

# Strain rate effect on deformation of pure Al in equal channel angular pressing

## تأثير معدل الانفعال على تشكل الالومنيوم النقي في الكبس ذات القنوات المتساوية (الكبس الزاوي)

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### الخلاصة

تم دراسة تأثير معدل التشوه الشديد في المدى اللدن (SPD) من خلال الكبس الزاوي ذات القنوات المتساوية (ECAP) على البنية البلورية والتحسين في مقاومة المادة. تم تنفيذ الكبس الزاوي في درجة حرارة الغرفة لعينة بمساحة مقطع مربعة وذلك للحصول على حجم اصغر للحبيبات. وقد تم فحص البنية البلورية باستخدام المجهر الإلكتروني الماسح لتقييم البنية البلورية للمواد التي تم عليها الكبس الزاوي. تم ايضا دراسة الخواص الميكانيكية للمواد المضغوطة بواسطة الكبس الزاوي. الاختبارات التي اجريت على العينات المضغوطة تظهر أن الزيادة في معدل الانفعال على الالومنيوم النقي يؤدي إلى انخفاض في معدل زيادة الصلادة. أيضا، عند استخدام معدل انفعال اقل فانه يؤدي الى الحصول على بنية مجهرية متجانسة من النوع (حبيبية متساوية في جميع الاتجاهات) equiaxed grain افضل من معدل الانفعال العالي كما نلاحظ زيادة في مقاومة الخضوع تصل الى ثلاث اضعاف بعد الكبس الزاوي.

### Abstract

The role of the rate of severe plastic deformation (SPD) during equal-channel angular pressing (ECAP) on microstructure and strength are studied. ECAP was performed at room temperature to refine grain size of pure Al of square cross-section. The examination of the microstructure has been undertaken using a scanning electron microscope. The mechanical properties of the ECAP processed material are investigated. The tests show that the hardness increase is affected by the amount of the strain rate on pure Al. Also, it was observed that a lower strain rate produces a homogeneous microstructure with equiaxed grains more quickly than with higher strain rate and the yield strength increases up to three times.

### Keywords:

Equal channel angular pressing, ECAP, Pure Aluminum, Hardness, Compression rate

## 1. Introduction

Ultrafine Grained (UFG) metals (Nanocrystalline metals or nonmetals) have grain size ranges between 0.1–1  $\mu\text{m}$  compared to tens or hundreds  $\mu\text{m}$  of common metals. This structural change affects many mechanical properties of UFG metals, have 3-5 times higher strength.

Nanocrystalline and UFG materials processed by severe plastic deformation

(SPD) methods have been the subject of intense studies in the last decades [1]. Among all SPD techniques, equal channel angular pressing (ECAP) has the advantages of producing large samples [2] and has the potential for commercialization [3]. This process has proposed by Segal et al. [4] to achieve ductility and later it has been developed by Valiev et al. [5, 6]. In this method, as illustrated in Fig. 1, a billet is pressed through a die having two intersecting channel angle of  $\phi$  and an

outer arc of curvature  $\psi$  during this process an equivalent strain of approximately 1 is achieved [1]. A major advantage achieved in equal-channel angular extrusion (ECAE) is that the material cross section is unchanged during extrusion, which means that the material can be deformed repeatedly to attain a high total strain [7]. Also, those materials can be used as super high strength materials, intelligent metal materials and super plastic materials. Two superior features of UFG materials produced by ECAP are very high strength and the potential of super plasticity at lower temperatures and higher strain rates [8, 9]. In applications, the nonmaterial is promising for developing the systems of microelectronics, informatics, and micro electro-mechanics [1, 8]. Recently, the small overall internal combustion engine and the high strength threaded articles have been successfully used in practical application. Another application close to implementation is medical implants made of UFG CP titanium [10]. Other applications will certainly follow, taking advantage of high strength and weight savings so valued in the aerospace and automotive industries [11, 12].

Plastic deformation of the material is caused by simple shear in a thin layer at the crossing plane of the channel passages. This shear, together with rotation, converts the material rectangular vertical volume (a b c d), plotted in Fig. 1, towards the parallelogram horizontal volume (a<sub>1</sub> b<sub>1</sub> c<sub>1</sub> d<sub>1</sub>).

The equivalent plastic strain  $\varepsilon_N$ , in this case is about 1.15 [4]. It can be calculated by the equation:

$$\varepsilon_v = \frac{N}{\sqrt{3}} \left[ 2 \cot \left( \frac{\Phi}{2} + \frac{\Psi}{2} \right) + \Psi \operatorname{cosec} \left( \frac{\Phi}{2} + \frac{\Psi}{2} \right) \right] \quad (1)$$

**Table I Chemical composition of the materials used in this work**

Component	Fe	Si	Mn	Mg	Al
by wt %	0.062	0.041	0.001	0.001	balance

Where,  $N$  is the number of passes. Since the strain required for changing the

microstructure is usually 4–8, the billet has to be pressed repeatedly through the die. This also gives the opportunity for billet rotation about its axis between each pass of ECAP (Fig. 2). The three fundamental options for this rotation are called A (no rotation), C (180° rotation) and B<sub>C</sub> (90° rotation in the same direction).

It has been reported that four processing route can be used for ECAP [13]. Some researchers have concluded that route B<sub>C</sub> (in which the billet is rotated 90° clockwise or anti-clockwise) is the most effective route for producing UFG material [14], whilst others have suggested that route A (without any rotation) is more effective [15]. The absence of the strain hardening is another important characteristic of the deformation behavior of these materials [16]. Also there is some evidence of decreasing hardness/strength during ECAP as the number of passes increases [9, 17, 18]. Till now there have been little attempts to analyze this event.

In the present work the role of strain rate on the evolution of microstructure and mechanical properties of ECAP material – Al in our study - is investigated.

## 2. Experimental procedure

### A. ECAP Experiments

The experiments were conducted using purity aluminum of composition given in Table I. Aluminum was chosen because its symmetric crystal structure (FCC) leading to geometrically similar dislocation densities, and because aluminum and its alloys are widely used in engineering applications.

The pure Al was provided by Egypt Aluminum Company in Naga Hammady, in the form of extruded rods and was cut into billets of length of 37 mm and 12 mm x 12 mm cross-sections to be processed by ECAP.

The ECAP process carried out at room temperature with different ram speeds of 1, 3 and 10 mm.s<sup>-1</sup> (corresponding to initial strain rates of 0.03, 0.09 and 0.3 s<sup>-1</sup>, respectively) and a

split die having an internal channel with an angle of  $\phi = 90^\circ$  and an outer angle of curvature of  $\psi = 20^\circ$  where the two parts of the channel intersect.

All extrusions (ECAP) were conducted using an Instron 8505 machine of 120 ton and 100 ton static and dynamic loading capacities, respectively (Fig. 3). The die and sub-press equipment (punch and clamps) materials were made of tool steel. Molybdenum disulfide ( $\text{MoS}_2$ ) and Teflon were used as lubricants.

### ***B. Mechanical Testing***

The exit billets were sectioned perpendicular to their longitudinal axes by wire cutting machine and then mounted and finishing as shown in Fig. 4. The Vickers hardness ( $\text{kg/mm}^2$ ) was measured using an Instron universal hardness testing machine at load of 10 kg. A series of individual measurements was recorded on each polished section whereby the Vickers indenter was moved over the surface and measurements of the Vickers hardness,  $H_v$ , were recorded in a regular grid pattern with spacing between each separate measurement of 3 mm.

A tensile testing machine with 500 N load cell was used for the tension test of the processed sample. The tensile characteristics were determined by the standard tensile tests on specimens that were cut from the work piece (Fig. 5) with their axis parallel to the pressing direction after one pass of ECAP. The gauge length of the tensile specimens was 6 mm and the cross section was  $1.23 \text{ mm} \times 1.73 \text{ mm}$  (depth  $\times$  width), as shown in Fig. 6.

### ***C. Microstructural Characterization***

The average grain size of the material deformed by ECAP is measured using scanning electron microscope. Samples with thickness of about 0.5 mm have been cut at the centers of the pressed samples subjected to one pass, and perpendicular to the pressing (extrusion) direction. For SEM images the surface of

the sample was polished by electro polishing in a 100 ml  $\text{HClO}_4$  + 900 ml  $\text{CH}_3\text{OH}$  solution at  $-20^\circ \text{C}$  and a voltage of 50 V.

## **3. Results and Discussion**

### ***A. Grain Refinement***

Fig. 7 shows the microstructure after ECAP by electron microscope for pure Al at different press rates. Microstructure and grain size are changed during ECAP as a result of large plastic deformation that takes place in a narrow region (shear zone) at the intersection of the two channels. The microstructure obtained is quite complex and depends on the extrusion parameters (ram speed, die angle, total strain, temperature, etc.).

As expected a refined grain structure is obtained for all ram speeds. However, the measurements show an increase in the refined grain diameter with the increase in compression speed. According to the measurements, the mean grain size of the pure aluminum in its original state was 390 nm and the mean grain size of the material after ECAP is decreased to 235, 240 and 310 nm with press rates of 1, 3 and 10  $\text{mm.s}^{-1}$ , respectively. So, the change is about 39.7%, 38.5%, and 20.5% and which is a remarkable degree of grain refinement compared to the un-annealed condition of the as-received sample. In order to understand this interesting observation we suggest the following mechanism illustrated in Fig. 8. The mechanism differentiates between the motions of dislocations in both rapid- and slow-pressing. During the slow pressing the dislocation piles-up at the grain boundaries and at junctions as the energy level required overcoming the barrier of the grain boundary is insufficient. These pile-ups gather and can be the source of a new grain boundary resulting in a total high level of grain refinement.

In the case of speed pressing the dislocations possess higher energy level and thus have better chances to cross the grain boundary. The resulting grain has a

higher density of dislocations, but the grain refinement is not as fine as with small rate ECAP.

### **B. Hardness**

Generally, the hardness increases with ECAP after one pass as mentioned in [20, 21]. Hardness distributions on the cross-sectional plane for pure Al prior and after of a single pass of ECAP are plotted in Fig. 9. The x-axis in the figure represents the locations of individual hardness point on a cross-sectional plane perpendicular to its extruded axis with respect to the midpoint of the section. It can be observed that the variation of the hardness values along the cross-section of the sample after ECAP (one pass) is minimal. It seems that these values are not affected by the pressing rate. This indicates a more homogeneous microstructure of the sample through the cross-section after ECAP.

It can be seen that the sample exhibits a homogeneous hardness distribution throughout the whole cross-sectional plane before ECAP with an average hardness value of  $\sim 19$ . After a single pass of ECAP at compression rate  $1 \text{ mm.s}^{-1}$  (initial strain rate  $0.03 \text{ s}^{-1}$ ), a much greater value of hardness,  $\sim 39$  was achieved.

Fig. 9 shows the variation of Vickers hardness on pure Al at different rates (compression speed: 1, 3, and  $10 \text{ mm.s}^{-1}$  or initial strain rates: 0.03, 0.09, and  $0.3 \text{ s}^{-1}$ ), where the hardness increase approximately by 51, 48.9, and 39.7%, respectively. The Vickers hardness increases more with the decrease in press rate on pure Al materials. This result is in good agreement with our grain diameter measurements and our interpretation. In slow pressing the high amount of grain boundaries serves as a barrier to the penetration of the hardness indenter resulting in higher measuring values of hardness, Fig. 10(a). The high dislocation density helps through the easier slip action the indenter to penetrate inside the surface and thus producing lower

hardness value than in the slow pressing case, Fig. 10(b).

### **C. Strength**

The engineering stress-strain curves of pure Al before and after ECAP with press rate of  $0.03 \text{ mm.s}^{-1}$  are represented in Fig. 11. The yield strength of pure Al before ECAP (as-received material) is 35 MPa. The yield strength increased to 93 MPa after one pass of the extruded billet (the strength increases by approximately 3 times after ECAP) due to the UFG of pure Al. This increase in the yield strength is expected as the grain size was refined from 390 nm as-received to about 235 nm through the shearing of the sample. This refinement of grains acts as a strengthening mechanism as the free path of dislocations become shorter and more load is needed to overcome the resistance of dislocation motion through the grain boundaries.

## **4. Conclusion**

Equal Channel Angular Pressing is capable of producing bulk samples of ultrafine-grained materials and thus achieving advanced properties. The method is very attractive because of its potential for scaling-up in industrial applications. Investigations were carried out in this work to show the effect of the rate of ECAP process on pure aluminum. The following points can be concluded:

- 1) The increase in press rate on pure Al results in less hardness increase compared to slow press rate.
- 2) The increase in grain diameter is directly related to the increase in the press rate.
- 3) The change of mean grain size of the material after ECAP is about 39.7%, 38.5%, and 20.5% at press rate (1, 3, and  $10 \text{ mm.s}^{-1}$ ) and this means the degree of grain refinement is remarkable.
- 4) The yield strength increases by about 3 times after ECAP of the extruded billet with press rate of  $0.03 \text{ mm.s}^{-1}$ .

## 5. References

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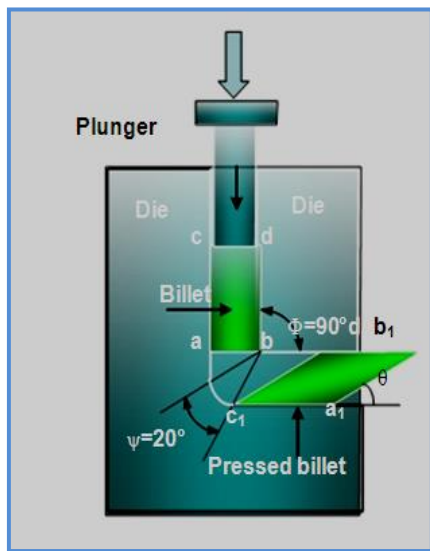


Fig. 1 Equal channel angular pressing (ECAP)

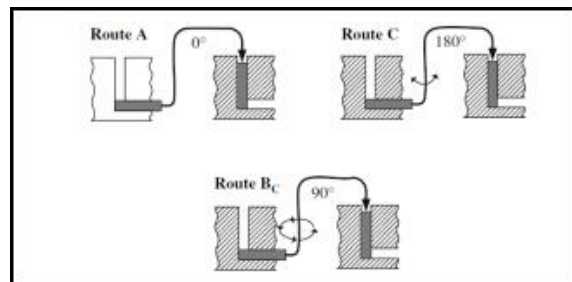


Fig. 2 Three basic options for billet rotation between consecutive passes through ECAP die

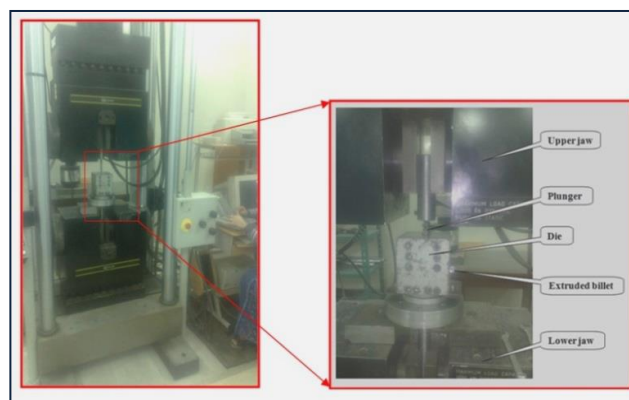


Fig 3 The die used in study mounted on instron machine

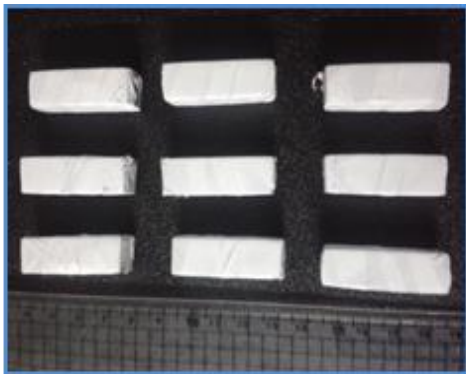


Fig. 4 samples before ECAP where they are Warped by Teflon



Fig. 5 The sample after ECAP

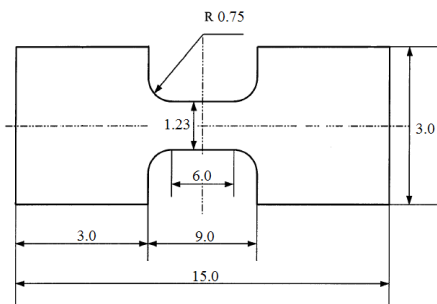


Fig. 6 The drawing of tension sample

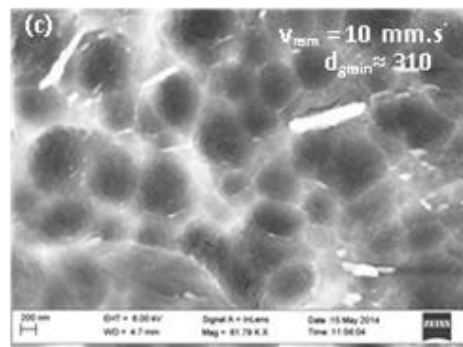
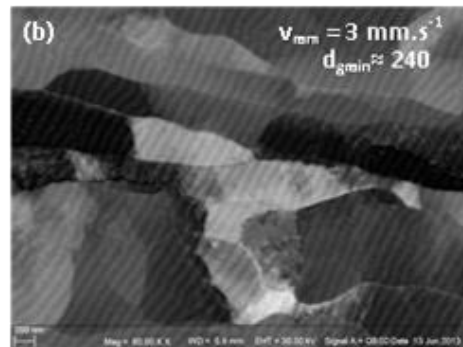
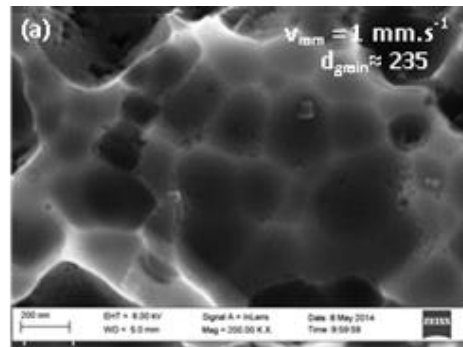
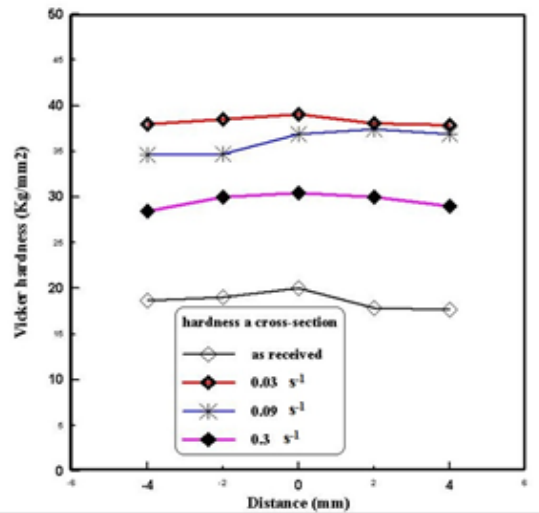


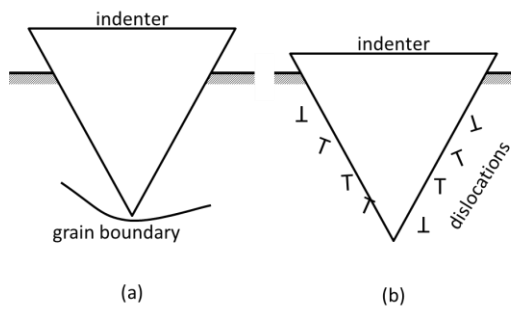
Fig. 7 The microstructure after ECAP for pure Al with electron backscattering diffraction (EBSD): a) compression rate  $1 \text{ mm.s}^{-1}$ , b) compression rate  $3 \text{ mm.s}^{-1}$  and c) compression rate  $10 \text{ mm.s}^{-1}$



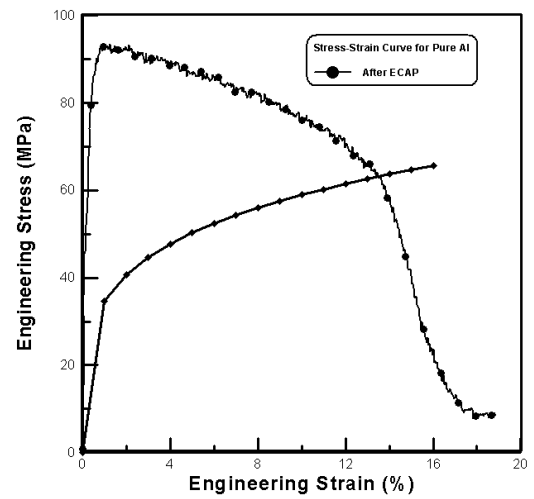
**Fig. 8** Role of dislocation motion on the final grain size during different ram speeds



**Fig. 9** Variation of Vickers across the cross-section for three different strains



**Fig. 10** Hardness indenter penetration in materials produced by (a) slow ECAP (b) fast ECAP



**Fig. 11** The stress – strain curve of pure Al after ACAP