# Ondřej HILŠER<sup>\*</sup>, Stanislav RUSZ<sup>\*\*</sup>, Radek ČADA<sup>\*\*\*</sup>, Michal SALAJKA<sup>\*\*\*\*</sup>, Jan DUTKIEWICZ<sup>\*\*\*\*\*</sup>

## INFLUENCE OF SEVERE PLASTIC DEFORMATION ON MECHANICAL PROPERTIES AND STRUCTURE OF ALUMINIUM ALLOYS

## VLIV VÍCENÁSOBNÉ PLASTICKÉ DEFORMACE NA MECHANICKÉ VLASTNOSTI A STRUKTURU HLINÍKOVÝCH SLITIN

#### Abstract

Article is devoted to analysis of ECAP (Equal Channel Angular Pressing) method, which uses a high degree of deformation to achieve a very fine-grained structure of formed material. Utilization of The ECAP technology enables attainment of required properties of selected materials by using of severe plastic deformation (SPD methods). In the experimental part the influence of the number of passes through forming tool with classical geometry (angle of 90° between channels) was studied to achieve maximum hardening (expressed by deformation resistance and achieved value of hardness HV10). Also the metallographic analysis (detection of achieved grain refinement) was carried out. From comparison of results achieved at both alloys it can be stated that for given forming by ECAP method the EN AW-8006 alloy is preferable, because higher strength degree was obtained by achieving of very fine grained structure. When using the same method of forming by ECAP method the EN AW-2024 alloy has lower hardening and structure refinement.

<sup>\*</sup> Ing., Department of Mechanical Technology, Faculty of Mechanical Engineering, VŠB – Technical university of Ostrava, 17. listopadu 2172/15, Ostrava-Poruba, 708 33, Czech Republic, tel.: (+420) 59 732 9412, e-mail: ondrej.hilser@vsb.cz

<sup>\*\*</sup> prof. Ing., CSc., Department of Mechanical Technology, Faculty of Mechanical Engineering, VŠB – Technical university of Ostrava, 17. listopadu 2172/15, Ostrava-Poruba, 708 33, Czech Republic, tel.: (+420) 59 732 3315, e-mail: stanislav.rusz@vsb.cz

<sup>\*\*\*</sup> prof. Ing., CSc., Department of Mechanical Technology, Faculty of Mechanical Engineering, VŠB – Technical university of Ostrava, 17. listopadu 2172/15, Ostrava-Poruba, 708 33, Czech Republic, tel.: (+420) 59 732 3289, e-mail: radek.cada@vsb.cz

<sup>\*\*\*\*</sup> Ing., Department of Mechanical Technology, Faculty of Mechanical Engineering, VŠB – Technical university of Ostrava, 17. listopadu 2172/15, Ostrava-Poruba, 708 33, Czech Republic, tel.: (+420) 59 732 9412, e-mail: michal.salajka@vsb.cz

<sup>\*\*\*\*\*\*</sup> prof. Ing.,D.Sc., Institute of Metallurgy and Materials Science, Polish Academy of Science, Reymonta street 25, Krakow, 30-059, Poland, tel.: (+48) 12 295 2861, e-mail: j.dutkiewicz@imim.pl

#### Abstrakt

Příspěvek je zaměřen na rozbor vlivu metody ECAP (Equal Channel Angular Pressing), která využívá vysokého stupně vnesené deformace, na dosažení velmi jemnozrnné struktury tvářeného materiálu. Použití technologie ECAP umožňuje docílení požadovaných vlastností u vybraných materiálů použitím vícenásobné plastické deformace (SPD metody, tj. Severe Plastic Deformation). V experimentální části by l zkoumán vliv počtu průchodů tvářecím nástrojem s úhlem mezi kanály 90° na dosažení maximálního zpevnění (vyjádřeného přetvárným odporem a dosaženou hodnotou tvrdosti HV10). Rovněž by la provedena metalografická analýza (zjištění dosaženého zjemnění zrna). Z porovnání dosažených výsledků u obou slitin lze konstatovat, že pro dané procesní parametry tváření metodou ECAP je vhodnější slitina EN AW-8006, protože dosahuje vyšší zpevnění vytvořením jemnozrnné struktury. Při použití shodného způsobu tváření metodou ECAP slitina EN AW-2024 vykazuje nižší zpevnění.

#### Keywords

severe plastic deformation, mechanical properties, fine-grained structure, forming, EN AW-2024, EN AW-8006, ECAP, hardness, transmission electron microscopy.

#### **1 INTRODUCTION**

Due to the validity of the Hall-Petch relationship (1) interest in research of materials with very fine grained structure is increasing. These UFG (Ultra Fine Grain) or NC (Nanocrystalline) materials can be classified as polycrystals with an average grain size below  $1 \mu m$ .

Yield stress can be calculated from formula:

$$\sigma_{\rm v} = \sigma_o + \mathbf{k} \cdot d^{-\frac{1}{2}} \tag{1}$$

where are:

 $\sigma_{\rm v}$  – yield stress [MPa],

- $\sigma_0$  stress for overcoming of Peierls-Nabarr friction stress of lattice[MPa],
- k constant, the measure of which is the value of shearing stress necessary for release of accumulated dislocations [–],
- *d* average grain size [nm].

Problematics of refining structure achieving is constantly being innovated by new knowledge, but the basic distribution of methods for UFG or NC materials production exists, so top-down and bottom-up methods.

Top-down methods are characterized as a production process which uses the synthesis (the process of joining of two or even more parts to one unit) of ultra-fine particles or nano-particles with the following compaction to a samples or blanks. At this method it must be reckoned with high incidence of defects, which are undesirable.

When using the bottom-up methods a structure without pores is achieved. Constrains of primary defects is a precondition for achieving a required superior properties. Among these methods a technologies using severe plastic deformation (SPD methods) arising in formed material in order to final structure refinement belong [1].

SPD methods utilize accumulated dislocations interactions, whose grouping leads to the formation of dislocation walls. By dislocation activities the disorientation of cell blocks arises. This disorientation leads to creation of dislocation boundaries inide of grains and with their further development their disorientation (higher than  $12^{\circ}$ ) is increasing and high-angle boundaries arise. By effect of these boundaries the resulting hardening of the material, which is reflected by increased

mechanical properties, arises. Dislocation walls can further divide thus lead to creation of new subgrains boundaries therefore additional refinement of structure asise.

Methods, using severe plastic deformation, can be divided into [1]:

- forming by single doses (ECAP, HPT, TE, CEC, RCS),
- semi-continuous processes (ARB),
- continuous processes (CONFORM, C2S2, DRECE [7-8]).

The basic principle of the utilization of SPD processes leading to refinement of initial grain is a positive effect of grain size, according to validity of (1), on the final mechanical properties of formed material. Material prepared by this way can be applied to the production process and can substitute the material with higher quality, which is more expensive.

## **2 THE ECAP PROCESS**

The ECAP principle is to use a shear deformation mechanism which is generated during pressing of the sample between the vertical and the horizontal channel build in the special forming tool (Fig. 1), these channels have an angle of  $90^{\circ}$  between themselves. Construction angles made in tool have an direct influence on deformation brought to the extruded sample, according to equation (2) [2].



Fig. 1 Principle of the ECAP technology [3]

Equivalent (von Mises) strain can be calculated from formula:

$$\varepsilon_{VM} = \frac{2 \cdot N}{\sqrt{3}} \cdot \left[ 2 \cdot \cot\left(\frac{\phi}{2} + \frac{\psi}{2}\right) + \phi \cdot \csc\left(\frac{\phi}{2} + \frac{\psi}{2}\right) \right]$$
(2)

where are:

 $\varepsilon_{\rm VM}$  – equivalent (von Mises) strain [–],

- N number of passes (number of forming operations) [–],
- $\phi$  angle of transition between the vertical and the horizontal channel of ECAP tool [°],
- $\psi$  angle of length curvature transition between the vertical and the horizontal channel of ECAP tool [°].

For creation of a very fine-grained structure formed by equiaxed grains separated by highangle boundaries the angle of  $\phi = 90^{\circ}$  and the deformation rout Bc [3] when the extruded sample after separate pass is always rotated about longitudinal axis by 90° in the direction of clock hands, is the best. Using of deformation route Bc (Fig. 2) leads to the achievement of the ultra-fine grained equiaxed structure.

For increasing of efficiency of the ECAP process the formed sample can be repeatedly extruded trough channel with the use of rotation of the sample between separate passes (used deformation routs are shown on Fig. 2). When using samples rotation the activation of secondary slip systems and increasing of the amount of accumulated dislocations arise. According to the experimental publications [1, 4, 5] the highest efficiency has the combination of ECAP with using of deformation route Bc.





Fig. 2 Processing routes used at extruding by ECAP method [5]

The following experiments were focused on determining of the effect of the ECAP method with the classical channel on the refinement of the structure and mechanical properties of selected aluminium alloys.

## **3 PROGRESS OF THE EXPERIMENTS**

### 3.1 Material

For realization of the experiments were used as casted aluminium alloys EN AW-2024 and EN AW-8006 were used (see Tab. 1).

**Tab. 1** Chemical composition of aluminium alloys EN AW-2024 and EN AW-8006 in the state after casting

Material	<b>Si</b> (wt %)	<b>Fe</b> (wt %)	<b>Cu</b> (wt %)	<b>Mn</b> (wt %)	<b>Mg</b> (wt %)	<b>Zn</b> (wt %)	<b>Ni</b> (wt %)	<b>Al</b> (wt %)
EN AW-2024	0,30	0,35	4,40	0,40	1,65	0,30	0,10	rest.
EN AW-8006	0,40	1,50	0,30	0,46	0,10	0,10	-	rest.

## 3.2 Extruding by ECAP method

Forming of aluminium alloys with the use of ECAP method was realized in the laboratory of the Department of Mechanical Technology, Faculty of Mechanical Engineering, VŠB – Technical university of Ostrava at the newly renovated DP2000 hydraulic press (Fig. 3).



Fig. 3 Workplace of VŠB-TUO for materials forming by ECAP method

Samples were extruded through tool with classical geometry of the channel (scheme is seen on Fig. 1,  $\phi = 90^{\circ}$  and  $\psi = 20^{\circ}$ ) and by selected deformation route Bc (maximal 4 passes to complete 360° rotation were realized). Extrusion was carried out at temperature of  $T = 22^{\circ}$ C and chosen extrusion speed v = 45 mm. min<sup>-1</sup> (deformation speed  $\dot{\varepsilon} = 0.01$  s<sup>-1</sup>).

# **4 TESTING RESULTS**

### 4.1 Flow stress

During experimental pressing at the hydraulic press DP 2000 the flow stress of formed material can be measured via the installed oil pressure sensor in the working cylinder (Fig. 4). Measured data show the complex picture of an ECAP method impact on material hardening.



Fig. 4 Scanning of flow stress curves during the ECAP process by the TestEmotion program

Figure 5 shows the effect of the ECAP method on the achieved hardening of specimens from material EN AW-2024.



Fig. 5 Process of flow stress of EN AW-2024 alloy according to number of passes through forming tool with classical geometry (the channel angle of 90°)

Figure 6 shows the effect of the ECAP method on the achieved values of hardening of EN AW-8006 alloy.



EN AW-8006

**Fig. 6** Processes of flow stresses of EN AW-8006 alloy according to number of passes through tool ECAP with classical geometry (the channel angle of 90°)

From Fig. 5 and 6 a positive effect of the ECAP method on the final hardening of both types of tested alloys is seen. The influence of the ECAP technology on achieving the fine-grained structure is detaily analyzed in chapter 5 Metallographic analysis.



Fig. 7 Influence of ECAP method on resulting maximum hardening of tested alloys

## 4.2 Hardness measurement

The hardness measurement was realized on a universal hardness tester HPO 250. The injection of tetrahedral pyramid with load of 9.8 kg, so the Vickers HV10 method, was used.

From the results of the processes of the measured hardness according to Vickers result that the ECAP method significantly increased the hardness of tested materials of about 48 % at EN AW-2024 alloy and 56 % at EN AW-8006 alloy (after fourth pass) compared with the initial state.



**Fig. 8** Measured values of hardness HV10 at tested materials EN AW-2024 and EN AW-8006 in the initial state and after after separate passes through a channel with angle 90°



Fig. 9 Percent dependence of hardness HV10 values change compared with initial state

From the results shown in Fig. 9 is seen that a substantial increase of hardness occurs after the first pass, in which the measured values of hardness increase of 30 % (EN AW-2024 alloy) and of 31 % (EN AW-8006 alloy).

In dependence of measured values of hardness depending on the number of passes the positive effect of passes number increasing on the hardness value for both tested alloys is evident. From the results of the hardness measurement can be concluded that EN AW-8006 alloy achieves a higher degree of hardness increase (expressed in percentage – Fig. 9) compared with the EN AW-2024 alloy.

From the results of the hardness measurement can be concluded that the EN AW-8006 alloy shows a higher degree of strain hardening. This assumption must be confirmed by microstructural analysis at a transmission electron microscope. From transmission electron microscope results can be defined, whether the hardening of both alloys was occurred by strain hardening or by fragmentation of original grains.

#### **5 METALLOGRAPHIC ANALYSIS**

For samples of alloys processed by ECAP method the metallographic analysis in order to evaluate the effect of verified method on the final grain refinement of structure was realized.

The structural analysis was carried out on the optical microscope NEOPHOT 2 and samples (after  $1^{st}$  and  $4^{th}$  pass) were before evaluation mechanically grounded (sandpaper on the SiC basis with roughness 800 and 1500) and then etched (3 % of Nital solution, time of immersion from 5 to 10 seconds, followed by washing and hot air drying).

Metallographic analysis was realized on the transmission electron microscope (TEM) Tecnai G2 F20 (200 kV)



a)

b)



c)

d)



From the microstructural analysis of extruded samples of EN AW-2024 alloy is seen that the number of passes through forming tool significantly affects the grain size. After each subsequent pass due to development of high degree of deformation at the grain boundaries an intense accumulation of dislocations occurs, angles of grain boundaries are changing from low-angle to high-angle, then a considerable number of sliding planes arises, while at the grain boundaries a reaction between border and internal dislocations allowing deformation of the grains (their refinement) arises. At the EN AW-2024 alloy the assumption of the number of passes influence on the structure refinement was confirmed.





a)

b)



c)

d)

**Fig. 11** Microstructural analysis of EN AW-8006 alloy (a – on the optical microscope after 1<sup>st</sup> pass, b – on the optical microscope after 4<sup>th</sup> pass, c – on the transmission electron microscope after 1<sup>st</sup> pass, d – on the transmission electron microscope after 4<sup>th</sup> pass)

Initial average grain size of EN AW-8006 alloy was 100  $\mu$ m. At the ECAP method after 1<sup>st</sup> pass the grain size of 15  $\mu$ m was achieved. After 4 passes the average grain size of 700 nm was evaluated. Inside the grains the small defects signalling the partial recrystallization are formed.

From the metallographic analysis on the transmission electron microscope the positive effect of ECAP forming method on the structure refinement of both alloys (EN AW-2024 and EN AW-8006) can be confirmed. In initial state both kinds of aluminium alloys have coarse-grained heterogeneous structure, where the average grain size was evaluated 150  $\mu$ m (EN AW-2024) and 120  $\mu$ m (EN AW-8006). After the 1<sup>st</sup> pass the great grain size reduction arises (34  $\mu$ m, respectively 20  $\mu$ m). This result corresponds with the results of hardness measurement were the maximum hardness increasing was evaluated after the first pass through the forming tool.



Fig. 12 Influence of the number of passes through channel with the angle of 90° on the achieved average grain size of alloys EN AW-2024 and EN AW-8006

Average grain size [μm]	Initial state	1 <sup>st</sup> pass	2 <sup>nd</sup> pass	3 <sup>rd</sup> pass	4 <sup>th</sup> pass
EN AW-2024	150	34	11	9	2.5
EN AW-8006	120	20	8.5	1.8	0.7

Tab. 2 Values of the average grain size of tested alloys EN AW-2024 and EN AW-8006

From the results shown in Fig. 12 and in the Tab. 2 the positive effect of forming by ECAP method on the achieved grain refining of both alloys can be confirmed. In case of the measurement results of the average grain size of EN AW-2024 alloy is clear that after 4<sup>th</sup> pass the grain size below 1  $\mu$ m is not achieved, so it cannot be classified as a material with very fine grained structure. The structure of EN AW-8006 alloy after the 4<sup>th</sup> pass is comprised by grains having an average size of 700 nm. In this case the material with very fine grained (UFG) structure was prepared after 4<sup>th</sup> pass through forming tool with classical channel and with the above defined process parameters.

### 6 CONCLUSIONS

Components produced by ECAP method have special properties and utilization. ECAP method is particularly suitable for the production of components with small dimensions, in relatively small quantities. This also corresponds to their application in practice. The products with high strength and precisely defined properties are applied especially in transport (automotive, electrotechnics, space and aviation technologies), but also in the consumer goods industry, sports equipment, etc.

When using of these materials in precisely defined conditions the functional properties can be increased respectively the properties can be affected by an appropriate texture obtained by forming. According to the requirements on the structure of material and its mechanical properties the optimal forming parameters can be determinated so that the products guarantee the operational safety and reliable of structural elements and units. Currently we register a specific application in practice – modification of mechanical properties of aluminium alloy EN AW-5005 to ensure greater extent of its applicability for the production of cooler parts (Fig. 13).



Fig. 13 Example of application of the material formed by ECAP method in practice at production of cooler parts

From the results of realized experimental works at alloys EN AW-2024 and EN AW-8006 it can be stated that the ECAP method is suitable for production of materials with very fine grained structure, which is characterized by increased strength parameters compared with the initial state.

From comparison of results achieved at both alloys it can be stated that for given forming by ECAP method the EN AW-8006 alloy is preferable, because higher strength degree was obtained by achieving of very fine grained structure. When using the same method of forming by ECAP method the EN AW-2024 alloy has lower hardening and structure refinement.

### ACKNOWLEDGEMENT

Results in the contribution were achieved at solving of specific research project No. SP2016/73 with the name of "*Research and Development of Production and Material Systems*" (,, *Výzkum a vývoj výrobních a materiálových systémů* ") solved in year 2016 at the Faculty of Mechanical Engineering of VŠB – Technical University of Ostrava.

#### REFERENCES

- [1] ZRNÍK, J. et al. Evropská strategie výrobních procesů (4): Příprava ultrajemnozrnných a nanokrystalických kovových materiálů extrémní plastickou deformací a jejich vlastnosti. Ostrava: Repronis, 2007. 76 s. ISBN 978-80-7329-153-2.
- [2] VALIEV, R. Z., ISLAMGALIEV, R. K. and ALEXANDROV, I. V. Bulk nanostructured materials from severe plastic deformation. *Progres in Material Science*, Vol. 45(2000), pp. 103-189. ISSN 0079-6425.
- [3] FIGUEIREDO, R. B., et al. Microstructure and Mechanical properties of Pb-4%Sb alloy by equal channel angular pressing. *Materials Research*, Vol. 9, No. 1, 2006, pp. 101-106. ISSN 1516-1439.
- [4] VALIEV, R. Z. and LANGDON, T. G. Principles of equal channel angular pressing as a processing tool for grain refinement. *Progres in Material Science*, Vol. 51(2006), pp. 881-981. ISSN 0079-6425.
- [5] TAŃSKI, T., SNOPIŃSKI, P., PAKIEŁA, W., BOREK, W., PRUSIK, K. and RUSZ, S. Structure and properties of AlMg alloy after combination of ECAP and post-ECAP ageing. *Archives of Civil and Mechanical Engineering*, Vol. 16, No. 3, 2016, pp. 325-334. ISSN 1644-9665.
- [6] HELLMIG, R. J.: Hochgradige plastische Umformung durch Equal Channel Angular Pressing (ECAP): Habilitation Thesis. Germany: TU Clausthal, 2008, 141 p.
- [7] RUSZ, S., KLYSZEWSKI, A., SALAJKA, M., HILŠER, O., ČÍZEK, L. and KLOS, M.Possibilities of application methods DRECE in forming of non-ferrous metals. *Archives of Metallurgy and Materials*, Vol. 60, No. 3, 2015, pp. 3011-3015. ISSN 1733-3490.
- [8] ČADA, R. Formability Evaluation of Low-Carbon Steel Strip. Transactions of the VŠB-Technical University of Ostrava, Mechanical Series. Vol. 57, No. 1, 2011, pp. 19-28. ISSN 1210-0471.