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MULTIPLE PLASTIC DEFORMATION FOR PREPARE OF NANO-STRUCTURAL MATERIALS BY ECAP METHOD

PŘÍPRAVA NANOSTRUKTURNÍCH MATERIÁLŮ VÍCENÁSOBNÝM TVÁŘENÍM TECHNOLOGIÍ ECAP

Anotace

Vývoj nanostrukturních materiálů je v současné době intenzívně vyvíjen na významných světových vědeckých pracovištích - Soul, Fukuoka, Los Angeles, Grenoble, Los Alamos, etc. – významnými vědeckým odborníky v oblasti tváření - Furukawa, Nemoto, Dobatkin, Langdon, Valiev, Stolyarov, Zhu, Lowe, Segal, ap..Jedná se zejména o technologii ECAP (Equal-Channel Angular Pressing). Je to jedna ze základních metod umožňujících dosažení velmi jemnozrnné struktury u neželezných kovů a jejich slitin. Neželezné kovy a jejich slitiny jsou velmi dobře recyklovatelné a v mnoha případech mohou nahradit ocele. Zároveň dojde ke snížení výrobních nákladů při výrobě součástí z těchto materiálů. Nacházejí neustále širší uplatnění zejména v automobilovém, zbrojním a leteckém průmyslu. V mnoha významných světových automobilkách je vývoj zaměřen na malolitražní automobil s vysokým podílem právě hlinikových a slitinových součástí.

Abstract

The investigation of nano-structure materials is subject of concentrated efforts of major research institutions in the world - Soul, Fukuoka, Los Angeles, Grenoble, Los Alamos, etc. – and eminent scientists - Furukawa, Nemoto, Dobatkin, Langdon, Valiev, Stolyarov, Zhu, Lowe, Segal, etc. In particular this concerns ECAP (Equal-Channel Angular Pressing) technologies. This technology represents a basic method for achieving super fine granularity structures. Especially non-ferrous metals and their alloys are of primary concern. Non-ferrous metals, and their alloys are subject of an easy recycling process, and they increasingly tend to substitute steel on a larger scale. At the same time, a major decrease of production cost for these materials, and their products can be noted. Their importance for applications by automobile industry is ever growing that is also the case for military and space industries. Major car producers in the world - Opel, Audi, Jaguar, Ford, Fiat, Volvo, Toyota – have launched production of small cars that are largely made from Al and its alloys.

1 Introduction

Principles of ECAP technology

Severe plastic straining is achieved in ECAP by pressing the sample through a die as illustrated schematically in Fig. 1. The sample is machined to fit within a channel which passes through the die in an L shaped configuration [2, 4]. For the situation where the angle between the two parts of the channel is equal to 90°, the test sample will undergo straining by shear as it passes from one part of the channel to the other: this shearing is illustrated in Fig. 2. It is apparent from Fig. 1 that the sample emerges from the die without any change in the cross-sectional dimensions. Thus, this process is distinct from the more conventional metal working processes such as rolling and extrusion where there is a concomitant reduction in the cross-sectional dimensions of the work piece. In practice, it is convenient to define three separate planes within the sample associated with ECAP: these planes are indicated in Fig. 1 and they are plane X perpendicular to the longitudinal axis and planes Y and Z parallel to the side face and the top face of the sample at the point of exit from the die, respectively.

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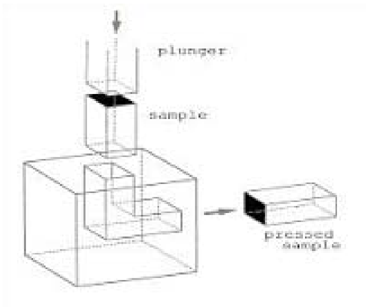


Fig.1 Schematic illustration of ECAP technology

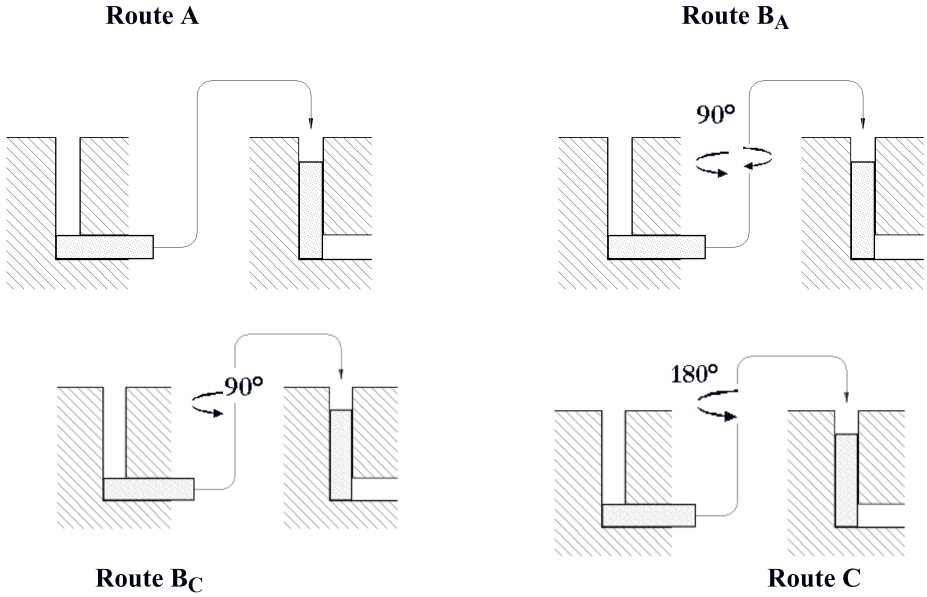


Fig. 2 The four processing routes in ECAP

To understand the effect of these different processing routes, it is instructive to examine the internal shearing patterns as illustrated in Fig. 3 where the planes labeled 1 through 4 denote the shearing which occurs on the first four pressings through the die: the planes designated X, Y and Z in Fig. 3 correspondent to the planes illustrated in Fig. 1on the as pressed sample. Inspection of Fig. 3 shows the shearing patterns are dependent upon the processing route. For example, in route C there are repetitive sheerings on the same plane whereas in route A there are two shearing planes intersecting at an angle of 90° and in routes B_a and B_c there are four distinct shearing planes intersecting at angles of 120° [4].

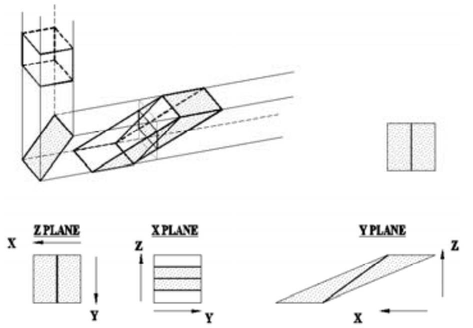


Fig.3. Shearing associated by a single passage through the die

2 Principles of shearing on passage through The ECAP die.

Since the cross-sectional area of the sample is unchanged on a single passage through the die, it is apparent that repetitive pressings may be undertaken in order to achieve very high total strains.

The strain imposed on the sample in a single passage through the die is dependent primarily upon the angle Φ between the two separate parts of the channel within the die. There is also a minor dependence upon the angle Ψ at the outer arc of curvature where the two channels intersect. In practice, however it can be shown that the imposed strain is close to ~1when Φ = 90°for all values of Ψ.

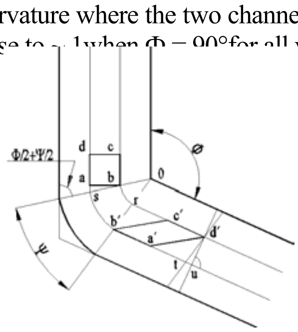


Fig. 4 Principle of equal – channel angular pressing where Φ is the angle of intersection of the two channels and Ψ is the angle subtended by the arc of curvature at the point of intersection.

In Fig.4, where Ψ represents an intermediate situation, the shear strain is γ = *a′u/d′u* , where *d′u* = *ad* and *a′u* may be obtained form the relationships *a′u* = (*a′t* + *tu*) = (*rc′* + *as*), *as* = *ad*cot (Φ/2+Ψ/2), *ab′*= *dc′*= (*as* + *os*Ψ) = *rc′*+ *od*Ψ and (*os* – *od*) = *ad*cosec (Φ/2+Ψ/2), so that a′u = *2ad*cot (Φ/2+Ψ/2) + *ad*Ψcosec(Φ/2+Ψ/2) [3]. Therefore, the shear strain for this intermediate condition is given by

$$\gamma = 2\cot\left(\frac{\phi}{2}+\frac{\Psi}{2}\right)+\Psi\cos ec\left(\frac{\phi}{2}+\frac{\Psi}{2}\right) \tag{1}$$

Deformation is dependent on single passages of sample through the channel, it is result from equation (1). Further, there is dependence on Φ angle. If the Φ angle is very small, the deformation is equal to multiple passages through the channel with big inner angle. For example, one passage through the angle of 90 degrees (mathematical equivalent to equation 1), is equal to two separate passages through the angle of 135 degrees. Often, it is difficult to use the ECAP tool with small Φ angle in the die. This problem was solved with using of tool with channel, which is created by two consecutive following big inner angles. It gives two shear planes for each protrusion. Although it is evident, that there grow up different shear system in the sample (value of Φ angle is changed). This is necessary to check out, whether the given method is accessible.

The SAED analysis was performed on target diameter 12,3 μm with microanalysis for 90 degrees angle and scheme for passage type B_c (fig. 2). Deformation achieved in every passage is reduced with growing size of Φ angle, it is result of equation 1.

Since the same strain is accumulated in each passage through the die, the strain after *N* cycles is therefore given by

$$\varepsilon_n = N\left[\frac{2\cot g\left(\frac{\Phi}{2}+\frac{\psi}{2}\right)+\psi\cos ec\left(\frac{\Phi}{2}+\frac{\psi}{2}\right)}{\sqrt{3}}\right] \tag{2}$$

Thus, the strain may be estimated from equation (2) for any pressing conditions provided the angles Φ and Ψ are known. A relationship is derived which may be used to calculate the imposed strain after any number of selected pressing cycles [1].

Influence of magnitude of plastic deformation on characteristics of the alloy AlCuMg is at the use of technology ECAP connected with increase of internal energy. Internal energy increases till the limit value, which depends on method of deformation, purity, grain size, temperature, etc. Increment of internal energy is directly related to the quantity and character of lattice defects in extruded alloy, i.e. that volume of energy absorbed by structure at deformation increases with contamination of the matrix, with reduction of grain size, with drop of deformation temperature [2].

As a result of non-homogeneity of deformation at the ECAP (selected planes and direction of slippage) the internal energy increment at different places of the formed alloy is also different. For example value of internal energy is different at slip planes, at the boundaries and inside the cells. It is possible to observe higher internal energy also in proximity of precipitates, segregations and hard structural phases. For usual technologies, pure metals, medium magnitude of deformation and temperature the value of the stored energy is said to be of approx. 10 $Jmol^{-1}$. Density of dislocations, concentration of vacancies and total surface of walls of cell structure increases at cold extrusion in proportion to magnitude of plastic deformation.

If no softening processes occur at forming, then dislocation density depends linearly on magnitude of plastic deformation in accordance with the well-known equation:

$$\rho = \rho_o + K \cdot \varepsilon \tag{3}$$

where ρ_o is a initial dislocation density,
K is a constant,
ε is magnitude of deformation.

Flow stress, which is necessary for continuing deformation, is a function number of lattice defects, particularly of dislocations, and it can be expressed by the equation:

$$\tau = \tau_o + k \cdot G \cdot b \cdot \rho^{\frac{1}{2}} \tag{4}$$

where τ_o is a value of initial flow stress,
k is a constant,
G, b are modules of elasticity in shear, Burgers' vector.

Size of sub-grains and magnitude of deformation are in direct relation, when size of sub-grains decreases with increased deformation.

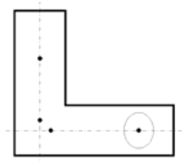
We have designed a tool and proposed a procedure for verification of development of structure at equal channel angular pressing. Normal AlCuMg alloys were used for manufacturing of the input semi-product. Further, there was made experimental tests on AlCu4Mg2 alloy by the ECAP tool.

3 Experimental verification AlCu4Mg2 alloy

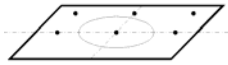
The experiments were aimed at verification of functionality of the proposed equipment, determination of deformation resistance, deformability and change of structure at extrusion of the alloy AlCu4Mg2. The experiments were made on the equipment, which is demonstrated in Figures Nos. 3 and 4. Original input examples were made from hot-formed semi-products.

Square section of the input samples was 8 x 8 x 28 mm. The samples were extruded at the temperature of approx. 20 ^{o}C [5, 6]. In order to increase deformation in the volume of the sample, the samples were turned after each internal extrusion around the longitudinal axis by 90^{o} and extruded again. Initial shape of the sample as well as shapes of sample after individual passages of extrusion are shown in the Fig 5.

For identification influence of the number of passages through on size grain, were made 4 passages through the channel. Analysis of microstructures was performed after the first and the fourth passage through. There are schematic shown places, by means of points, where was structure of the samples after passage through by method ECAP investigated, in figure 10.



Sample after the first passage through



Sample after the fourth passage through

Fig. 5 Investigated places on sample material after extrusion

There is shown microstructure of material AlCu4Mg2 (fig. 6) in the Y plane after first extrusion during indoor temperature by the passage type Bc and value of inner angle Φ equal to 90 degrees [7] .

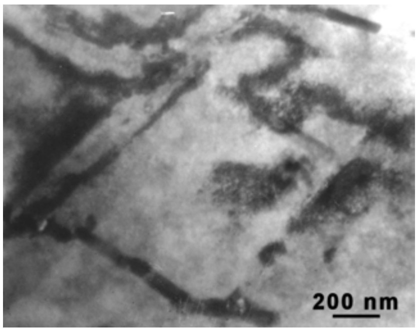


Fig. 6 Structure after the first passage by the ECAP tool
in AlCu4Mg2 alloy (middle of the sample)

Figure 7 and 8 show microstructure of material AlCu4Mg2 (Y plane) after fourth extrusion during indoor temperature by the passage type B_c and value of inner angle Φ is equal to 90 degrees.

The big boundary angles occur in the sample, at values of Φ angle among 90 – 115 degrees.

This microstructure control definitively certify, that successful utilize of ECAP method requires using channel with value of inner Φ angle near 90 degrees. Therefore this problem associated with extrusion of relatively hard materials, cannot be substitute by effort of achieving high total amount of deformation with small deformation increment by the multiple extrusion method through the channel with big value of Φ angle.

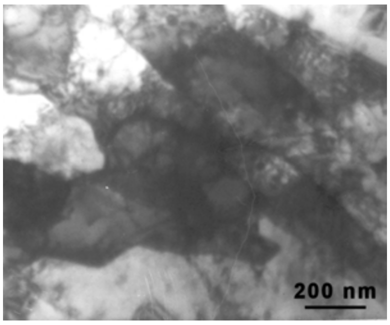


Fig. 7 Structure after the fourth passage
by the ECAP tool in AlCu4Mg2 alloy
(middle of the sample)

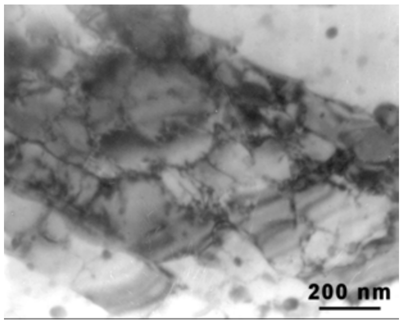


Fig. 8 Structure after the fourth passage
(1 mm from margins of the sample)

This microstructure control of extruded samples confirms, that the number of passages through highly affect on grain size. After every following passage through, by reason of very high total strain, it leads up to generation high number of shear planes. During this time, there happen to reaction between boundary and inner dislocations enabling deformation of grain (its refinement). It is fully certified premise increasing numbers of passages through on refinement of structure.

Achieved findings from the viewpoint of construction extruding tool

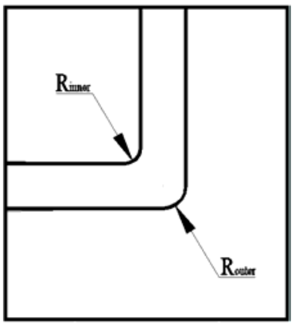


Fig. 9 Inner and outer radius of ECAP tool

There was optimized inner and outer fillet radius of passage of channel from vertical to horizontal position from the viewpoint of construction. It was made for AlCu4Mg2 alloy - $\mathbf{R_{inner} = (0,2 - 1)\ mm}$, $\mathbf{R_{outer} = (2,5 - 4)\ mm}$

From the viewpoint of friction, there is new knowledge. It is longitude shortening of horizontal part of channel and slight conical ness on exit – 1 degree. Longitude of horizontal part and value of friction affects on slope of sample (tablet) after extrusion and deny growing of possible displacements.

4 Conclusion

Belong to performed analysis of AlCu4Mg2 alloy structure on TEM, there was definitely certified suitability of designed construction of extrusion tool for ECAP technology. At the same time, there was verified the methodology of extrusion technology from the viewpoint of necessary number of passage through and suitable channel angle with appropriate inner and outer fillet radius for achieving nanosized grain. There was achieved annihilation of dislocation at grain boundaries by the high degree deformation and creation of high number of shear planes. The result of performed analysis is achieving large refinement of grain (from 100 to 200 nm) in whole bulk of sample, at initial middle of grain - 150 μm. Final results give us very good premise for next experimental works in this area.

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