. The effect of different manufacturing processes on the tensile strength and the elongation at a) room temperature, b) 100°C, c) 200°C

Heterogeneity of microstructure caused by the preparation of materials and compacting processes led to some abnormal results relating mechanical properties (plastic properties respectively strength properties at 200°C).

Measured values of mechanical properties are in accordance with the work [17].

### 3.3. Fracture analysis

Figure 7 illustrates the fracture surfaces of the tensile tested specimens of AZ61 alloy with 1 wt.% Al<sub>2</sub>O<sub>3</sub> particles as-cast and after 2 and 4 passes of ECAP.

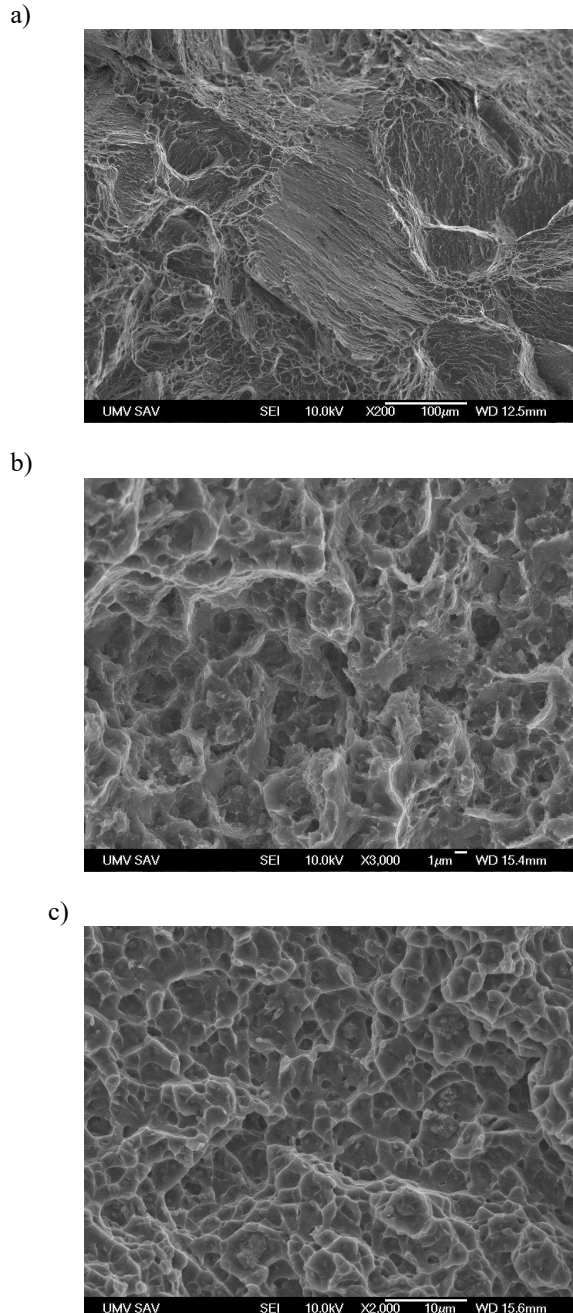


Fig. 7. Fracture surfaces of AZ61+1wt%Al<sub>2</sub>O<sub>3</sub> at 100°C a) as-cast material, b) after 2 ECAP passes, c) after 4 ECAP passes



As Fig. 7a shows, some obvious river patterns were found in the microstructure of the as-cast material. It was confirmed that the fracture of the as-cast AZ61 alloy was characterized by brittle cleavage fracture. It was not beneficial to improve the mechanical properties of the as-cast AZ61 alloys. The fracture of ECAP-ed AZ61 composites (Figs. 7b,c) belonged to the ductile fracture, which had a good effect on improving the mechanical properties in comparison with the as-cast material. The number of dimples increased with the increasing testing temperature. The fracture is formed mainly by  $Mg_{17}Al_{12}$  particles. The mean dimple size of the ductile fracture after four ECAP passes was 2.78  $\mu m$ . The calculated size of  $Mg_{17}Al_{12}$  particles was ca 0.9  $\mu m$ , [15].

#### 4. Conclusions

In this work the influence of equal channel angular pressing (ECAP) on the microstructure and mechanical properties of AZ61 with the different volume fraction of  $Al_2O_3$  particles has been investigated. The reduction of the grain size after ECAP proves that the current ECAP process is effective in the microstructure refining of Mg MMC. The grains in the ECAP-ed AZ61 with the different volume fraction of  $Al_2O_3$  particles are equiaxed and homogeneously distributed like those revealed in the as-extruded alloy, suggesting that recrystallization took place during the ECAP processing.

The effect of the different manufacturing processes on the yield and ultimate tensile strength and elongation at elevated temperatures was confirmed. The tensile strength at room temperature and 100°C increased with the number of ECAP passes as well as with the volume fraction of  $Al_2O_3$  particles. Significant changes of strength properties occurred at 200°C when the ultimate tensile strength and the yield stress decreased in the ECAPed materials in contrast to the materials without ECAP.

The fracture of ECAP-ed AZ61 alloys were characterized as the ductile fracture due to the existence of a large number of dimples, which had a good effect on improving the mechanical properties.

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#### Additional information

Selected issues related to this paper are planned to be presented at the 22<sup>nd</sup> Winter International Scientific Conference on Achievements in Mechanical and Materials Engineering Winter-AMME'2015 in the framework of the Bidisciplinary Occasional Scientific Session BOSS'2015 celebrating the 10<sup>th</sup> anniversary of the foundation of the Association of Computational Materials Science and Surface Engineering and the World Academy of Materials and Manufacturing Engineering and of the foundation of the Worldwide Journal of Achievements in Materials and Manufacturing Engineering.

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