

Microstructural Features and Mechanical Properties after Industrial Scale ECAP of an Al-6060 Alloy

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Abstract: Future applications of ultrafine-grained, high performance materials produced by equal-channel angular pressing (ECAP) will most likely require processing on an industrial scale. There is a need for detailed microstructural and mechanical characterisation of large-scale, ECAP-processed billets. In the present study, we examine the microstructure and mechanical properties as a function of location and orientation within large (50 x 50 x 300 mm³) billets of an Al-6060 alloy produced by ECAP (90° channel angle) with different magnitudes of backpressure. The internal deformation is analysed using a grid-line method on split billets. Hardness is recorded in longitudinal and cross-sectional planes. In order to further characterise the local, post-ECAP mechanical properties, tensile tests in different layers are performed. Moreover, low-voltage scanning transmission electron microscopy observations highlight relevant microstructural features. We find that the homogeneity and anisotropy of mechanical properties within the billets depend significantly on the geometry of the shear zone. We demonstrate that deformation gradients can be reduced considerably by increasing the backpressure: The opening-angle of the fan-shaped shear zone is reduced from $\psi \approx 20^\circ$ to $\psi \approx 7^\circ$ when the backpressure is increased from 0 to 150 MPa. Backpressures of 150 MPa result in excellent homogeneity, with a relative variation of tensile mechanical properties of less than 7 %. Our investigation demonstrates that ECAP is suitable for processing homogenous, high performance materials on a large scale, paving the way for advanced industrial applications.

Introduction

Manufacturing, characterisation and optimisation of ultrafine-grained bulk materials processed by ECAP has been of scientific interest for several years. Ultrafine-grained (UFG) materials typically exhibit average grain sizes in the range of 100 nm to 1 μ m. Compared to their conventional, coarse-grained counterparts, these materials often exhibit a significantly increased strength [1-4]. The majority of previous investigations have been done on materials processed by ECAP in laboratory scale. Only a rather small number of investigations were focused on large-scale ECAP [5-8]. Considering the needs of future practical applications, there clearly is a need for scaling up the ECAP technique. Moreover, detailed microstructural and mechanical characterisation of large-scale, ECAP-processed billets are required to broaden our understanding of size and scaling effects when scaling up from laboratory to industrial dimensions. In this paper, we report on the homogeneity of an UFG Al-6060 alloy processed by large-scale ECAP with different magnitudes of backpressure (BP). We examine the local microstructure and the corresponding mechanical properties, and we analyze the influence of shear zone geometry, which can be altered by applying different magnitudes of BP.

Materials and Experimental Procedure

Extruded bars (square cross-section: 50x50 mm²) of commercial purity Al-6060 (see Table 1) were solid-solution annealed at 525 °C for 2 hours, followed by quenching in water. The annealed material exhibits a grain size in the range of 200 to 500 μm. The billets were then processed at room temperature in an ECAP-die with an internal angle of $\Phi = 90^\circ$, resulting in an effective strain of approximately 1.1 [9]. The pressing speed was 20 mm s⁻¹. Different billets were processed by one pass each, with BPs of 0, 75, 150 or 200 MPa. To illustrate size, figure 1 shows a large-scale billet (50x50x500 mm³) processed by ECAP in the solution treated condition without BP.

Table 1: Chemical composition (wt.-%) of the investigated Al-6060 alloy.

	Si	Fe	Cu	Mn	Mg	Zn	Ti	V	Al
Al-6060	0.44	0.18	0.02	0.03	0.50	0.02	0.01	0.01	bal.

In order to characterise the geometry of the shear zone as a function of the applied BP, a ‘grid-line method’ was used: Prior to ECAP, billets were cut lengthwise, and an equidistant laser grid (squares of 5x5 mm²) was prepared on the interior surfaces (see also figures 3 and 4). Furthermore, hardness (HBW 62.5/2.5) was measured on the cross-sectional and longitudinal planes after ECAP to characterize the in-plane homogeneity of the post-ECAP deformation.

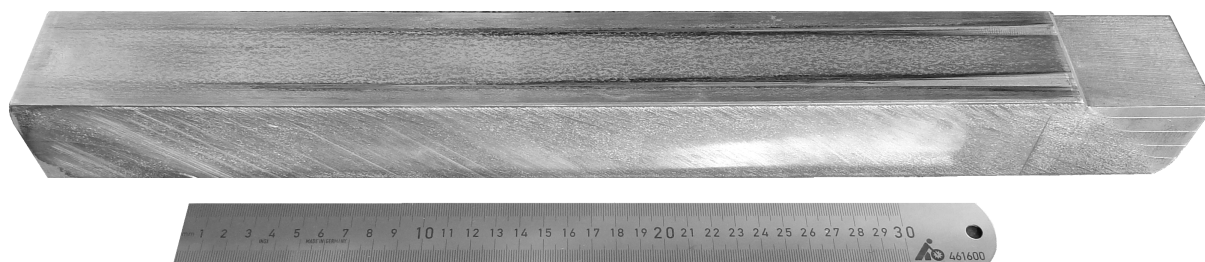


Fig. 1: ECAP-processed billet (50x50x500 mm³).

To investigate the local, post-ECAP mechanical properties, tensile tests were performed at room temperature with an initial strain rate of 10⁻³ s⁻¹. Tensile specimens oriented in extrusion direction (x-direction, see figure 2) were taken from five different layers (i.e., different z-values). After machining, the tensile specimens were subjected to an aging heat treatment at 170 °C for 20 min in order to achieve an optimised combination of strength and ductility, as reported in [10]. Finally, microstructural features of the as-pressed material were examined by optical microscopy and low-voltage scanning transmission electron microscopy analysis (STEM) in the longitudinal plane.

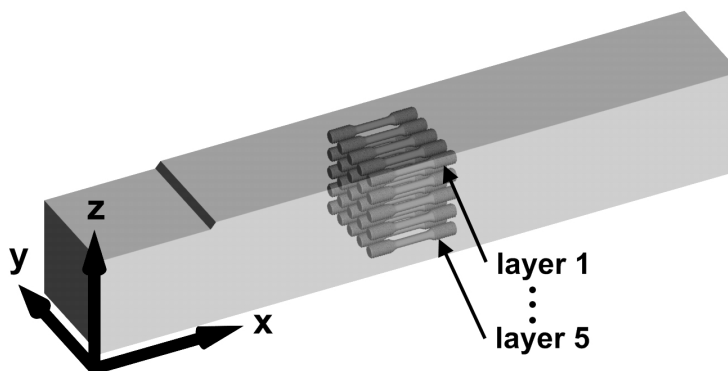


Fig. 2: Locations of tensile specimens and relevant directions used in this study.

Results and Discussion

Figure 3 shows the deformed laser grid in the shear zone of a billet that was ECAP-processed without BP. The opening angle of the fan-shaped zone can be estimated by considering the first deviation from linearity (i.e., the onset of curvature) of both vertical and horizontal grid-lines. Using this method, an opening angle of $\Psi \approx 20^\circ$ was determined for a billet pressed without BP. With a BP of 150 MPa, this angle can be considerably reduced to $\Psi \approx 7^\circ$, figure 4. In this case, the outer corner of the die was completely filled and no radius at the lower billet surface occurred. In figure 3, the deformation pattern of the grid lines that have already passed the shear zone exhibits some curvature from 0 mm to about 5 mm above the bottom. This indicates a reduced shear strain in the bottom region: If no BP is applied, the shear in the bottom part is superimposed with, or gradually replaced by, rotation around the y-axis. This leads to a decrease of the induced effective strain since the rotation does not contribute to the deformation. In contrast, perfectly sheared grid lines can be observed in the billet processed with a BP of 150 MPa. This is a strong indication of a more homogeneous, simple shear deformation.

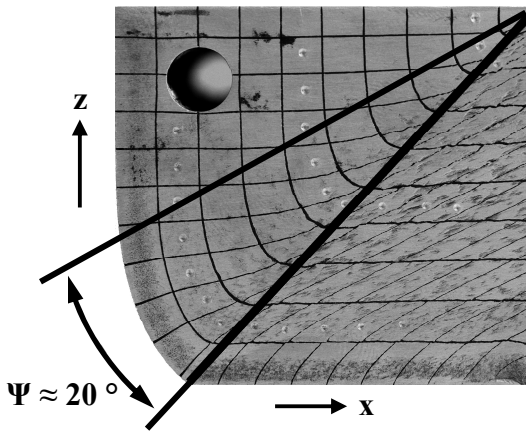


Fig. 3: Opening angle of fan-shaped shear zone after ECAP without BP.

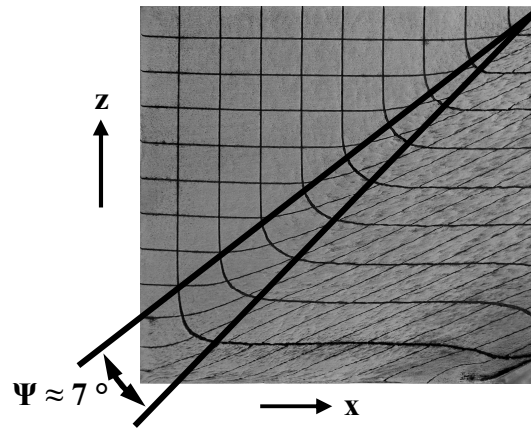


Fig. 4: Opening angle of fan-shaped shear zone after ECAP with a BP of 150 MPa.

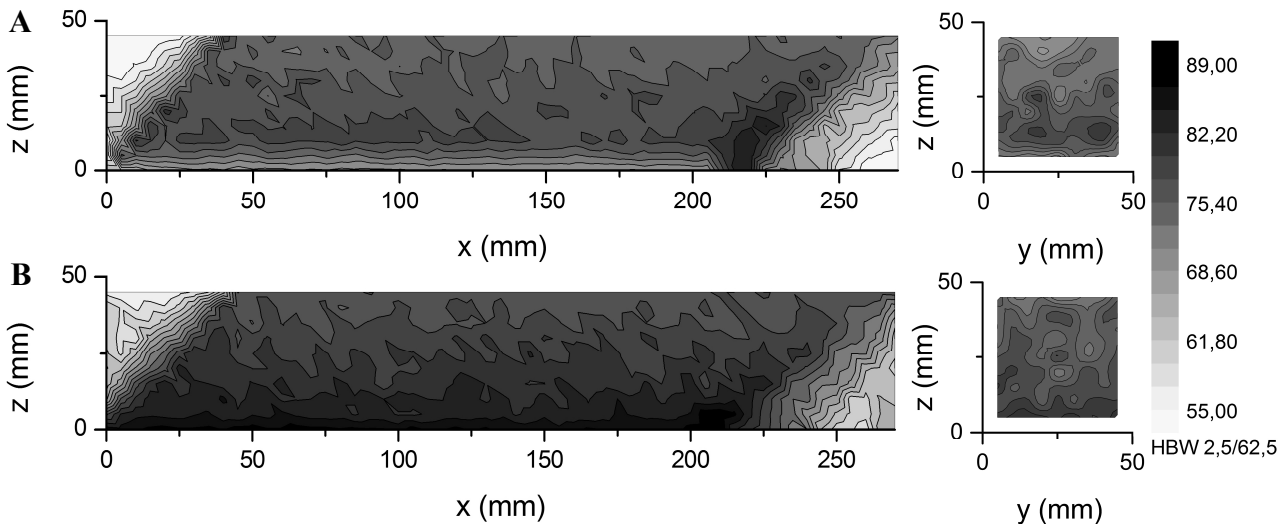


Fig. 5: Distribution of hardness in longitudinal and transversal planes. The billets were processed (A) without BP and (B) with a BP of 150 MPa.

Figure 5 shows the results of hardness measurements taken from the longitudinal and transversal planes of the Al-6060 alloy after one pass without and with (150 MPa) BP. Each longitudinal (transversal) plot was calculated from 550 (81) indents, with a spacing of 5 mm. It is obvious that the applied BP also influences the hardness distribution. Processing *without BP* (figure 5 A) leads to

a heterogeneous distribution of hardness from top to bottom of the billets. Hardness increases from the top of the billet to a region 10-15 mm above the bottom to a maximum of 80 HBW. Below this maximum, a local minimum (70 HBW) is observed. It is likely that this minimum occurs due to the large opening angle of the shear zone, which results in a lower shear strain at the bottom of the billet. In contrast to the observed variations in z-direction, the lengthwise distribution is very homogeneous. The hardness along x-direction is almost constant, with the exception of a rather localised maximum that occurs in regions deformed at the beginning of ECAP-processing (see figure 5 A at $x \approx 220$ mm). Figure 5 B represents the hardness distribution in a billet processed with a BP of 150 MPa. A local minimum at the bottom of the billet, as shown in figure 5 A, disappears due to the applied BP. The same observation can be made for the localised maximum at the beginning of the billet. Only a slight increase of hardness from top to bottom within the billet remains, which shows a deviation of less than 10 % (for comparison, commercially extruded materials exhibit deviations of about 10 %).

In order to evaluate the distribution (and a potential heterogeneity) of local mechanical properties, we now consider the results of tensile testing. Figure 6 summarizes the stress-(plastic) strain curves of the material in different layers (see figure 2) after ECAP-processing *without* BP. The 0.2 % proof stress varies from 216 MPa (layer 1) to 255 MPa (layer 4), which is equivalent to a relative deviation of ≈ 18 %. The corresponding values of ultimate tensile strengths (UTS) vary in a similar range. Layers 1 and 2 exhibit the lowest, and layer 4 the highest, UTS, with a maximum difference of 29 MPa. Parameters describing ductility vary between the considered layers as well - the elongation at fracture and the uniform elongation (UE) differ by about 7 % and 1.5 %, respectively. Figure 7 shows the stress-(plastic) strain curves of the material in the different layers after ECAP *with* a BP of 150 MPa. It is obvious that deviations between the mechanical behaviour of individual layers are significantly reduced. In this case, the 0.2 % proof stresses of all layers are close to that of layer 4 in the billets processed without BP. The relative deviation of proof stresses as well as of UTSs within these layers is reduced to less than 7 %. Moreover almost no differences in terms of UE are observed.

The absolute deviations of UTS and UE as a function of the applied BP are compared in figure 8 (figure 8 includes additional, preliminary results for BPs of 75 and 200 MPa). These data clearly demonstrate that BP plays a key role in homogenizing the distribution of mechanical properties inside the billet.

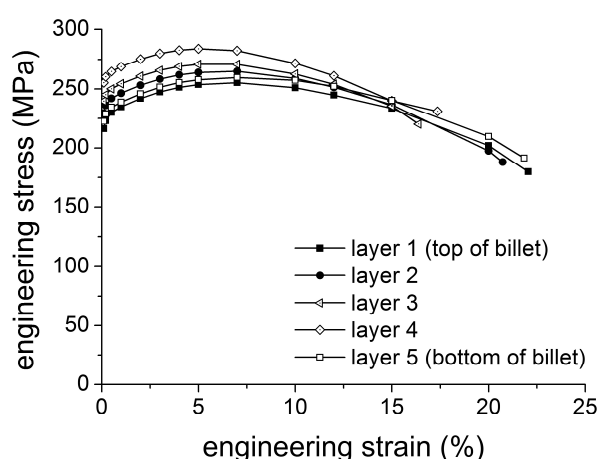


Fig. 6: Stress-(plastic) strain curves for the material after ECAP-processing with no BP in x-direction for different layers within the billet.

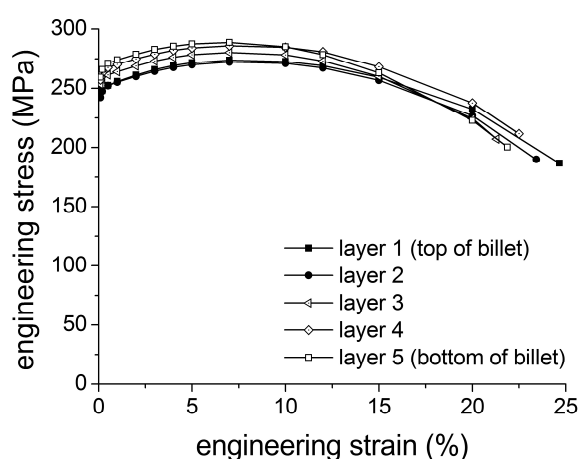


Fig. 7: Stress-(plastic) strain curves for the material after ECAP-processing with BP (150 MPa) in x-direction for different layers within the billet.

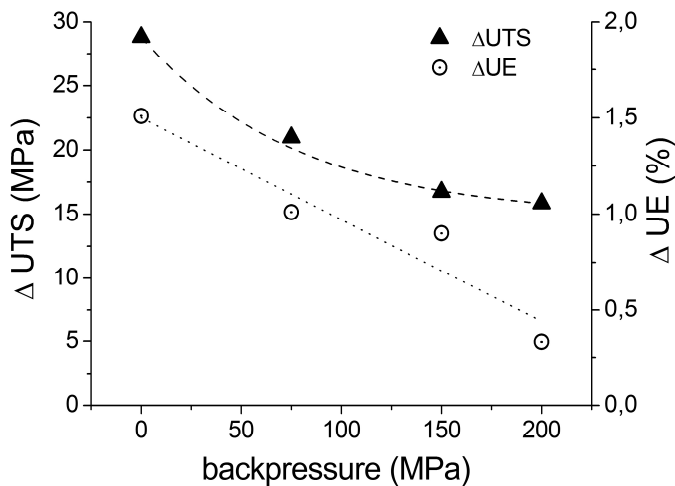


Fig. 8: Absolute deviations of UTS and UE in x-direction between different positions (layers) in the billet as a function of BP during ECAP.

Figures 9 and 10 show the microstructure in the longitudinal plane (x-z-plane, see also figure 2) of the ECAPed material, processed *without BP* in layer 1 (lower tensile strength, figure 9) and layer 4 (high tensile strength, figure 10). The optical micrographs (figures 9 A and 10 A) indicate the formation of micro-shear bands that are typical for simple shear deformation of aluminium. The STEM micrographs (B, C) show a cell structure with a cell/subgrain-size less than 1 μm . Interestingly, despite the different mechanical properties, no distinct differences in terms of cell size or dislocation density between these two locations could be identified in our preliminary STEM analysis. We note that

Xu et al. [11] reported similar observations; they documented a somewhat higher area fraction of equiaxed grains in regions of higher hardness, and more elongated grains in low-hardness regions. They [11] argue that the grain shape tends to become more equiaxed when the imposed strain is sufficiently large. While there is a need for further STEM characterization to confirm the microstructural tendencies for a larger volume of the ECAPed material, our STEM results are in good agreement with the observations of Xu et al., and similar mechanisms may likely result in the differences in mechanical properties documented in figures 6 to 8. Finally, considering local maxima and minima in the hardness distributions of individual billets (in particular at no or low BP), further investigations (most importantly: X-ray analysis) are needed to take the evolution of textures at different locations into account.

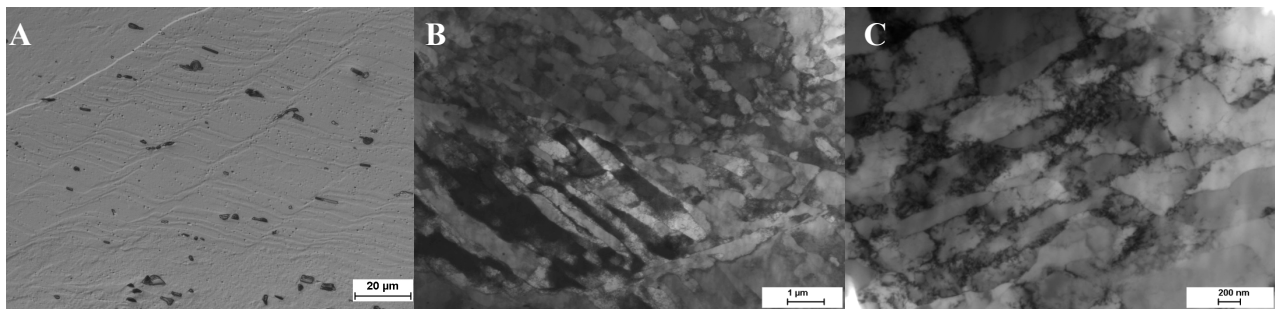


Fig. 9: Microstructure of the ECAP-material processed without BP in layer 1, analyzed by (A) optical microscopy, (B) and (C) STEM.



Fig. 10: Microstructure of the ECAP-material processed without BP in layer 4, as observed by (A) optical microscopy, (B) and (C) STEM.

