Preparing Nanostructural Materials by ECAP Method of Severe Plastic Deformation

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ABSTRACT: *The technology, ECAP – Equal Channel Angular Pressing, belongs to technologies of accelerated development and represents top items of R&D agenda in the world. The development of nano-structure materials and nanotechnologies in general represents a principal domain of EU RP 6. The R&D network development and commencement of concrete project implementation should start in 2003 and onwards.*

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, Langdon, 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. The objective has been to bring down the overall vehicle weight, which is of immediate consequence, taking into account production costs, petrol consumption, CO2 and Nox emissions that are all lower, which is in favour of environmental sustainability for production industries.

Aluminium alloys of super fine granularity structure are basic intermediate products realised by ECAP technologies. The state of super fine granularity facilitates forming of material in the so-called 'superplastic state'. The achievement of the desired structure depends primarily on the tool geometry, number of passages through the die, magnitude and speed of deformation, process temperature, and lubrication mode.

1 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 /1, 2/. 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.

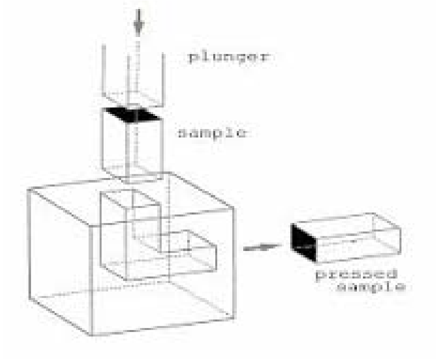

Figure 1- Schematic illustration of ECAP technology

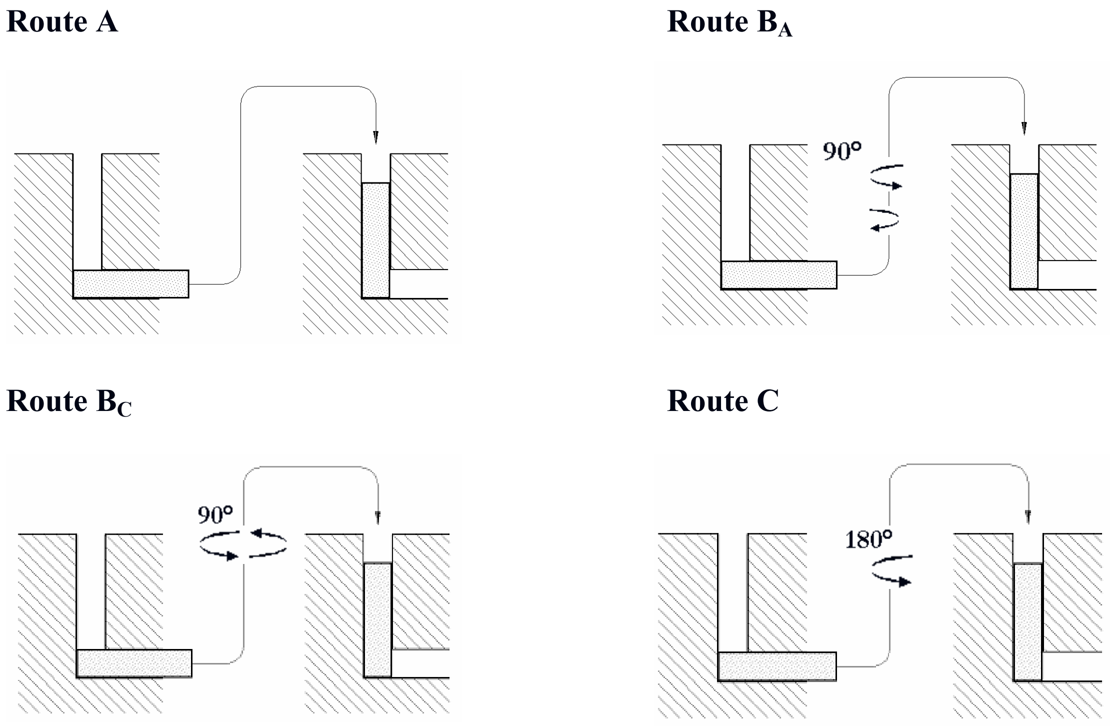

Figure2 - The four processing routes in ECAP

To understand the effect of these different processing routes, it is instructive to examine the internal shearing patterns as illustrated in Fig. 4 where the planes labelled 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. 4 correspondent to the planes illustrated in Fig. 1 on the as pressed sample. Inspection of Fig. 4 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 Ba and Bc there are four distinct shearing planes intersecting at angles of 120° [3,4].

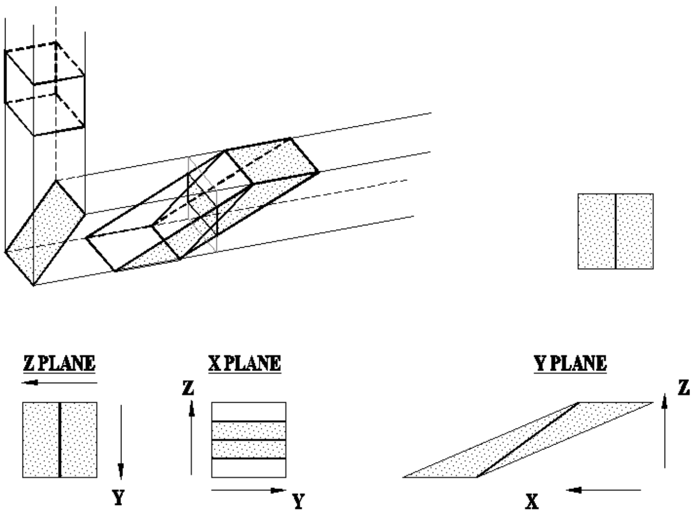

Fig. 3. Shearing associated by a single passage through the die

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.

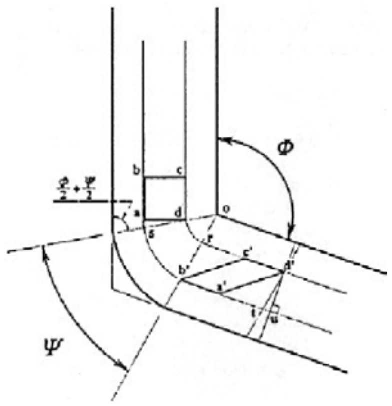

Figure 4. Principle of equal – channel angular pressing where Φ is the angle of intersection of the two channels and Ψ is the angle subtended by the arc of curvature at the point of intersection.

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 = adcot (Φ/2+Ψ/2), ab′= dc′= (as + osΨ) = rc′+ odΨ and (os – od) = adcosec (Φ/2+Ψ/2), so that a′u = 2adcot (Φ/2+Ψ/2) + adΨcosec(Φ/2+Ψ/2), Therefore, the shear strain for this intermediate condition is given by

$$\gamma = 2\cot\left(\frac{\phi}{2}+\frac{\Psi}{2}\right)+\Psi\,\mathrm{cosec}\left(\frac{\phi}{2}+\frac{\Psi}{2}\right) \qquad (1)$$

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

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

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].

Plastic deformation realised with use of the ECAP technology represents a complex process, which depends on great number of factors, such as homologous temperature of deformation Th, (Th = Ttav/Tt), grain size dz, strain rate $\dot{\varepsilon}$, magnitude of octahedral stress at deformation σ_8, particularly in relation to the magnitude of the modulus of elasticity E (σ_8/E represents homologous stress), but also on density of structural surfaces (particularly dislocations; vacancies), on purity, etc. ECAP cold deformation is significantly dependent on the latter factors.

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 [4]:

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

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. Our target was to obtain after extrusion the semi-products with a fine-grain structure. Such a structure on one hand increases strength properties and plasticity, and on the other hand it is possible to use it at selected cases for subsequent deformations under conditions of „super-plastic state“. Obtaining of the necessary structure in extruded samples depends primarily on the tool's geometry, number of passes through the die, obtained magnitude of deformation, temperature, etc.

3 EXPERIMENTAL VERIFICATION

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 No. 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 samples after individual stages of extrusion are shown in the Fig 5.

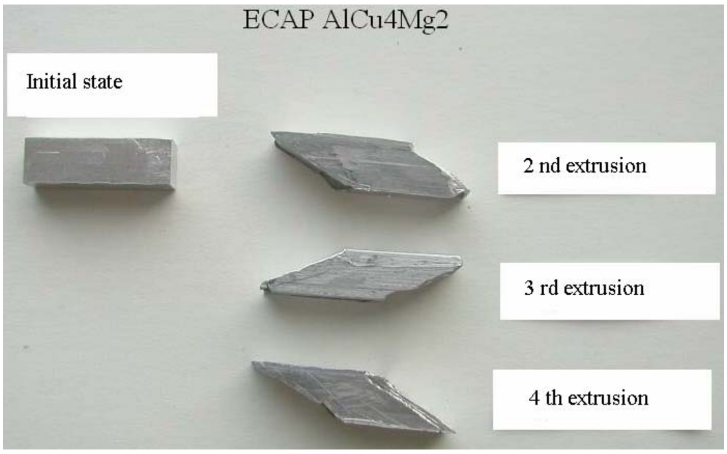

Figure 5. Initial state and states after extrusion

Deformation forces were measured at extrusion. There was also the average value of true deformation resistance and strain rate calculated. Structure analysis was made with use of the transmission electron microscope. The initial structure contained usual inter-metallic phases, corresponding to the given composition of the alloy, which precipitated predominantly at the grain boundaries.

The average grain size in cross direction was determined by quantitative metallographic methods. It varied around 150 μm [7, 8]. The basic mechanical properties were determined by tensile tests: strength Rm = 220 MPa, ductility A_5 = 15 % and hardness HB (2,5/62/30) ~ 70.

Examples of structures analysed by TEM after first and fourth passage through the ECAP die in different places of sample.

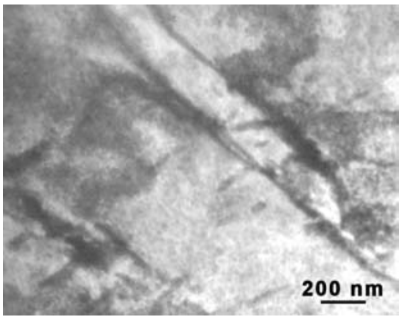

Figure 6. Pattern structure of ALCu4Mg2 alloy after the first passage through of ECAP die (middle of specimen)

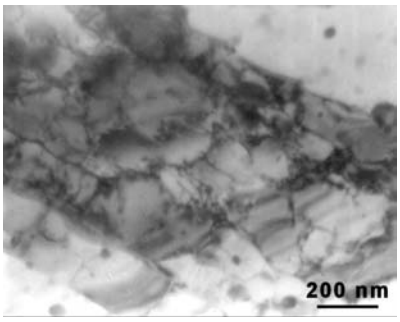

Figure 7. Pattern structure of ALCu4Mg2 alloy after the 4th passage trough of ECAP die (middle of specimen)

4 CONCLUSION

Structural analysis of AlCu4Mg2 alloy made by TEM has demonstrated a perfect suitability of the ECAP die design. It has been also demonstrated that the extrusion technology is suitable for attaining of grain nano-structure in the material investigated concerning the number of extrusion cycles needed and the appropriate canal angle with corresponding internal and external bend radii. High deformation degrees and providing for a great number of shearing planes, grain boundary dislocations were eliminated. The process results in a very fine grain structure (100-200 nm) throughout the sample overall volume, at which the starting average grain size was 150 nm. The results attained make for success of further investigative efforts in the area.

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