# MICRO-HARDNESS EVOLUTION OF Al-ALLOY AA 3004, PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING

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## Abstract

Equal Channel Angular Pressing (ECAP) is a very interesting method for modifying the microstructure in producing materials with ultrafine grain in sub-micrometer or nanometre range. Experiments demonstrate that these ultrafine structures may exhibit, by comparison with large - grained polycrystals, major differences in some fundamental properties. A nanostructured AA 3004 alloy was used to investigate the evolution of microhardness and microstructure on the cross-sectional plane X, after processing by ECAP at room temperature for up to six passes. The measurements show the average microhardness increases significantly after two passes with additional increases in subsequent passes. Microhardness values increase by more than two times after 6 passes. There is a reduction in grain size from of ~ 50µm in the initial condition to ~ 12 µm after 6 passes. The results also show that the microstructure and the microhardness evolve with increasing strain so that, after a total of 6 passes, the structure is almost homogenous throughout the cross-sectional plane of the billet.

**Keywords:** Al alloy; SPD; ECAP; micro-hardness; homogeneity

## 1 Introduction

Extensive studies of equal-channel angular pressing (ECAP) and other severe severe plastic deformation (SPD) techniques, over the past two decades [1–3] have established the processing techniques and have shown the possibility of producing ultrafine grain (UFG) structure in many metals and alloys. Grain size reduction is one of the most attractive ways of improving the mechanical properties of metallic materials. It is known that the strength of all polycrystalline materials is related to the grain size, through the Hall-Petch equation which predict an increase in yield strength ($\sigma_y$) with a decrease in grain size (d): $\sigma_y = \sigma_0 + kd^{-1/2}$ [4, 5].

Several different SPD procedures are now available but the most popular appears to be Equal Channel Angular Pressing (ECAP), that was first proposed in the 80 s, by Segal. In processing by ECAP the sample is pressed through a die containing two channels equal in cross-section, intersecting at an angle $\varphi$ that is generally close to $90^o$ (Fig. 1). Since the sample emerges from

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the die without experiencing any change in the cross-sectional dimensions, repetitive pressings may be undertaken in order to impose very large strains. In principle, the shear strain imposed on the billet during ECAP is homogeneous [2,3] but in practice, the strain may be affected by several factors that lead to inhomogeneities in the internal microstructure [19,20]. Microhardness measurements became a standard procedure for evaluating the strength and the level of homogeneity in samples processed to produce ultrafine grain sizes. Thus, by taking measurements along linear traverses it is possible to obtain quantitative information on the variations of hardness and homogeneity on selected planes of sectioning after ECAP. In the last decades several studies focused on the mechanical behavior and the microstructural evolution of pure Al and Al alloys processed by ECAP [6-15]. However, very little information is available at present on the evolution of microhardness and on degree of homogeneity existing along the orthogonal and longitudinal axes of billets processed by ECAP [17,18], although this information is important if the pressed billets will be used in industrial application. The present research was undertaken with the objective to use microhardness measurements to evaluate the level of homogeneity within the pressed sample of AA 3004, processed by ECAP.

Earlier investigations, conducted on samples processed by either ECAP [10-15] or high-pressure torsion (HPT) [21], demonstrated a direct correlation between microhardness measurements and the average grain sizes determined using transmission electron microscopy. Accordingly, it is reasonable to anticipate that a homogeneity in the hardness measurements will correspond also to a reasonable homogeneity in the internal microstructure. Earlier reports described the evolution of homogeneity in the alloy Al-6061, where after 6-passes neglegible increase in the micro-hardness is observed [17,18]. Hence, in this study we present our results of the micro-hardness changes upon 1 to 6 individual passes. Furthermore, scanning electron microscopy (SEM) was employed in order to establish the relation between the average grain size GS (*d*) of each processed sample and its micro-hardness (Hv).

## 2 Experimental procedure and materials

The experiments were conducted using a commercial Al-3004 alloy with a composition, in wt. %, of 1.1 % Mn, 1,2 % Mg, 0.6% Fe, 0.4% Cu, and 0.4% Si. This alloy is commonly used for the production of beverage can bodies, for construction, roofing, Al-ceilings, shutters, computer cases, decoration, etc. Samples for ECAP were cut in the form of cylinders with the lengths of ~10 cm. They were subjected to ECAP at room temperature, using a solid die containing a single channel, having a diameter of 1 cm, bent into an L-shaped configuration. Within the die there was an angle of $90^o$ between the two parts of the channel and an additional angle close to $20^o$ at the outer arc of curvature where the two channels intersect. Using this die configuration gives an imposed strain of ~ 1 on each passage of the sample through the die [1]. Since the cross-sectional dimensions of the cylinders remain unchanged in ECAP, samples were deformed repetitively for up to six passes through the die giving a maximum total strain of ~ 6 and identified by the number of passes as 1X, 2X, 3X etc. Prior to pressing each billet was coated with a lubricant containing a suspension of molybdenum disulfide in a mineral oil, to decrease the friction.

All samples were pressed using route $B_C$ in which the sample is rotated by 90° in the same sense between each consecutive pass [1], where this processing route was selected because it leads most expeditiously to an array of equiaxed grains, separated by an array of boundaries with high angles of misorientation**.** The pressed billets were sectioned perpendicular to their longitudinal axes and all microhardness measurements were taken on the cross-sectional X plane (Fig. 1). Each billet was carefully polished to a mirror-like finish and the Vickers microhardness, Hv, were taken on the surface of each sample using a HSV- 30 SHIMADZU microhardness tester equipped with a Vickers indenter. For each measurement, a load of 300 gf was applied for a dwell time of 15 s. On the cross-sectional plane three traverses were made at distances of 2.0 mm from the central line and for each separate traverse, the individual measurements were recorded in incremental steps of 250 μm. The individual microhardness measurements were plotted against the position on the cross-sectional plane and also in a three dimensional form, to provide a direct visual representation of the data. The microhardness of the material in the unpressed condition was also measured to provide a comparison with the as-pressed billets.

A digitalized scanning electron microscopy (SEM) system of JEOL JSM-T220A SEM was used as an imaging tool for samples obtained after 1- 6 passes. Prior to the SEM scanning, the billet surfaces were etched with 5 % solution of hydrofluoric acid. Grain size was estimated with HGS Measure software and Scanning probe image processor (SPIP) for statistical operations.

## 3 Results and discussions

In this work, the individual values of the Vickers microhardness were recorded along three separate traverses for each sample, and in every traverse there was a total of 15 individual measurements. **Fig. 1-6** plot the values of microhardness for the central traverse and the two traverses adjacent to, but displaced by 2.0 mm from the central one: results are shown after

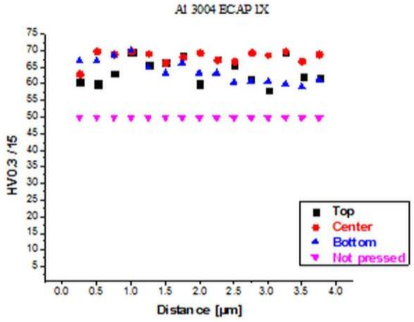


**Fig.1** Micro-hardness plots after a 1 pass on the X plane

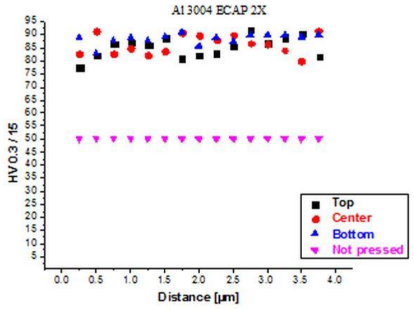


**Fig.2** Micro-hardness plots after 2 passes on the X plane

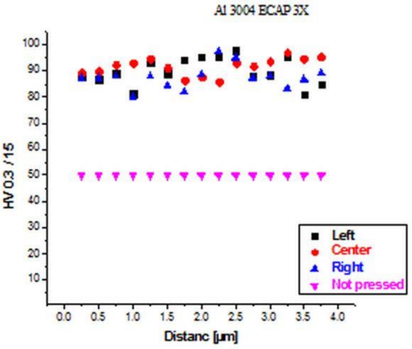


**Fig.3** Micro-hardness plots after 3 passes

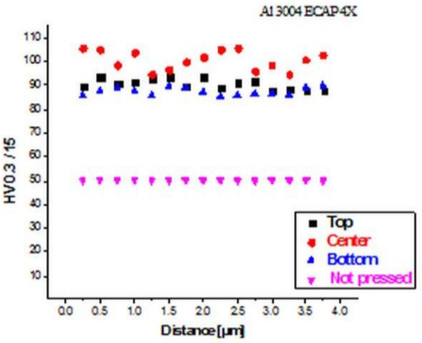


**Fig.4** Micro-hardness plots after 4 passes

(**Fig. 1**) one pass, (**Fig. 2**) two passes, (**Fig. 3**) three passes, (**Fig. 4**) four passes, (**Fig. 5**) five passes and (**Fig. 6**) six passes, respectively.

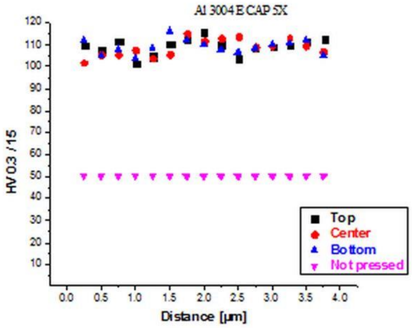


**Fig.5** Micro-hardness plots after 5 passes

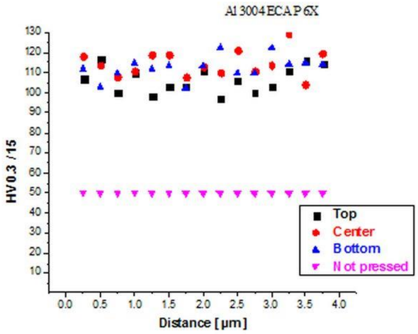


**Fig.6** Micro-hardness plots after 6 passes

The lower dashed lines in all figures, represent the value of microhardness in the unpressed condition. In order to provide a direct and simple visual representation the data are plotted in a three-dimensional form, wherein the microhardness values represented on the vertical axis, and these plots are shown on the right hand side of **Figs. 1-6**. The average values of Hv against the number of passes in ECAP are shown in **Fig. 7**.

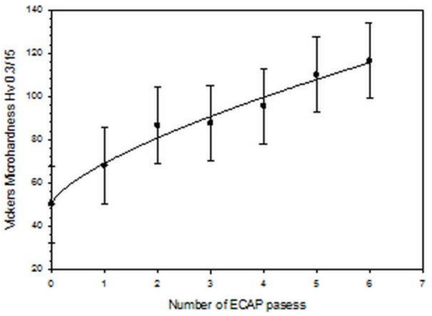


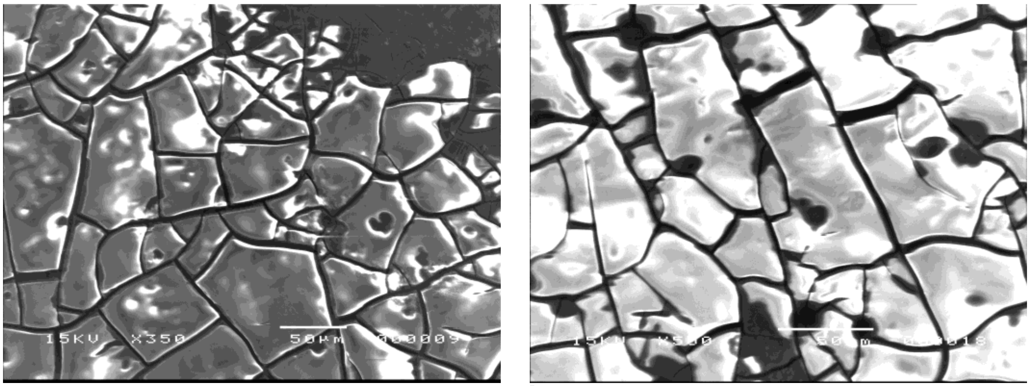
**Fig.7** Vickers micro-hardness vs ECAP passes

These plots demonstrate that the hardness increases after a single pass from an initial value of ~ 50 to values for all traverses within the range of ~ 60 –120 and there is an significant increase after two passes so that almost all values of Hv are larger than 80. This increase continues to three to six passes when almost all values of Hv are about 120.

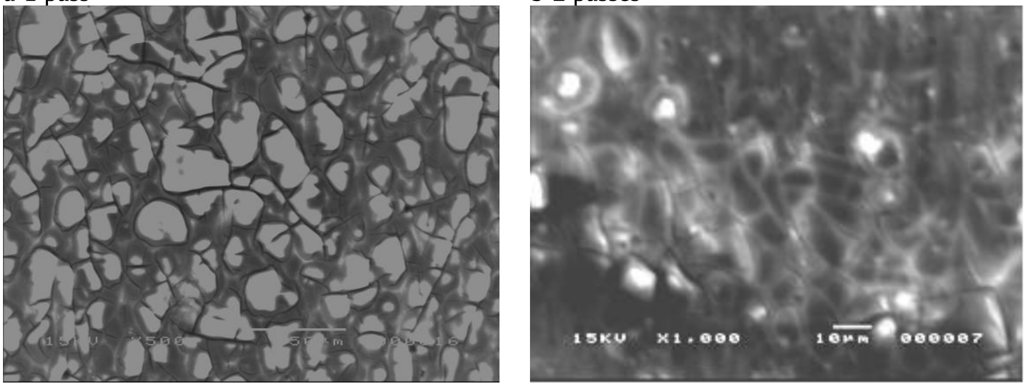
It is apparent from **Fig. 1 to 6** that despite some scatter in the average value of Hv, the measurements taken after one, to six ECAP passes, along the three traverses, are essentially constant. A second conclusion is that the values of Hv recorded adjacent to the top and bottom surface of the billet, are slightly lower than in the center. After six passes in **Fig. 6**, the hardness values are almost homogeneously distributed through the cross sectional plane, for the three traverses. These results confirm, that processing by ECAP provides excellent homogeneity on cross-sectional plane and there is an evolution towards greater homogeneity with increasing number of passes through the die (6 passes).

As shown in **Fig. 8** the hardness increases by aproximately a factor of two after 2 passes and thereafter the microhardenss continues to rise but to a smaller amounts. These trends are generally consistent with earlier data where the hardness measurements were taken on X plane [11,17,18].

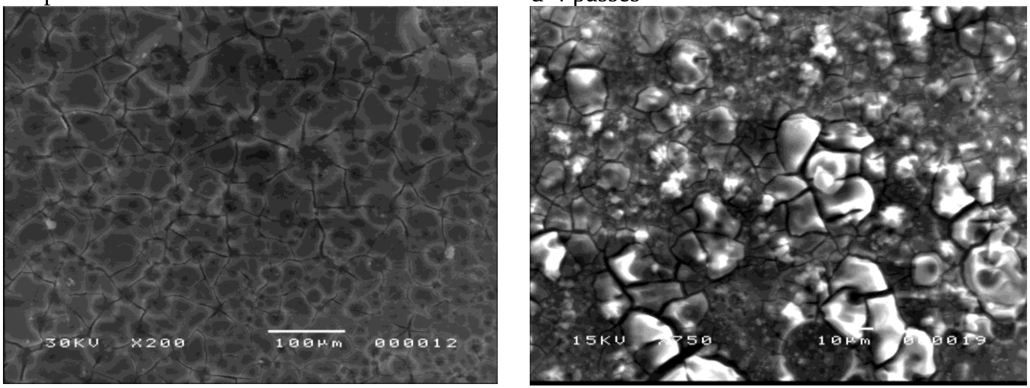
Earlier studies demonstrated there is a good correlation between the values recorded in microhardness measurements and the internal microstructures within the material [19-21]. Accordingly, it is concluded that processing by ECAP produces an excellent homogeneous microstructure in the the cross-section of the billet of Al-3004 material.



a-1 pass b-2 passes



c-3 passes d-4 passes



e-5 passes f-6passes

**Fig.8** Microstructure of AA3004 Al-alloy upon single (1X) to six (6X) ECAP passes

A more visual representation may be achieved by plotting the data in a three-dimensional form, wherein the microhardness values represented on the vertical axis. These plots confirm the increasing hardness with increasing numbers of passes and the gradual evolution into an essentially homogeneous microstructure after six passes of ECAP. Earlier reports described the development of homogeneity in the Al-6061 alloy cross-sectional planes, and on the longitudinal plane [17,18]. The present results are consistent with these reports.

**Fig. 8 a-f** presents SEM microstructure of AA3004 samples processed by ECAP for single pass, 2, 3, 4, 5 and 6 passes, respectively. All the individual images reveal transition of the compact large grain microstructure to smaller grain microstructure, depending on the number of ECAP passes.

The mean grain size(GS=*d*) of each ECAP processed sample was determined by the linear intercept method using the HGSMeasure software. The average grain size in dependence of ECAP passes is given in **Fig. 9**, where the first point (zero passes) refers to the unpressed alloy. The initial grain size of ~ 50μm after 6 passes is reduced to ~ 12 μm; the significant grain refinement was observed after 3 passes, while thereafter the grain size continues to decrease with very small amounts.

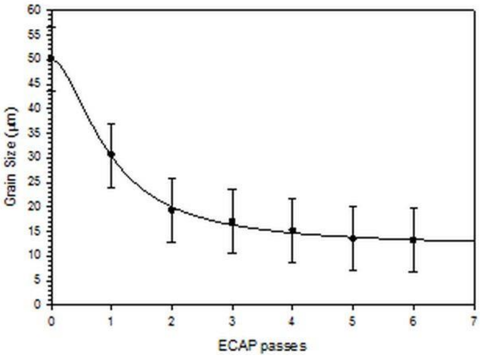


**Fig.9** Dependence of average grain size (*d*) on the number of ECAP passes

The results show that both the micro-hardness and microstructure evolve in a consistent manner. For both parameters, there is a gradual evolution with increasing strain (number of passes) so that, after 6 passes of ECAP, there is an almost homogenous structure throughout the cross-sectional plan of billet.

It was demonstrated that the reduction in the grain size lead to a significant improvement in the strength of the studied alloy at ambient temperatures.

## 4 Conclusions

1. Samples of commercial Al-3004 alloy were processed by ECAP at room temperature through one, two, up to six passes. Measurements of the Vickers microhardness Hv were recorded on cross-sectional plane X along three traverses in order to investigate the evolution of microhardness. The microstructures were examined by SEM and the grain size of the pressed material was determined using the linear intercept method.
2. The results show that the microstructure and the microhardness evolve with increasing strain so that, after a total of 6 passes, the structure is almost homogenous throughout the

cross-sectional plane of the billet. The values of Hv recorded adjacent to the top and bottom surface of the billet, are slightly lower than in the center, but after six passes the hardness values are almost homogeneously distributed through the cross sectional plane, for the three traverses.

3. The average microhardnes increases by aproximately a factor of two after 2 passes and thereafter the microhardenss continues to rise but to a smaller amounts.
4. There is a reduction in grain size from of ~ 50μm in the initial condition to ~ 12 μm 6 passes.

The present results are consistent with earlier reports on evolution of homogeneity of Al alloys processed by ECAP.

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