ABSTRACT. Equal channel angular pressing (extrusion) is one of the techniques applied to produce ultra-fine grained nanocrystalline structured metals and alloys. This fine-grained nanocrystalline structure is obtained by imposing severe plastic strain to the bulk material. In this study sample probes of pure aluminum were collected from different stages of the extrusion process and prepared for being examined via nanoindentation. A series of nanoindentation tests was conducted for assessing the change of the mechanical characteristics of the pure aluminum due to the undergone plastic deformation. As a result of the nanoindentation tests indentation hardness and indentation modulus were obtained. The experimental results show that the mechanical properties depend strongly on the microstructure evolution during the equal channel angular extrusion. This investigation was made by means of nanoindentation experiments using Nanoindenter G200 (Agilent Technologies). It was suggested a connection tendency between increasing to a certain level of the hardness and the introduced by ECAE severe plastic deformation strain.

KEY WORDS: Equal channel angular extrusion, nanoindentation, indentation modulus, indentation hardness.

1. Introduction

Equal channel angular extrusion (ECAE) is well accepted method to produce bulk ultrafine-grained (UFG) materials from regular coarse-grained materials. The ECAE-processed materials typically show superior mechanical properties as higher strength, wear resistance, ductility, and high strain-rate super-plasticity [1]. This method consists in pressing a rod sample through a die with no change in the cross-sectional area and has the advantage to lead to significant strengthening of the material at ambient temperatures [2] allowing producing relatively large pieces that are free of contamination and pores.

ECAE is based on the implementation of simple shearing concentrated between crossing planes of equal in cross section channels. An enhancement of the method is passing the work-piece through two parallel channels. This procedure is called equal channel two-angular extrusion (ECTAE). The work piece deformed by parallel channels is subject two times to simple shearing in opposite directions within two zones and without taking the sample out and without rotating it.
The ECAE process in a channel with a cross-section of 10.6x10.6 mm and a length of sample rod over 55 mm was simulated in [3]. It was found that the in this case the optimal filling up of the deformation space is achieved for the angle of crossing between the channels $\Phi = 135^\circ$, and when the distance between enter/exit parallel channels (K) is equal to 7 mm. These parameters are established by means of computer simulation with CAD/CAE software Quantor Form 2D/3D. In the present paper the evolution of the material properties due to ECTAE under these optimal parameters is addressed by means of an advance technique for local material characterization, namely displacement sensing indentation also known as nanoindentation or instrumented indentation testing (IIT).

2. Local material properties characterization

Nanoindentation is well accepted method for characterisation of the material properties at a local scale. This method was already applied to ECAE processed metallic materials in order to investigate the development of the local properties and their homogeneity, e.g. [4], [5]. In our study, we employed nanoindentation to investigate the evolution of the local material properties with pressing passes and to compare these properties when indentation is performed in a grid without excluding grain boundaries and when the indentation tests are done on etched samples with the care to be apart of the grain boundaries.

3. Sample preparation and experimental procedure

It has been experimentally shown that in order to achieve satisfactory ultrafine-grained microstructure the material has to be processed by ECAE up to multiple passes. Because the aim of microstructure refinement is to gain better material properties it is therefore interesting to investigate the evolution of the local material characteristics with the number of ECAE passes as well as depending on the main ECAE processing parameters as die angles and channel to channel distance.


The sample rods of 55 mm length were prepared of pure Al 99.9999. The initial grain size is 100-150 $\mu$m. The sample rods were deformed at ambient temperature by ECAP route with 4 passes and ECAP die with angle of intersection of two channels $\Phi=135^\circ$ and the outer angel $\Psi=15^\circ$. This setting allows an accumulation of the effective strain of 1.10 to 1.15 for each pass.

The scheme of the ECTAE setting is given in Fig. 1. Probes after the first pass were cut at three locations as it is schematically shown in Fig. 1. In our case there were 4 ECTAE passes with turning the rod in 180$^\circ$ in the second pass, and in 90$^\circ$ in the third and forth passes.

We prepared 5 samples for nanoindentation by making cuts at three different locations during the first ECTAE pass (samples S1, S2, S3) and one cut after the
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second pass (sample S4) and one cut after the forth pass (S5), as indicated in Fig. 1. The samples were fixed in epoxy resin for being suitable for testing. The five samples were first polished and after the first series of nanoindentation tests the same samples were exposed to etching in order to make the grain interfaces better visible.

The samples were polished and etched electrolytically according ASTM E 407 99 ASM Handbook “Metallography and Microstructures” methodology. Grinding is performed of sandpapers from 400 to 2000, preliminarily covered with paraffin in order to avoid formation of deep scratches. The specimens were polished by diamond paste 1 µm and ¼ µmq. The polishing was done by a water suspension of amorphous SiO2 and the final electropolishing was done by using electrolyte composed of 400 ml ethanol, 70 ml distilled H2O, 30 ml HClO4 and by means of “Polectrol” apparatus of “Struers”. The microstructure of technical grade pure aluminium is developed by the Keller solution – 95 ml H2O, 2.5 ml HNO3, 1.5 ml HCl, 1.0 ml HF.

Fig. 1. ECTAE procedure and the location of the sample extraction (first pass).

3.2. Experimental procedure
Nanoindentation tests were carried out using Agilent G200 Nanoindenter equipped with sharp Berkovich three-sided diamond pyramid with centerline-to-face angle of 65.3° and 20 nm radius at the tip. The specific Berkovich indenter geometry was calibrated against data on standard sample of fused silica. Two series of nanoindentation were realised, one before and the second after sample etching.

The procedure used to assess the indentation hardness \( H_{IT} \) and modulus \( E_{IT} \) consists in cyclic loading-unloading program and employs the method of Oliver and Pharr for determining \( H_{IT} \) and \( E_{IT} \) using the measured load-displacement curves [6]. The maximum applied load is 50 gf for all tests; the number of times to load is 10,
time to load is 15 s and the percent to unload at each load cycle is 90% of the maximum applied load. For each cycle i, the indenter penetrates the surface at a rate defined by (Maximum Load/Time to Load)*(2^i/2^Number of Times to Load). Loading for the cycle ends when the load on sample reaches Maximum Load*(2^i/2^Number of Times to Load). At the peak load for the cycle, the load on sample is held constant for a period equal to peak hold time of 20 s. The stiffness is calculated using the unloading part of the load displacement curve at 50% unload.

In order to have sufficient data for proper statistics each indentation test consists of 9 single indentations. For the test series after etching the selection of the individual indentation site was made not to be at the grain boundaries, while for the tests before etching indentations were grouped in 3x3 grid matrix with regular distance between the individual indentations of 150 µm.

4. Results and discussion

The nanoindentation tests were used to obtain the load displacement curves and to determine the local material characteristics, namely indentation modulus and indentation hardness at different indentation depths and under different loads.

4.1. Indentation hardness and indentation modulus

Indentation modulus \( E_{IT} \) and indentation hardness \( H_{IT} \) are determined employing Eq.(1) and Eq.(2),

\[
E_{IT} = \left(1 - v_i^2 \right) \left[ \frac{2}{S} \sqrt{\frac{A}{\pi}} - \frac{1 - v_i^2}{E_i} \right]^{-1}
\]

where the subscripts \( s \), and \( i \) correspond to sample and indenter, respectively. The unloading stiffness or the so called contact stiffness is defined as the slope \( S = dP/dh \) of the upper portion of the unloading curve during the initial stages of unloading.

Although the calculation of \( E_{IT} \) modulus requires the knowledge of the sample’s Poisson’s ratio, the dependence on \( v_i \) is weak. The analysis shows that with an uncertainty of 40% in Poisson’s ratio there is only 5% uncertainty in \( E_{IT} \) modulus. Indentation hardness, \( H_{IT} \), is defined as the ratio of the maximum load to the projected contact area \( A(h_c) \) at that load:

\[
H_{IT} = \frac{P_{\text{max}}}{A(h_c)}
\]
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The results for all the samples representing the average of 9 calculated $EIT$ and $HIT$ at each of the ten load cycles are given in Fig. 2. The indentation modulus changes moderately from sample to sample and more significantly with increasing the load at which it is determined. The value of $EIT$ at maximum indentation load of 500 mN is equal to 80 GPa. However, the indentation hardness increases
significantly after the first angle pass (sample S2). With respect to the indentation hardness the best performance has the sample S4 taken after the second pressing pass. The hardness of sample S4 is almost constant with the indentation depth and decreasing with increasing the load up to the load of 10 mN and after that slightly increases to the highest along all samples hardness at 500 mN. The hardness of the other samples shows distinguishable and continuous increase in value with decreasing the indentation depth. Next, it is well seen that sample S1 (as prepared before pressing) has significantly lower indentation hardness along the whole loading interval, which obeys the same trend of continuously increasing HIT with decreasing the indentation load.

Figure 3 gives the comparison of the indentation modulus and indentation hardness determined before and after sample etching. The hardness shows no significant difference depending on the way location of the indentation was chosen, while indentation modules for samples S1, S3 and S5 before and after etching are visibly different. It is difficult to make conclusion if this difference is due to probably going during the indention into the grain interfaces more often than into solely the grains.

![Fig.3. HIT a) and EIT b) in GPa at load 500 mN: before etching- black; after etching- grey nanoindentation tests were used to obtain the load displacement curves and to determine the local material characteristics, namely indentation modulus and indentation hardness at different indentation depths and under different loads](image)

**4.2. Imprint images**

The images after indentation are given in Fig. 4 for all etched samples and at as prepared condition (sample S1 before etching). While for sample S1 it is easy to distinguish the grain boundaries and grains are sufficiently larger than the indents, this is not the case for samples S4 and S5. The second image in Fig. 4 shows the imprint grid matrix of 9 indentations on sample S1 before etching (with label S1-1). The imprint grid matrix for the other samples has the same dimensions and

![imprint images](image)
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arrangement and it may be concluded that even for the as prepared sample S1 we may have done indentations across or at a grain boundary.

Fig. 4. Imprints after nanoindentation at maximum load of 500 mN.

5. Conclusions

In the present work the evolution of the elastic and plastic characteristics of pure Al samples manufactured by equal channel two angular extrusion process is assessed by means of displacement sensing indentation technique IIT. Two characteristics of the mechanical properties, indentation hardness and indentation modulus, of samples taken at different stages of the pressing process and at three different location along the processed rod (billet) were determined, compared and analysed. It may be concluded so far that the determination of the indentation modulus depends on whether indentation locations are chosen arbitrary or solely within the grains. Indentation hardness increases most significantly after the first angular pressing (S2) during the first pressing pass and reaches its maximum after the second ECTAE pass (S4). Finally, the results presented in this paper demonstrate the capability of nanoindentation technique to assess the local material properties and to give inside the microstructure evolution during equal channel angular extrusion and its influence on the material characteristics of the final product.

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