

Review paper

ECAP – New consolidation method for production of aluminium matrix composites with ceramic reinforcement[#]

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Abstract

Aluminium based metal matrix composites are rapidly developing group of materials due to their unique combination of properties that include low weight, elevated strength, improved wear and corrosion resistance and relatively good ductility. This combination of properties is a result of mixing two groups of materials with rather different properties with aluminium as ductile matrix and different oxides and carbides added as reinforcement. Al_2O_3 , SiC and ZrO_2 are the most popular choices of reinforcement material. One of the most common methods for producing this type of metal matrix composites is powder metallurgy since it has many variations and also is relatively low-cost method. Many different techniques of compacting aluminium and ceramic powders have been previously investigated. Among those techniques equal channel angular pressing (ECAP) stands out due to its beneficial influence on the main problem that arises during powder compaction and that is a non-uniform distribution of reinforcement particles. This paper gives an overview on ECAP method principles, advantages and produced powder composite properties.

Keywords: powders, extrusion, pressing, aluminium/ceramic composites

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I. Introduction

Aluminium matrix composites are a fast developing group of metal matrix composites due to its wide range of potential applications in different branches of industry but mostly automotive and aerospace. Improving properties of aluminium by introducing different oxides and carbides is somewhat well accepted idea due to possibility of generating aluminium based material with improved mechanical and Young's modulus, while keeping beneficial properties of aluminium itself like high ductility and toughness. Previously investigated aluminium based metal matrix composites (MMCs) were reinforced with different types of ceramic particles with Al_2O_3 , SiC, ZrO_2 , and SiO_2 being most common ones [1]. It has been shown that using ceramic particles as reinforcements rather than some continuous reinforcement type provides materials with more isotropic properties and relatively cheaper and simpler production process with particle size being key parameter for obtained

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Figure 1. ECAP die geometry and general principle



Figure 2. Two part ECAP die

properties of the composite material [2]. Reducing particle size significantly benefits overall mechanical properties so current trends in MMC production mostly include use of nanoparticles creating thus different types on nanocomposites [2]. Like with other types of composite materials, properties of MMCs depend strongly on production methods and parameters. Major problem of conventional powder metallurgy methods for production of metal matrix composites is tendency of smaller particles specially nanoparticles to form clusters during pressing which overall contribute to increased material porosity and poor mechanical properties. Also direct extrusion of Al based MMCs is limited in industrial production due to rather high pressing loads necessary for powder consolidation without very high temperatures [3]. Equal channel angular pressing has shown to be very efficient for powder consolidation at low temperatures under relatively low pressing forces.

II. Equal channel angular pressing

Severe plastic deformation, SPD, has been investigated for some time now. Initial field of interest was using SPD techniques as methods for refining grain size in different metals at room temperature by imposing large shear on the material thus improving their mechanical properties. Most commonly used SPD methods included high pressure torsion, groove rolling, accumulated roll bonding and equal channel angular pressing, ECAP, which proved to be very efficient technique for obtaining ultrafine grain structure.

During equal channel angular pressing material in form of a billet is pressed through a die with geometrically equal channels intersecting at the certain angle Φ and severely deformed. Since channels are of same shape and size, sample does not experience any transverse section change during processing. Figure 1 gives a general ECAP process setup. When this procedure is repeated for several times material is exposed to intense plastic strain. Die for ECAP is usually constructed of two parts so that easy extraction of billet after one pass is enabled, Fig. 2. Very important factor that will influence quality of final product and adequate pressure during ECAP is tight and safe joining of these two parts.

There are four basic routes for ECAP processing based on billet rotation between individual ECAP passes. Every of these routes are attributed to different slip system and strain value. During route A there is no rotation of the billet, while during route C the billet gets rotated for 180° between each pass. For routes B_A and B_C rotation of 90° in the same and opposite direction occurs between passes. [4]. Figure 3 shows described basic routes of ECAP.

ECAP being so popular went through many changes and alterations during time. Many of those modifications were aimed at reducing forces needed for pushing the billet through intersecting channels [4] and thus enabling production of larger samples while increasing materials' deformation.

ECAP process parameters include lubrication, temperature, pressing routes, speed, force, channel geometry and back pressure presence. Pressing temperature depends mostly on type of billet material. For materials of high ductility such as aluminium no heating of the die is necessary, whereas for pressing Ti alloys temperatures need to be elevated. Processing powders with ECAP at low temperatures enables production of bulk materials with grain size from nano to micron size scale. Desired type of microstructure will be key variable when deciding on optimal pressing route since single route involves specific slip systems. Channel cross section size is governed by maximum force since its reduction will reduce necessary pressing forces [5]. Materials processed by this technique will display properties governed by deformation behaviour during pressing through the die in which die geometry has very important role. Other important variables include general properties of pressed material such as strain hardening behaviour and previously mentioned process parameters [6].



Figure 3. Basic routes for ECAP pressing

IIL. Utilizing ECAP for production of Al MMCs

Later development of ECAP technique was directed to obtaining efficient method for powder metallurgy at relatively low temperatures. In last couple of years ECAP was also used as a method for production of rapidly solidified metallic powder alloys having amorphous structure and outstanding mechanical properties [6,7]. During powder consolidation raw powders have to be diffused in solid phase at temperatures below materials' melting point. The surface of powder particles is covered by oxide layer which acts as an obstacle during particle bonding. In order to consolidate powders that barrier needs to be broken so clean particle surface can interact with each other. ECAP as a method proved to be efficient in doing since it generates severe plastic deformation through high pressure which imposes high shear stress on powder mixture thus enabling particle



Figure 4. Pressed sample [12]

consolidation. This can be performed with rather low forces applied, making this potential production process for wide range of industrial applications [8]. First attempts of reinforcing ductile materials like aluminium and copper with ceramic particles have been made research for automotive industry purposes. This type of new materials would possess best of both materials: ductility of metal matrix with increased strength of ceramic reinforcement particles.

In recent years research in that area has been expanded on aluminium nanocomposites production as a method to substitute production of such composites by in situ formations of reinforcement nanoparticles in melt which were very complex and limited procedures. The main problem of using nanoscale ex situ reinforcement particles is a difficulty to obtain uniform distribution of such particles due to their tendency to form clusters [2]. Many analyses conducted by Mohseni, Balog, Athreya etc. of Al based MMCs consolidation characteristics and their obtained properties showed ECAP to be a good method for solving consolidation problems [7,9,10]. These investigations showed similar densification behaviour of powder composite materials and regular metallic powder materials. Composite powder materials were shown to have lower strength and density due to mentioned cluster formation and need for extra pressure in order to enable softer metal particles to fill voids between hard ceramic particles [11]. Applying back pressure in the outlet channel during pressing proved to have very positive effect on reducing parti-



Figure 5. SEM micrographs of Al-Al₂O₃ (15 vol.%) for different number of ECAP passes: a) 1st pass, b) 2nd pass, c) 3rd pass and d) 4th pass [14]

cle clustering and increasing density thus enabling more uniform structure of produced composites.

Procedure for obtaining Al MMCs starts with milling of powder mixture which is usually composed of atomized aluminium powder and reinforcement particles such as SiC, Al_2O_3 , AlN etc. of different particle sizes. Obtained loose powder mixture is then loaded in copper capsule where it can be heated for some time after which it goes to unheated ECAP die. Then pressing of the capsule through the die is conducted with adequate speed and main and back pressure is applied depending on the material and size of the pressed sample. After several passes of ECAP, pressed samples (Fig. 4) are removed from the capsule and prepared for mechanical and microstructural characterization [12].

IV. Microstructure and mechanical properties of ECAP AI MMCs

4.1 Microstructure

Many studies investigated properties of composites produced with this technique with special attention to obtained materials' density, hardness, Young's modulus etc. During ECAP numbers of significant changes of material microstructure occur such as intense grain and second phase particle refinement, formation of structure characterized by equiaxed grains and large angle grain boundaries. The result of a single ECAP pass is a directed shear texture with visible sub grain bands. With following passes matrix structure gets homogenized and low angle grain boundaries after first pass transform to higher angle boundaries. In addition, simultaneous reduction of grain size and additional accumulation of dislocations occur, which finally results in elongated subgrain breakup and formation of equiaxed structure. Mentioned dislocations build-up has similar effect on present reinforcement particle conglomerates which are broken and uniformly distributed in the light metal matrix while porosity of samples is reduced as shown on Fig. 5.

Described structure is characterized by high level of energy stored and is prone to recrystallization, i.e. grain coarsening under certain conditions. Introduction of ceramic particles showed to have a positive effect on reducing grain recovery tendency for materials processed by ECAP.

4.2 Mechanical properties

The influence of these microstructural changes on mechanical properties of matrix material is significant and can be described with Hall-Petch relationship between grain size and strength. Hall-Petch strengthening is a phenomenon occurring as a result of grain boundaries and dislocations presenting obstacles for dislocation movement which leads to strengthening effect.

The reduction of grain size, which takes place during ECAP, creates larger overall grain boundary area hence contributing to Hall-Petch effect described with following expression:

$$\sigma_v = \sigma_o + (k_v/d^{1/2}) \tag{1}$$

with σ_y being yield strength, σ_o being yield strength of single crystal, k_y being strengthening coefficient, *d* grain diameter.

From this equation it can be seen that strength of material is inversely proportional to grain diameter square root with σ_o and k_y being materials constants. Previous studies have shown that there is a critical grain size under which no additional strengthening occurs [6,10,12].

Besides Hall-Patch phenomena connected to ECAP processing nature there is another strengthening effect specific for metal matrix composites. The influence of ceramic particles on composite properties can be described with the following expressions [13]:

$$\Delta R_{p,C} = \Delta \sigma_a + \Delta \sigma_{KG} + \Delta \sigma_{SKG} + \Delta \sigma_{KF}$$
(2)

with $\Delta R_{p,C}$ being the increase in tensile strength of aluminium materials generated by addition of reinforcement particles.

 $\Delta \sigma_{\alpha}$ presents yield strength increment due to induced dislocations and can be calculated as:

$$\Delta \sigma_{\alpha} = \alpha \cdot G \cdot b \cdot \rho^{\frac{1}{2}} \tag{3}$$

with α being constant, G shear modulus, b Burger's vector and ρ dislocation density.







Figure 7. Tensile test curves for different Al-SiC composite compositions produced by two different methods, FE and ECAP [15]

Dislocation density ρ can be calculated with following expression:

$$\rho = 12\Delta T \left(\Delta C \cdot \Phi_{p} / b \cdot d \right) \tag{4}$$

where ΔT is temperature difference, ΔC is thermal expansion coefficient difference between matrix and particle, Φ_p is volume content of particle and d is the particle size.

The influence of the grain size on yield strength $\Delta \sigma_{_{KG}}$ analogue to the one described by Hall-Patch equation (1) can be expressed as:

$$\Delta \sigma_{\kappa G} = k_{\gamma I} \cdot D^{-1/2} \tag{5}$$

with

$$D = d \left[(1 - \Phi_p) / \Phi_p \right]^{1/3} \tag{6}$$

where k_{y_l} is a constant, D is a generated grain size.

 $\Delta \sigma_{_{SKG}}$ presents subgrain size changes contribution to the yield strength and can be calculated with following expressions:

$$\Delta \sigma_{SKG} = k_{Y2} \cdot D_s^{-1/2} \tag{7}$$

$$D_{\rm s} = d \left(\pi \cdot d^2 / 6 \Phi_p \right)^{\frac{1}{2}} \tag{8}$$

where $k_{_{Y2}}$ is a constant, D_s is resulting subgrain size. Strain hardening contributing factor $\Delta\sigma_{_{KF}}$ is given by:

$$\Delta \sigma_{\rm \tiny KF} = K \cdot G \cdot \Phi_{\rm \tiny n} \, (2b/d)^{1/2} \cdot \varepsilon^{1/2} \tag{9}$$

with *K* being constant, *G* shear modulus and ε elongation [13].

In many studies comparison between properties of conventionally pressed samples to ones produced by ECAP was conducted and usually showed higher values of properties such as hardness, Young's modulus and tensile strength for ECAP-ed samples. Such comparison of two different processing methods and obtained hardness and tensile test values of Al- SiC composites with different SiC particle content is given on Figs. 6 and 7 [15].

V. Conclusions

Many advantages and aspects of using equal channel angular pressing as a powder consolidation method for production of aluminium matrix composites reinforced with ceramic particles have been presented in this paper. The process has a wide range of parameters such as particle size, reinforcement material type and content, pressing routes, speed and die design which all can be specifically adjusted in order to engineer desirable properties of final product such as hardness, strength, wear resistance etc. Besides variability, ECAP has shown beneficial influence on consolidation of nano-sized particles eliminating problems concerning fine particle conglomerate formation which opens space for the development of brand new approach to consolidating nano-structured materials. In order to make it widespread industrial method for fabrication MMCs, future efforts should be directed to automation of the process mostly concerning die geometry i.e. rotation of the sample in between passes for different routes making it a more continuous process.

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