Abstract
An apparatus for verification of Equal Channel Angular dressing (ECAP) technique was installed at the VŠB–Technical University Ostrava and it was used for investigation of influence of deformation on development of structure and mechanical properties. Microstructural development of aluminium alloy 6082 during ECAP pressing was investigated to understand the mechanisms of grain refinement and strain accommodation. The samples were extruded at room temperature. Cross-section of original samples was 20 x 20 mm and their length was 110 mm. Deformation forces were measured during extrusion, resistance to deformation was calculated and deformation speed was determined approximately. Analysis of structure was made with use of light microscopy and SEM. Mechanical properties of samples after extrusion were determined by tensile test and by so called penetration test.

Keywords: aluminum alloys, ECAP, structure, mechanical properties

1. INTRODUCTION

Extrusion by ECAP method enables obtaining of a fine-grained structure in larger volumes. Products made by this technique are characterised by high strength properties, Fig. 1.

\[ \sigma_y = \sigma_0 + k d^{-1/2} \]

where \( \sigma_y \) is the yield stress, \( \sigma_0 \) is a material’s constant for the starting stress of dislocation movement (or the resistance of the lattice to dislocation motion), \( k \) is the strengthening coefficient (a constant unique to each material), and \( d \) is the average grain diameter.

Fig. 1 Schematic representation of the variation of yield stress as function of grain size in mc, ufc and nc metals and alloys [1]
The Hall–Petch relation predicts that as the grain size decreases the yield strength increases. The Hall–Petch relation was experimentally found to be an effective model for materials with grain sizes ranging from 1 millimetre to 1 micrometre [2-3]. Consequently, it was believed that if the average grain size was decreased even more to the nanometre length scale, the yield strength would increase as well. However, experiments on many nano-crystalline materials demonstrated that if the grains reached a size small enough, the critical grain size, which was typically less than 100 nm, the yield strength would either remain constant or decrease with the decreasing grain size [4]. This phenomenon has been termed as reverse or inverse Hall–Petch relation. A number of different mechanisms have been proposed for this relation. As suggested by Chinh et al., they fall into four categories: (1) Dislocation based (2) Diffusion based (3) Grain boundary shearing based (4) Two phase based. Other explanations, that have been proposed to rationalize the apparent softening of metals with nano-sized grains, include poor sample quality and suppression of dislocation pileups [5]. Many of the early measurements of a reverse Hall–Petch effect were likely the result of unrecognized pores in samples. The presence of voids in nano-crystalline metals would undoubtedly lead to their weaker mechanical properties. The pileup of dislocations at grain boundaries is a hallmark mechanism of the Hall–Petch relationship. However, once grain sizes drop below the equilibrium distance between dislocations, this relationship should no longer be valid. Nevertheless, it is not entirely clear what exactly the dependency of yield stress should be on grain sizes below this point.

2. DEVELOPMENT OF STRUCTURE

Influence of magnitude of plastic deformation on properties of metallic materials is connected with an increase of internal energy. Internal energy increases right to the limit value, which depends on the manner of deformation, purity, grain size, temperature, etc. As a result of non-homogeneity of deformation at the ECAP technique the internal energy gain differs at different places of the formed alloy. For example the value of internal energy is different in slip planes, at boundaries and inside cells. It is possible to observe higher internal energy also in proximity of precipitates, segregates and solid structural phases. For usual techniques, pure metals, medium magnitude of deformation and temperatures, the value of stored energy are said to be approx. around 10 J.mol⁻¹ [6,7]. At cold extrusion density of dislocations increases with magnitude of plastic deformation [8]. Density of dislocations depends linearly on magnitude of plastic deformation in accordance with the well-known equation [9]:

\[ \rho = \rho_0 + K \cdot \varepsilon \]  

where \( \rho_0 \) is initial dislocation density (10¹⁰ to 10¹² m⁻²), \( K \) is a constant, \( \varepsilon \) is magnitude of deformation.

Flow stress necessary for continuation of deformation is function of number of lattice defects [10]:

\[ \tau = \tau_0 + k \cdot G \cdot b \cdot \rho^\frac{1}{2} \]  

where \( \tau_0 \) is initial flow stress, \( k \) is a constant (\( k = 0.3 \)), \( G \) is modulus of elasticity in shear (\( G_{6082} \approx 25\text{GPa} \)) and \( b \) is Burgers’ vector (\( b = 0.3 \) to 0.4 nm).

3. EXPERIMENTAL PROCEDURE

The objective of experiments consisted in verification of deformation behaviour of the given alloy, determination of resistance to deformation, formability and change of structure at extrusion of alloys. Content of individual elements in the alloy is given in the Tab. 1.

<table>
<thead>
<tr>
<th>Contents of elements</th>
<th>Mg</th>
<th>Si</th>
<th>Mn</th>
<th>Fe</th>
<th>Cu</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>In mass [%]</td>
<td>1.10</td>
<td>0.88</td>
<td>0.92</td>
<td>0.45</td>
<td>0.09</td>
<td>0.20</td>
</tr>
</tbody>
</table>
During the process a metal billet is pressed through a die consisting of two channels, equal in cross section and intersecting at an angle $\Phi$. The billet undergoes essentially simple shear deformation but it retains the same cross-sectional geometry, so that it is possible to repeat the pressings for a number of passes, each one refining the grain till the extent, which is determined by the material characteristics.

Deformation forces were measured during extrusion and pressures in the die were calculated. At extrusion with the radius of rounding of edges ($R_v = 2$ mm; $R_{vn} = 5$ mm) the pressure in the die varied at the 1st pass around $\tau_{\text{max}} = 620$ MPa, and it gradually increased in such a manner that at the fourth pass its magnitude was approximately $\tau_{\text{max}} = 810$ MPa. At extrusion through a die with smaller radii of rounding ($R_v = 0.5$ mm; $R_{vn} = 2$ mm) the pressure at the first pass was approx. $\tau_{\text{max}} = 780$ MPa, and at the third pass it was approx. $\tau_{\text{max}} = 1560$ MPa [11]. Significantly higher values of resistance to deformation and strengthening at extrusion are related to high absolute value of octahedral stress, which either contributes to more difficult formation of dislocations or decelerates their movement. Another factor, which influences significantly flow stress and development of micro-structure, is the angle $\Phi$, which is formed by the axis of vertical and horizontal channel. This angle determines magnitude of shearing strain in individual passes and it can be expressed by the relation:

$$\gamma = 2 \cotg(\Phi/2)$$  \hspace{1cm} (4)

Shearing strain at the angle $\Phi = 90$ achieves the value 2, and normal deformation the value 2.3. Smaller angle $\Phi$ leads to higher shearing stress at each pass. We have checked the size of the angle $\Phi$ in the range from $90^\circ$ to $120^\circ$ with use of technological route $B_C$. We have ascertained, that refining of grains is the most efficient (under the same magnitude of deformation), at the angle of $90^\circ$. This is given by the fact that two slip planes in the sample make in this case the angle of $60^\circ$. For materials, forming of which is more difficult, it is more advantageous to apply the angle $\Phi = 120^\circ$ together with higher extrusion temperature. It is possible to calculate the magnitude of accumulated deformation from the relation:

$$\varepsilon_{\text{ac}} = \frac{N}{\sqrt{3}} \left[ 2 \cotg \left( \frac{\phi}{2} + \frac{\psi}{2} \right) + \psi \cotg \left( \frac{\phi}{2} + \frac{\psi}{2} \right) \right]$$  \hspace{1cm} (5)

where $N$ is number of passes through a die, $\Phi$ is angle channels, $\Psi$ is additional angle.

4. EXPERIMENTAL RESULTS AND DISCUSSION

4.1. Micro-structure
Structure of initial original samples is shown in Fig. 2 and structure of samples after individual passes is shown in Fig. 3 – Fig. 5.

![Fig. 2 Structure of initial sample 6082 Al alloy](image1)

![Fig. 3 Structure of the samples after the 1st pass ECAP](image2)
The structure contains ordinary inter-metallic phases corresponding to the given composition of the alloy. Average grain size in transverse direction was determined by quantitative metallography methods and it varied around 150 µm. Change of shape of the front and rear end of the sample and maintenance of integrity at individual stages of extrusion depend on the level of lubrication and on radii of rounding of edges (R_v, R_m) of the extruding channel. After individual passes accumulation of deformation strengthening occurred, the basis of which was in the formed sub-structure, which can be seen in Fig. 6 taken by an electron microscope.

Fig. 4 Structure of the samples after the 2nd pass ECAP
Fig. 5 Structure of the samples after the 3rd pass ECAP

Fig. 6 Sub-structure of 6082 aluminum alloy after ECAP extrusion : a) after the 1st pass, b) after the 2nd pass, c) after the 3rd pass, d) after the 4th pass
4.2. Mechanical properties determined by tensile test

We have verified influence of rectangular extrusion on mechanical properties with use of classical mechanical tensile test and the so called small punch test. We made from samples after application of the ECAP technique miniature test specimens for tensile test. Obtained values of tensile strength varied for the aluminium alloy within the range from $R_m = 220$ to $230$ MPa. Obtained values of tensile strength correspond very well with the values obtained by simulation and with approximate values based on the results of measurements of hardness. In the frame of evaluation of influence of the ECAP technique on mechanical properties we have made also tensile tests of investigated materials, but without application of the ECAP technique. We have tested altogether 4 test specimens with cross-section of $2.5 \times 5$ mm.

On the basis of the realised experiments we have determined tensile strengths, which, for the 6082 aluminium alloy, have been found to be $R_m = 175$ MPa. As it follows from comparison of strength properties resulting from rectangular extrusion, the strength of the aluminium alloy was increased approximately by 25%. We have performed a fractography analyses on broken halves of test specimens. Results of the above mentioned analyses, including their graphical presentations are given below.

4.3. Mechanical properties determined by small punch test

We made from the samples after application of the ECAP technique three test specimens in the form of disc with diameter of 8 mm and thickness of 0.5 mm. Basic mechanical properties were determined on the basis of small punch test, the principle of which consists in penetration of special puncher with spherical surface through the flat disc-shaped sample, which is fixed between the upper holder and the lower die [12,13]. On the basis of realised experiments it is possible to state that tensile strength of the aluminium alloy obtained by small punch test varies in the range from $R_m = 250$ to $260$ MPa, which demonstrates very good agreement with the values of tensile strength obtained by the standard tensile test ($R_m = 220$ to $230$ MPa).

4.4. Analysis of fracture areas in the aluminium alloy

Analysis of fracture areas was made with use of scanning electron microscope JEOL – JSM 5510. From visual viewpoint the fracture area looked as planar and fine-grained with indistinctive shear fractures. It was determined by detail micro-fractographical observation that fracture area was formed exclusively by mechanism of trans-crystalline ductile failure with morphology of various pits – see Fig. 7a. These cavities contained big number of minuscule particles – see Figures 7b, 7c (→).

![Fig. 7 Transcrystalline ductile fracture after ECAP](image-url)
5. CONCLUSIONS
We have verified experimentally behaviour of the 6082 aluminum alloy after extrusion. Method ECAP is a potential tool for refining of grain in polycrystalline metals. This procedure makes it possible to obtain after 4 passes the grain size of approx. 1±μm. In order to obtain an optimum microstructure it is necessary to apply more passes with turning of the sample between individual passes by 90° around the longitudinal axis. After 4 passes development of sub-structure occurs. When the die with the angle of 90° is used more intensive deformation is achieved and resistance to deformation is higher than at extrusion with higher angles. Radii of rounding of working edges of extruding channel must correspond to conditions for laminar flow of metal.

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LITERATURE