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GRAIN REFINEMENT THROUGH SEVERE PLASTIC DEFORMATION (SPD) PROCESSING

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Abstract. There is considerable current interest in processing metallic samples through procedures involving the imposition of severe plastic deformation (SPD). These procedures lead to very significant grain refinement to the submicrometer or even the nanometer level, resulting in advanced physical properties. Among various SPD processes, Equal Channel Angular Pressing, High pressure Torsion and Accumulated Roll Bonding have been widely used for many metals and alloys. In the present work, we present an overview of the most used methods of SPD for grain refinement and the production of bulk nanostructured materials with enhancement in their mechanical and functional properties. In order to examine the potential for using ECAP to refine the grain size and improve the mechanical properties, two commercial 5754 Al alloy and AA 3004 , were selected for study. Processing by ECAP gives a reduction in the grain size and an increase in the microhardness.

1. INTRODUCTION

Although the mechanical and physical properties of all crystalline materials are determined by several factors, the average grain size of the material generally plays a very significant and often a dominant role. Thus, the strength of all polycrystalline materials is related to the grain size, d, through the Hall-Petch equation which states that the yield stress σ_y, is given by

$$\sigma_y = \sigma_0 + k\, d^{-1/2} \tag{1}$$

where σ_0 is termed the friction stress and k is a constant of yielding. It follows from Eq. (1) that the strength increases with a reduction in the grain size and this has led to an ever-increasing interest in fabricating materials with extremely small grain sizes.

Nanostructured materials, in which the structural features (e.g., grains and/or domains separated by low-angle grain boundaries) are smaller than 100 nm in at least one dimension, have attracted worldwide research interest for more than a decade because of their unique properties. For example, the combination of high strength with high ductility has been reported

for some nanostructured metals and alloys: this is a rare, if not impossible, combination of mechanical properties for coarse-grained metals and alloys [1,2]. Among the many techniques available for producing nanostructured materials, severe plastic deformation (SPD) is the most popular and most rapidly developing one. SPD processing refers to various experimental procedures of metal forming that may be used to impose very high strains on materials leading to exceptional grain refinement. The unique feature of SPD processing is that high strain is impossed at relatively low temperatures (usually less than 0.4 T_m) without any significant change in the overall dimensions of the workpiece. Another feature is that the shape is retained due to the use of special tool geometries, which prevent free flow of the material and thereby produce a significant hydrostatic pressure. The presence of this hydrostatic pressure is essential for achieving high strains and introducing high densities of lattice defects that are necessary for exceptional grain refinement. SPD-produced nanomaterials are fully dense and their large geometric dimensions make them attractive for efficient practical applications. Fabrication of bulk nanostructured materials using severe plastic deformation is becoming one of the most actively developing areas in the field of nanomaterials, and SPD materials are viewed as structural and functional materials of the next generation of metals and alloys. Today, SPD techniques are emerging from the domain of laboratory-sale research into commercial production of various ultrafine-grained (UFG) materials [3].

The aim of this paper is to present an overview of the most used methods of severe plastic deformation for the production of bulk nanostructured materials with very significant enhancement in their mechanical and functional properties. Also, in order to examine the potential for using ECAP to refine the grain size and improve the mechanical properties, two commercial Al alloys, Al-5754 and AA 3004 were selected for study.

2. DIFFERENT SPD TECHNIQUES

Grain refinement is easily achieved by SPD through the introduction of large plastic deformations which induce the generation of severe crystal lattice rotations and simultaneously an extremely high number of dislocations rearranging into new low and high-angle grain boundaries [4-7] and deformation twins and twinned boundaries. This phenomenon known as the process of “crystal fragmentation” has created a world-wide interest and many investigations are now underway to determine the properties of materials processed by SPD [8-12]. There are several different types of SPD processes, but the three most common ones are equal-channel-angular Pressing (ECAP), high-pressure torsion (HPT) and accumulative roll-bonding (ARB). These three procedures are fundamentally different, but they each produce exceptional grain refinement to at least the submicrometer level.

2.1. Accumulative Roll-Bonding (ARB)

A technique which increases the available strain is **accumulative roll bonding(ARB**) (Fig. 1), in which the material is rolled, stacked and re-rolled, thus maintaining the sample thickness. Therefore, the achieved strain is unlimited in this process because repetition times are

endless in principle and have achieved ultra fine grains with less than 1μm. This technique has been applied to aluminium alloys [13] and steels [14] with total strains of up to 8, resulting in sub-micron grain sizes. Critical factors in successful accumulated roll bonding are surface preparation and cleaning, the deformation temperature, and the amount of strain. Depending upon the crystal structure, the microstructures have GS within the range of ~ 70-500 nm.

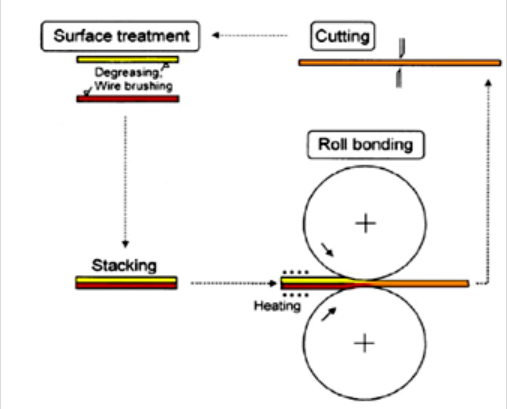

Fig. 1: Schematic showing the accumulative roll bonding process.

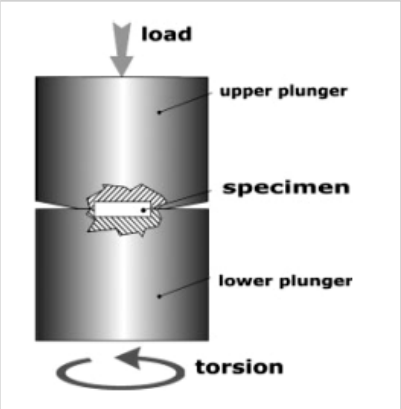

Fig. 2: Schematic of HPT set-up

2.2. High Pressure Torsion (HPT)

The HPT method is shown schematically in Fig.2. Disc sample is torsionally deformed under high pressure of several GPa, many times between plungers, on which an outer pressure is applied. High friction forces between the rough dies and the ingot ensure deformation by *shear* during rotation of the plunger. Provided that the number of rotations is high enough, an almost homogenous nanostructure can be achieved. The main advantages of the HPT technique are that it gives high quality UFG materials with grain sizes < 100 nm and with this method can be processed brittle materials as intermetallics and semiconductors. But the main disadvantage is that the specimen dimensions are fairly small, with maximum discs diameter of 10-20 mm and up to 1mm in thickness; for this reason this method has limited industrial use [10].

2.3. Equal Channel Angular Pressing (ECAP)

Although several different SPD processing procedures are now available, the most promising technique appears to be equal-channel angular pressing (ECAP) where a bulk sample is pressed through a die constrained within a channel which is bent through an abrupt angle (Fig.3) [10]. Advantages of processing by ECAP include the retention of a constant cross-sectional area through the imposition of high strains and the ability to process samples without the introduction of either contamination or residual porosity. The strain imposed on the sample in each passage through the die is dependent primarily upon the angle, Φ, between the two parts of the channel (90◦ in Fig. 3) and also to a minor extent upon the angle of curvature, Ψ, representing the outer arc of curvature where the two channels intersect (0◦ in Fig. 3). The

amount of equivalent strain depends upon the two angles, φ and Ψ, and given through a relation, developed analytically, that depends on the geometry of the process. The ECAP experiments conducted to date, have generally used dies having φ =90◦ and Ψ =0◦. This corresponds to an equivalent strain of 1.1 in a single pass [10].

Furthermore, since the cross-sectional area remains constant, the samples may be rotated between consecutive passes through the ECAP die in order to activate different slip systems [5]. At the present time, processing by ECAP is conducted using either route A where the sample is not rotated between consecutive passes, route B_A where it is rotated by 90° in alternate directions between each pass, route B_C where it is rotated by 90° in the same direction between passes or route C where it is rotated by 180° between passes (Fig.5). The evidence available to date shows that, when using a die where the channel is bent through an abrupt internal angle of 90°, route B_C is the optimum processing route for achieving a homogeneous microstructure containing a large fraction of boundaries having high angles of misorientation [6].

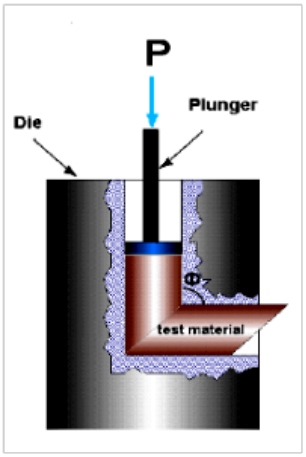

Fig. 3: Schematic view of ECAP die.

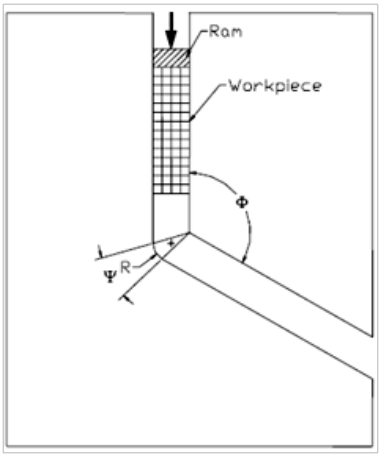

Fig. 4: A section through the ECAP die.

The application of this procedure is currently under investigation for many materials ranging from Al, Cu, Mg and Ni alloys to eutectic and eutectoid alloys and intermetallics.

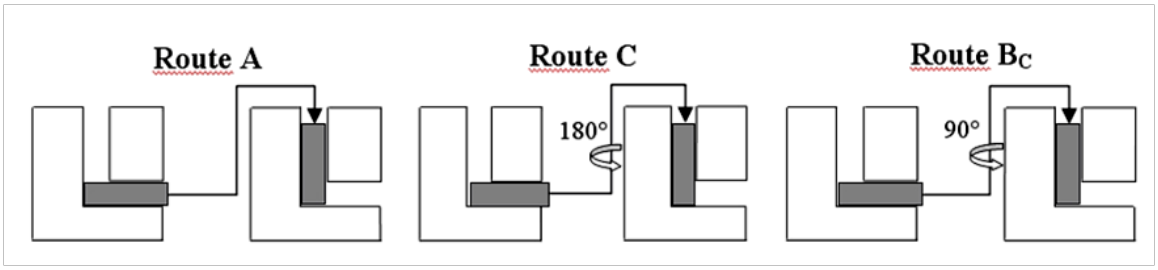

Fig. 5: Scheme of the processing routes in ECAP.

3. IMPROVEMENT OF MECHANICAL PROPERTIES OF COMMERCIAL AL ALLOYS PROCESSED BY ECAP

3.1 Experimental procedure

The experiments were conducted on two light-weight commercial aluminum Al- 5754 alloy having the following composition in wt%: 2.4-2.6% Mg, 0.1-0.6% Mn, 0.4% Cr, 0.4% Fe, 0.4% Si, 0.2% Zn with the balance as Al, and on Al -3004 alloy with a composition, in wt.%, of 1.1% Mg, 1,26 % Mn, 0.6% Fe, 0.4% Cu, and 0.4% Si. Observations by optical microscopy revealed a grain size of ~70 μm in the as received condition, for the Al-5754 alloy. The samples in the forms of cylinders with lengths of ~ 10 cm, were subjected to ECAP up to a total of 7 passes, equivalent to an imposed strain of ~ 7 for the Al-5754 alloy and up to 4 passes for AA 3004 . The pressings were performed at room temperature, using the route $B_{C.}$

Small pieces were cut from the as-pressed cylinders. Each of these pieces was polished and then used to measure the Vickers microhardness Hv, using a load of 100 g, applied for 15 s.

3.2 Experimental results and discussion

AA 5754: Microstructural examinations of samples pressed through 2 to 6 passes revealed an array of reasonably equiaxed grains having average sizes of < 1 μm. An example of the as pressed microstructure is shown in Fig.5, after 4 passes. Measurements indicated average grain sizes of ~ 0.3-0.4 μm, in the as-pressed condition, demonstrating that ECAP is an effective procedure for attaining an ultrafine grain size.

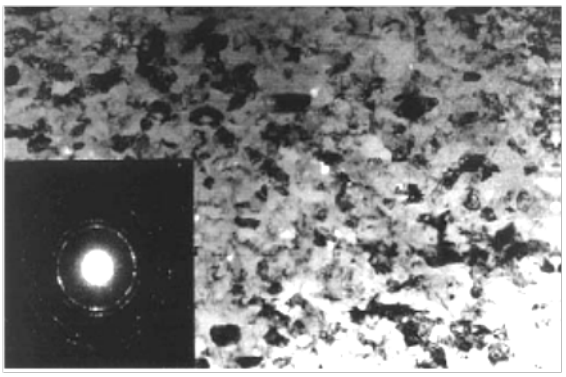

Fig. 6: Microstructure observed by transmission electron microscopy after ECAP through 4 passes.

Fig.7 shows the variation of the microhardness with the number of passes, where measurements were taken on 2 orthogonal planes and the first point (zero passes) refers to the unpressed alloy (Al-5754) .

Two conclusions may be reached from this plot. First, The hardness is essentially independent of the plane of sectioning. Second, the value of Hv increases abruptly after a single pass, but thereafter increases slowly with additional passes. Similar results are attained also for the AA 3004.

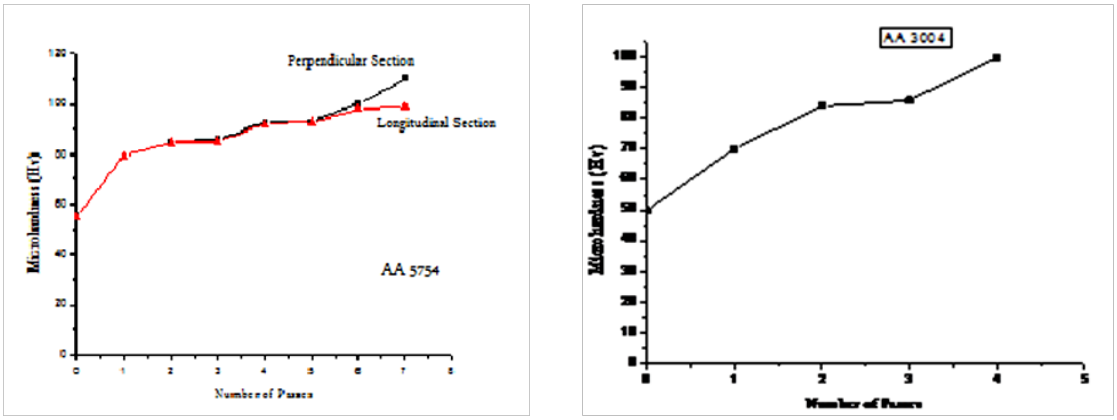

Fig. 7: Microhardness Hv versus number of passes in ECAP

4. CONCLUSIONS

This investigation demonstrates that ECAP was an effective tool for achieving a very substantial reduction in the grain size of the commercial 5754 Al alloy. In these experiments the initial GS of ~ 70 μm in the as received alloy, was reduced to ~ 0.3 - 0.4 μm by ECAP through up to 7 passes. The reduction in grain size gave an increase in the microhardness of the alloy. There was an immediate increase in the microhardness at a strain ~ 1 with minor additional increases with subsequent straining. Similar results were observed for the AA 3004.

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