Equal channel angular extrusion (ECAE) of intermetallics

Anumalasetty Venkata Nagasekhar

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OF INTERMETALLICS

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Equal Channel Angular Extrusion (ECAE) of Intermetallics

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School of Materials Science and Engineering

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ABSTRACT

Various techniques of severe plastic deformation (SPD) are used with the purpose of fabricating bulk ultrafine grain materials and for the compaction of advanced powders. Equal channel angular extrusion/pressing (ECAE/ECAP), which is one of the SPD techniques, has unique ability to generate huge strains, high strain rates, absence of shape change, and minimal load requirements. ECAE has thus been used for enhancing the microstructure and mechanical properties of tubular materials as well as for compaction of powders through the powder in tube (PIT) technique. Compared to the conventional metal forming techniques like extrusion, rolling and drawing, this process has many advantages: unidirectional deformations can be produced under relatively low pressures and loads in massive products, and it also promotes the formation of equi-axed grains with high-angle grain boundaries.

The properties of the ECAE processed materials depend solely on the uniformity of strain distribution (strain homogeneity) achieved in the deformed materials. The strain homogeneity of the deformed materials is sensitive to tool angles, material properties, frictional conditions, speed and temperature of the process. Finite element simulations of ECAE process were carried out with Abaqus/Explicit to obtain the optimal strain homogeneity of the deformed materials, to study the deformation behavior of the sample in the die, and evaluate the dead zone/corner gap formation. The simulations were carried out for various tool angles under ideal (elastic-perfectly plastic material with frictionless contact between die and sample) and realistic (elastic-strain hardening material with frictional contact between the die and sample) conditions. The results of both conditions showed that a channel angle of 90° and outer corner angle of 10° are optimal for achieving strain homogeneity in the deformed samples without involving any detrimental effects. Multipass ECAE simulations were also carried out with these tool angles for Route A (no rotation of the sample in between the passes) & Route C (180° rotation of the sample in between the passes), to study the influence of processing route on strain homogeneity. The degree of strain homogeneity achieved is higher in Route A compared to Route C.

Validation of FEA and experimental results of bulk copper were carried out by conducting single pass ECAE experiments and simulations. FEA strain

homogeneity was found to have good conformity with hardness measurements, along with good correlation between the experimental and FEA peak load calculations.

Single pass ECAE processing of PITs was carried out for two different powders by using three different tube materials. Studies were conducted for the first time to understand the process mechanism of ECAE of PIT. For hard and brittle intermetallic magnesium boride (MgB_2) powder, the process was found to be primarily due to compaction and shear sliding of the powder with localized deformation of the tube. The occurrence of localized deformation during ECAE was discussed in detail. Compaction efficiency was found to depend not only on the material of the tube but also on the homogeneity of stress and strain in the composite PIT. Various frictional stresses and localized-deformation were found to be the reasons for stress-strain inhomogeneity. In the case of copper powder, even though localized-deformation occurred, higher inter-particle friction and low yield strength of the powder allowed densification nearer to theoretical density of the powders.

Multipass ECAE processing of MgB_2 PITs were carried out by using LCS and Fe tubes to study the influence of processing route, and number of passes on powder compaction efficiency. MgB_2 PITs with LCS tube, processed via different processing routes of ECAE up to four passes, showed the density enhancement of powder compacts in the following descending order of processing routes: Route A, Route B_A, Route C, and Route B_C. MgB_2 PITs with Fe tube, processed via Route C up to six ECAE passes, showed the density enhancement with the increase in pass number.

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NOMENCLATURE

T_m	Melting temperature
SPD	Severe plastic deformation
IGC	Inert gas condensation
HPT	High pressure torsion straining
ECAE	Equal channel angular extrusion
ECAD	Equal channel angular drawing
ARB	Accumulative roll bonding
CGP	Constrained groove pressing
ϕ	Channel angle/Die angle
ψ	Outer corner angle
ε	Equivalent plastic strain/Effective strain
CIP	Cold isostatic pressing
HIP	Hot isostatic pressing
P	Punch pressure
σ_o	Yield stress
CAD	Channel angular deformation
T_C	Critical temperature
J_C	Critical current density
H_C	Critical Field
PIT	Powder in tube
DMCUP	Dense material cooled under pressure
ε^{Pl}	Plastic strain
σ_f	Flow stress
σ	Normal stress
τ	Shear stress
μ	Coefficient of friction
ρ	Density of the material
γ	Shear strain
E	Young's modulus
ν	Poissons ratio
n	strain hardening exponent

K	strength coefficient
PEEQ	Equivalent plastic strain/effective strain

CHAPTER 1

INTRODUCTION

1.1 SEVERE PLASTIC DEFORMATION (SPD)

Microstructure with ultrafine (nanometer and sub-micron) grains is of interest to both scientists and engineers due to their ability to improve physical and mechanical properties. Severe plastic deformation (SPD) is one of the techniques used to produce microstructure with ultrafine grains in metals and alloys. SPD employs large plastic deformations under high pressures at relatively low temperatures (usually less than $0.4T_m$). The materials processed with SPD techniques have several advantages compared to the materials processed by other nano-material processing techniques such as inert gas condensation (IGC), rapid quenching and ball milling. One of the advantages it offers is the possibility to fabricate bulk metallic nano-materials free of residual porosity and impurities. Another major advantage is its capability to use monolithic ingots and powders as the starting materials (for the formation of nano structures in various metallic materials like, metals, alloys, and intermetallics). The SPD technique also facilitates the consolidation of ultrafine powders to form fully dense ($> 98\%$) bulk materials. Conventional deformation techniques, such as drawing and rolling can result in significant refinement of microstructure at low temperatures. But they usually result in the formation of cellular structure with low-angle grain boundaries. The SPD technique can promote the formation of ultrafine-grained structures with high-angle grain boundaries [1].

1.2 EQUAL CHANNEL ANGULAR EXTRUSION (ECAE) AS SPD

The SPD techniques like high pressure torsion straining [HPT], equal channel angular extrusion [ECAE] (or) equal channel angular pressing [ECAP], multi axis forging, cyclic extrusion and compression, accumulative roll bonding [ARB], equal channel angular drawing [ECAD] and constrained groove pressing [CGP] were used by different researchers based on the specific application [2 – 6]. Among the recently developed SPD techniques, a lot of attention has been devoted to high pressure torsion straining and equal channel angular extrusion, due to the success in achieving ultrafine grains in bulk materials and consolidation of ultrafine powders

nearer to theoretical density. High pressure torsion straining has one main disadvantage compared to equal channel angular extrusion, i.e. the specimen dimensions are fairly small (maximum sample dimensions are 20 mm diameter and 1 mm thickness) [7]. In view of this the most promising SPD technique is thus equal channel angular extrusion /pressing.

1.3 PRINCIPLE OF ECAE

Equal channel angular extrusion or equal channel angular pressing (hereafter referred to as ECAE or ECAP) is a novel deformation process capable of imparting a large amount of plastic strain to bulk material by the application of uniform simple shear. Figure 1.1 shows the principle of ECAE [8, 9] with both sharp corner and round corner intersection of channels. The tool for ECAE consists of two channels of equal cross-section that intersect at angle of ' ϕ '. Typical values of ' ϕ ' are 90° to 150° . The channel intersection can be abrupt or contains a round transition zone. A well-lubricated billet of almost the same cross-section is placed into the first channel and a punch then extrudes it into the second channel. Under these conditions the billet moves inside the channels as a rigid body, and deformation is achieved by simple shear in a thin zone (AB) at the crossing plane of the channels. In this way the complete billet (except small end regions) is deformed in a uniform manner.

Figure 1.1 Schematic diagram showing the principle of Equal Channel Angular Extrusion with (a) sharp corner and (b) round corner intersection of channels. (A-B indicates the simple shear zone).

After the extrusion stroke, the punch retreats and the deformed billet is withdrawn from the second channel. The process can easily be repeated a number of times with the same tool.

1.4 ECAE FOR MATERIALS PROCESSING

The following salient points resulted from various studies [10-18] highlights the uniqueness of the ECAE process.

- A large equivalent deformation per pass and an extremely large total effective deformation after multiple passes can be imparted to the sample without any appreciable change of initial billet cross section.
- Uniform structure and properties are developed throughout the worked materials.
- A variety of different microstructures, substructures and textures can be developed by modifying the planes and direction of shear at each pass facilitated by different combinations of billet rotations about its own axis between extrusion passes.
- Due to the formation of fine equiaxed grains in materials subjected to ECAE, the strength is considerably increased while maintaining significant ductility.
- Equi-axed grains of sub-micron size can be obtained through ECAE processing, facilitating superplastic behavior in some alloys. Further, the temperature for superplastic deformation may be reduced and superplastic behavior obtained at comparatively higher strain rates if the grain size is reduced to still lower values.
- ECAE is an effective mechanism for elimination of volume defects such as pores, voids and cavities and can be used to deform and breakdown coarse as - cast structures.
- ECAE has been effectively used to consolidate the powders.
- ECAE is an effective method to produce thixoforming feed stock.
- ECAE processed material has shown considerable increase in fatigue life and fatigue limit under constant load testing.
- ECAE processed materials have proven better resistance to stress corrosion cracking.
- High coercive states can be achieved in ECAE processed materials.
- ECAE is a promising technique for improving the mechanical and microstructural properties of tubular materials with low extrusion pressures.

- ECAE can be done at cold, warm and hot working conditions with standard metal working presses.

ECAE can be applied to a wide variety of materials including alloys of Al, Mg, Cu, Ni, Ti, steels, super alloys, intermetallics, ceramics, powders, polymers, composites etc. As a result of the above advantages, ECAE may be considered as an important deformation technique providing a lot of scope for further research.

It has been shown by earlier research that ECAE results in grain refinement and enhancement of mechanical and physical properties. Most of the published research has dealt with fundamental issues in materials science like development of sub-grain structure, grain refinement, formation of shear bands, and development of texture in solid materials. Very little work related to processing of intermetallics and powders has been published.

1.5 MAGNESIUM BORIDE (MgB_2) POWDER AND ITS COMPACTION

MgB_2 is the recently discovered (in year 2001) high temperature intermetallic superconductor that becomes superconducting at 39 K, one of the highest transition temperatures (T_C) of the currently available metallic superconductors [19]. MgB_2 has some peculiar properties compared to perovskites (high temperature superconducting cuprates) like simple crystal structure, low material density, lower anisotropy, larger coherence lengths and high J_C values due to weak link free grain boundaries and lower cost. MgB_2 is a brittle intermetallic compound readily available in powder form, which starts to decompose at relatively low temperatures, about 1000°C. To overcome the problem with magnesium volatility, powder in tube (PIT) method was successfully applied for sintering of magnesium boride and fabrication of ironclad MgB_2 ribbons and tapes [20]. But PIT method or similar methods of sintering do not result in a fully dense material. At the same time there is a need to develop new methods to fabricate fully dense MgB_2 to enhance super conducting properties, and to increase environmental robustness, mechanical strength and fracture toughness. Porosity is also detrimental for achieving a high, mirror like quality of surface, specifically important for some applications like microwave devices and for processing methods like laser ablation and magnetron sputtering. Fundamental research on properties of magnesium boride also needs fully dense material. To achieve this high density in MgB_2 several authors [21 – 27]

used conventional and advanced powder metallurgy techniques like, sintering under high pressure, multi anvil pressing with high pressure and high temperature, hot isostatic pressing [HIP], HIP with DMCUP (dense material cooled under pressure) cycle, shock wave consolidation, magnetic pressing. However, with these techniques there are limitations on the sample size obtained and theoretical density that can be achieved. In addition, the energy requirements for these techniques are very high. In order to overcome these problems and to achieve full density in bulk MgB_2 , this project concentrates on the most promising SPD technique, ECAE, to compact the commercially available MgB_2 powder.

1.6 ADVANTAGES OF ECAE COMPACTION

Apart from the advantages of ECAE in materials processing mentioned in section 1.4, compaction of powders through ECAE has the following advantages [28-38]:

- ECAE processed powder compacts are able to preserve the initial mechanically alloyed fine scale microstructures
- Development of homogeneous and fine microstructures through different routes of ECAE compaction
- ECAE processed compacts showed enhanced hardness and strength compared to ECAE deformed ingots
- Able to remove the oxide layers of the powders
- ECAE compaction also causes bulk mechanical alloying
- Possible to scale the process to greater dimensions
- Design of materials is possible through ECAE processing of powders
- Low energy requirements compared to conventional & advanced powder metallurgy techniques, thus the cost involved would be correspondingly lower

1.7 OBJECTIVES OF THE WORK

Based on the above discussion the following objectives are defined for the work:

- Selection of optimal ECAE tool angles through finite element analysis (FEA)
- ECAE processing and compaction of intermetallic MgB_2 powder through powder in tubes (PITs)

- Understanding of the ECAE processing mechanisms of PITs

1.8 THESIS ORGANIZATION

Background and motivation for the current studies are discussed under introduction in Chapter 1. More details regarding the ECAE process, and its potential applications, latest developments regarding the ECAE process are detailed clearly under literature review in Chapter 2. Use of finite element analysis (FEA) for the current studies and its procedure, results and discussion of FEA are detailed in Chapter 3 under FEA of ECAE. Chapter 4 explains the materials and methods used for the current work. Chapter 5 focuses on the FEA and experimental results validation. Single pass ECAE processing of powder in tubes (PITs) and the processing mechanism of ECAE of PITs were discussed in Chapter 6. Results obtained from the multipass ECAE processing of powder in tubes (PITs) were presented in Chapter 7. Chapter 8 highlights the conclusions made from the study and the recommendations for future work.

CHAPTER 2

LITERATURE REVIEW

2.1 EQUAL CHANNEL ANGULAR EXTRUSION (ECAE) PROCESS

To optimise the effect of plastic deformation on the structure and properties of the material, the forming pressure must satisfy several requirements like uniformity of stress and strain states, accurate control of the intensity of deformation and stress state (hydrostatic pressure), control of the spatial development of the deformation and texture formation, and achieving high and ultra-high rates of deformation. These conditions have to be achieved with minimal damage and change in the cross-section of the sample. Conventional methods like rolling, forging and pressing do not meet most of these requirements.

The structure formation should be the most intensive if the macroscopic slip planes coincide with the crystallographic directions. In pure shear there is continuous rotation of crystallographic directions relative to slip lines, whereas in the case of simple shear, the orientation of the elements of the microstructure relative to the active system of slip lines is retained. The optimum scheme of plastic structure formation is simple shear, for which the micro-mechanism of deformation coincides with macro-mechanism of deformation at any instant in any finite volume of the material. Implementation of schemes of simple shear usually involves considerable difficulties, and special methods have to be used to achieve simple shear with a degree of approximation adequate for practical purposes. ECAE is one of the methods to achieve the plastic deformation by simple shear without varying the cross section of the sample [8].

The principle of ECAE is illustrated schematically in Fig. 1.1, where two channels of equal cross-section intersect at angle ' ϕ '. Extrusion may be conducted using a square configuration as in Fig. 1.1 (a) or round corner with curvature at the outer point of contact between the two channels as in Fig. 1.1 (b) [8, 9].

The equivalent plastic strain (ε) per pass is dependent upon the channel angle and the outer corner angle as given by

$$\varepsilon = [2 \cot (\phi/2 + \psi/2) + \psi \operatorname{cosec} (\phi/2 + \psi/2)] / \sqrt{3} \quad (2.1)$$

where, ε = Equivalent plastic strain, ϕ = Die angle or Channel angle, ψ = Angle subtended by the arc of curvature at the outer point of contact between the two channels.

For a sharp corner die $\psi = 0$

$$\text{then} \quad \varepsilon = (2/\sqrt{3}) \cot (\phi/2) \quad (2.2)$$

$$\text{and} \quad \gamma = 2 \cot \phi/2 \quad (2.3)$$

where, γ = Shear strain.

For a round corner die ' ψ ' lies in between $\psi = 0$ to $\psi = \pi - 2\phi$

$$\text{then} \quad \varepsilon = [2 \cot (\phi/2 + \psi/2) + \psi \operatorname{cosec} (\phi + \psi/2)] / \sqrt{3} \quad (2.4)$$

$$\text{and} \quad \gamma = [2 \cot (\phi/2 + \psi/2) + \psi \operatorname{cosec} (\phi + \psi/2)] \quad (2.5)$$

$$\text{The punch pressure,} \quad P = \varepsilon \cdot \sigma_o \quad (2.6)$$

where, ε = Equivalent strain imparted through one pass, σ_o = yield stress

Both analyses assume frictionless conditions due to well-lubricated specimens. The above equations are developed based on geometric considerations for ideal conditions (perfectly plastic material, and frictionless contact between the die and sample) of the ECAE [10]. The above analysis does not consider the realistic case (strain hardening and frictional contact between the die and sample). In addition, development of governing equations for ECAE under realistic conditions is a very complicated process. Hence, to find the strain distributions, pressure requirements under realistic conditions, finite element modeling was chosen in current study.

The mechanics of extruding an elastic-plastic material through two inter connecting ducts with equal transverse cross-section was examined, in which deformation in simple shear was implemented. Distinctive features of the process of formation of microstructures were noted when simple shear was applied as well as the production advantages of the corresponding deformation patterns. The possibility of achieving

abnormally high strength and ductility in metals by strain hardening through simple shear was established [8, 39, 40].

2.1.1 Stress and Strain Analysis of ECAE Process

The stresses and strains developed during ECAE deformation of bulk material was analyzed under frictionless and frictional contact between the die and processed materials by using slip line theory [8, 10, 41, 42], and upper bound technique [9]. Strain hardening of material is neglected in all these studies [8-10, 41, 42]. The stresses and strains developed during channel angular deformation (CAD) were analyzed by taking into account the friction between dies and material. From the analysis the pressure for ECAP has been obtained since the equal channel angular pressing is a special form of CAD [43]. The pressures needed for non-friction equal channel angular pressing (ECAP) of perfectly plastic material [10, 44] and strain hardening materials are analyzed by analytical approximations (upper-bound technique) and numerical (finite element) method [44].

2.1.2 ECAE Process Parameters

Experimental studies on aluminium alloys have identified four processing parameters that influence the microstructure of the alloy that undergoes the ECA Pressing.

- Angle between channels
- Rotation of sample between pressings
- The strain rate of the ECAE Process
- The temperature of the ECAE Process

2.1.2.1 Die Angle

In ECAE, the material is deformed successively by simple shear at the crossing plane of the two channels (the shear plane). The orientation of the shear plane with respect to the longitudinal axis of the extruded specimen depends on the intersecting angle between the two channels. Experiments were conducted to investigate the influence of the channel angle ' ϕ ' on the development of an ultrafine grain structure in pure Al. Tests were performed using dies having channel angles from 90 to 157.5°. It has been experimentally shown that even though a large plastic strain can be achieved in a sample by passing several times through a die having large value

of ϕ with ease, an array of ultra fine grains cannot be produced. But an ultrafine-grained structure, with high angle grain boundaries, can be achieved most readily by imposing a very intense plastic strain on the sample in each separate passage through the dies having ϕ close to 90° . An equiaxed grain size close to $1\mu\text{m}$ was produced in pure Al, under optimum conditions at room temperature by using a die with $\phi=90^\circ$. Four passes are required give a total strain of 4 and by rotating the sample by 90° between each pass [45].

2.1.2.2 Routes of ECAP

The multiple ECAP passes make it possible to rotate the billet around its longitudinal axis between adjacent passes, creating different ECAP routes. Four basic rotation routes (as shown in Fig. 2.1) have been used to study the microstructural development during the ECAP. They are defined by the rotation scheme of the billet around its longitudinal axis between two consecutive passes:

Route A : the billet is not rotated

Route B_C : the billet is rotated 90° clockwise

Route B_A : the billet is rotated 90° clockwise and counter clockwise alternatively

Route C : the billet is rotated 180° [46].

The shearing characteristics associated with ECA pressing for six different processing routes haven been examined and the optimum processing procedure is known for the development of texture [47]. The microstructure evolution and the development of a homogeneous microstructure of grains, separated by high angle boundaries in pure aluminum, occurred more rapidly in Route B_C than in Route B_A [48]. ECAP at room temperature using die of 90° on pure Al up to a strain of ~ 4 by four different routes showed that Route B_C was better in producing grains separated by high angle boundaries [49].

CP-Ti was processed to obtain ultrafine-grained states using ECAP routes B_C, B_A and C with a 90° die in the temperature range of $400\text{--}450^\circ\text{C}$. Route B_C was the most effective in grain refinement, yielding equiaxed grains with an average grain size of 260nm , while routes B_A and C yielded elongated grains. Route B_A is the least effective in grain refinement. The ECAP route does not have significant effect on

the micro-hardness. Route B_C does not produce Ti billets with better mechanical properties than routes B_A and C. However, it is considered that the optimum route due to its capability of producing better surface quality and equiaxed grains [46].

Figure 2.1 Rotation schemes for ECAP routes: Route A, Route C, Route B_A, and Route B_C [46].

2.1.2.3 Strain Rate of ECAE

The influence of pressing speed in ECAP was investigated using samples of pure Al and Al-1%Mg alloy over a range of pressing speeds from 10^{-2} to 10 mm s^{-1} . The results show that the speed of pressing has no significant influence on the equilibrium grain size at least over the range used in these experiments. However, the nature of microstructure is dependent on the pressing speed, as recovery occurs more easily at the slower speeds, as the microstructure is allowed to equilibrate. There is also indirect evidence for the advent of frictional effects when the cross-sectional dimensions of the samples are at or below $\sim 5 \text{ mm}$ [50].

2.1.2.4 Processing Temperature

The experiments were conducted to evaluate the influence of pressing temperature when materials are processed by ECAP. The tests were conducted using samples of pure Al, Al-3%Mg alloy and Al-3%Mg-0.2%Sc alloy at temperatures up to 573 K. All samples were pressed to sufficiently high strains to give homogeneous microstructures. The result shows two important effects. First, the measured grain size tends to increase with increasing pressing temperature. Second, even though grain refinement is achieved at all temperatures, there is a transition from arrays of

high angle boundaries at the lower temperatures to low angle boundaries at temperatures above 473 K in pure Al and at 573 K in the Al-3% Mg alloy. The Al-3%Mg-0.2%Sc alloy exhibits high angle boundaries at all pressing temperatures. The inability to achieve high angle boundaries at the higher pressing temperatures in these two materials is attributed to the higher rates of recovery, which lead to dislocation annihilation rather than the absorption of dislocations into the subgrain walls. The grain sizes after ECAP at high temperatures are consistently slightly larger than the grain sizes measured after ECAP at room temperature and then static annealing for one hour at the higher temperatures. This difference is a consequence of dynamic grain growth that occurs during the very rapid deformation imposed by the process of ECAP [51].

2.1.3 Finite Element Analysis and Physical Modelling of ECAE

2.1.3.1 Finite Element Analysis

The properties of the extruded materials are strongly dependent on the shear deformation behavior during ECAE and are controlled mainly by the following parameters like, die geometry (die/channel angle and outer corner angles), material properties (strain hardening behavior) and process conditions (friction, speed and temperature). To study the influence of these parameters on deformation behavior several authors conducted finite elements simulations [52 - 77]. As per equation (2.1) the strain induced in the deformed material is dependent on the channel angle and outer corner angle of ECAE tooling. The FE analyses reported by different authors show that the strain induced in the material also depends on the above-mentioned parameters. Theoretically it is very difficult to incorporate the influence of all the above-mentioned parameters into a holistic analysis without the help of finite element analysis.

The influence of tool angles on the shear deformation behavior during ECAE process has been analyzed without frictional conditions [52 - 56, 71, 75]. The influence of material behavior, strain rate, and the effects of strain hardenability and strain rate sensitivity on plastic flow and deformation inhomogeneity were studied using FE analysis [57 - 61, 72, 75]. To understand the influence of friction, material properties, back pressing plunger shape and backpressure in the ECAE process,

finite element simulations and experiments were carried out [58, 62 - 65, 71, 72, 75, 77 - 79].

Non-isothermal 2D and 3D FE simulations of ECAE process showed that die design and friction conditions are the most important parameters governing the formation of dead metal zones during extrusion [66]. The increase in temperature and temperature distribution in the work piece during ECAP were investigated by using the finite element method for Al-1%Mg and Al-3%Mg at different pressing speeds and were compared with the published experimental and analytical results [67]. Both the isothermal and non-isothermal simulations were carried to study the influence of ECAE process parameters on strain homogeneity of the deformed materials [66, 76] and the results showed that the material with low strain rate sensitivity, low friction, and large corner angle might lead to more uniform deformation and homogeneous distribution of strains.

Deformation behavior [74, 80 - 84], load requirements [80, 81], stresses generated [80, 81], and strain induced [68, 74, 80 - 83] through different routes of multipass ECAE was analyzed from FE simulations. The factors, which influence the load required and stress distribution in equal cross section lateral extrusion have been analyzed [69, 80]. Finite volume method simulations have been used to study the deformation behavior of single pass and multipass (via different routes) ECAE above the room temperatures. The effective strain distribution is compared with the Vickers hardness measurements in the cross section of the sample to analyze the strain homogeneity of ECAE [85, 86, 87].

2.1.3.2 Physical Modelling

An experimental study of the ECAE process using plexi glass tools and plasticine billets was carried out. This was to examine how well the theoretical models of the process describe the local shear strain increase and the element inclination angle decreases experimentally as the number of extrusions increases [88].

Physical modelling of ECAE was carried in a metallic die by using the plasticine billet, made of discs and spherical balls, to observe the influence of initial shape of billet constituents on the microstructure refinement. The number of ECAE passes

needed for microstructural refinement was observed by thorough mixing of the constituents [89].

Fig. 2.2 shows the plasticine billet, made of discs of alternate colors, in the ECAE die before deformation. The ECAE die with $\phi = 90^\circ$ and $\psi = 0^\circ$ was used for these studies, and extrusion were carried out with a speed of 1mm/sec. The billets processed via two different routes (Route A & B_C) up to 15 passes were shown in Fig.2.3. High degree of refinement and thorough mixing of two color layers was observed in Route A (Fig. 2.3a). Material accumulation at some regions, ten times refinement in size of constituents was also observed with the same route as shown in Fig. 2.3a. Much higher degree of mixing and refinement with almost no material accumulation, and discontinuous layers were observed with Route B_C (Fig. 2.3b).

Fig.2.2 Plasticine disc assembly in ECAE die before deformation [89].

Physical modelling of single pass [53] and two turn ECAE [80] was conducted by using two layer plasticine and wax respectively to validate the numerical simulation results and to observe the possible problems of the real process.

Grid distortion experiments were conducted to observe the shear deformation during ECAE. To observe the shear deformation during ECAE, pure aluminum, grids drawn inside the sample, is processed with a 90° sharp angle die. The measu-

Fig.2.3 Plasticine billet after 15 passes, matching surfaces of vertical section (a) processed via Route A (b) processed via Route B_C. The cracks in the billets were due to the billet drying up outside the die and were not a consequence of deformation [89].

-red shear angle of deformed grids was in agreement with that predicted by theory. Shear deformation was relatively uniform except at the top and end parts of the billet. Shear deformation of the area that is near the surface, which passes through the inner corner of the die is very uniform while that the region near the surface, which pass through the outer corner of the die is not uniform. It was observed from the experimental conditions the non-uniform area occupied about 15% of the billet. An empty dead zone in the outer corner of the die was observed. This dead zone is believed to be responsible for the non-uniform deformation in the part, which passes through the outer corner of the die [90]. Whereas the cylindrical copper rod embedded with aluminum plugs processed through 90° ECAE die, showed non-uniform deformation in the 10 - 12% of the lower part of the cross-section of the billet [71]. Dead zone is a cavity area (formed near the outer corner of the die), which was influenced by material properties and frictional conditions. During ECAE, billet surface near the dead zone receives no constraint forces from the die walls. Dead zone formation during the ECAE deformation was analyzed by both experimental and simulations [57, 90].

2.1.4 Microstructural Properties

Microstructure with ultrafine and nanometer grains causes improvement in mechanical and physical properties. ECAE is the most promising technique for producing this type of microstructure in bulk materials. Most of the aluminium, copper, nickel, titanium, steel and their alloys achieved this type of structure with high angle grain boundaries in bulk materials by ECAE.

For a severely deformed alloy to have a true submicron or nanocrystalline grain structure:

- The average spacing of high angle grain boundaries (HAGBs) (i.e. boundaries misoriented by $> 15^\circ$) must be less than 1 μm , or 100 nm, in all orientations and,
- The proportion of HAGB area relative to the total boundary area in the material must be greater than 70%.

Minimum fraction of HAGBs is therefore required to produce a stable grain structure [91].

ECAE experiments on high purity aluminium (99.99%) showed that the grain size reduced from 1 mm to 4 μm after single ECAE pass, but the microstructure consists of parallel bands of subgrains further divided by many boundaries having low angle of misorientation. Subsequent ECAE passes with different routes of ECAE caused the evolution of low angle boundaries into high angle boundaries [92]. For a 5056 Al-Mg alloy processed through ECAE, the structure is composed of fine sub grains and a lamellar-like structure with a large fraction of low-angle boundaries and a high average dislocation density inside the grains and subgrains [13]. The grain refinement to submicrometer level was also achieved in six different commercial Al alloys using ECAP [93, 94]. Microstructural examination on pure titanium deformed by a single pass of ECAP showed an elongated grains composed of multiple $\{1\ 0\ \bar{1}\ 1\}$ micro twins dominating the microstructure. This indicates that the severe plastic deformation imposed on titanium is accommodated by $\{1\ 0\ \bar{1}\ 1\}$ twins rather than by dislocation slip [95]. Pure nickel, subjected to ECAP (99.9%), showed refinement in grain size from $\sim 80\ \mu\text{m}$ to $\sim 0.3\ \mu\text{m}$ and the homogeneous microstructure of very fine grains separated by high angle boundaries [96]. Four ECAP passes with a strain of 4, produced ferrite grain refinement in low carbon

steel from 30 μm to $\sim 0.2 \mu\text{m}$ [97]. Ultrafine grain size with diameter of 160 nm is obtained in Cu-Cr-Zr alloy by 12 ECAE passes [98]. ECAE consolidated WC-Co cermets showed the average grain sizes are (range of 0.5 – 3.7 μm with 1.6 μm average) smaller than conventionally sintered material (range of 0.5 – 12 μm with 2.1 μm average) [28].

2.1.5 Mechanical Properties

2.1.5.1 Ultimate Tensile Strength, Yield Strength, Elongation to Failure (%)

Mechanical properties like tensile strength, yield stress and hardness of different materials were improved without significant degradation in ductility and toughness when processed through ECAE.

ECAE processed Armco Iron showed the ultimate tensile strength (UTS) and elastic limit (0.005%) increased by a factor of 2.8 and 4 times the annealed material [10]. UTS of low carbon steel increased 70% for single pass ECAP [99]. The tensile tests on six different aluminium alloys showed significant increase in proof stress after the first ECAP followed by a gradual increase with a further increase in strain. Elongation to failure exhibits a large decrease after the first pressing but it remains the same or increases with a further increase in imposed strain. These results suggest a potential enhancement of toughness in the alloys [93, 94]. Single pass ECAE of magnesium and magnesium alloys has showed improvement in mechanical properties [100]. The two-step process warm ECAP followed by cold extrusion can effectively improve the strength of CP-Titanium. The two step process utilized two strengthening mechanisms to strengthen CP-Titanium: (a) grain boundary strengthening due to small grain size during ECAP (b) dislocation strengthening from cold extrusion [101]. An increase in yield strength and UTS was observed in ultrafine-grained titanium after different passes during ECAE [46]. ECAE processed commercially pure tubular titanium showed good improvement in mechanical properties compared to the conventionally extruded titanium [17, 18].

2.1.5.2 Hardness

The hardness values at the central part of the ECAP consolidated 2024Al-3Fe-5Ni alloy increased with increase in number of passes and it also showed uniform tendency in the increment. The hardness values are remarkably higher than that of

conventionally hot extruded 2024Al-3Fe-5Ni alloy [31]. Three passes were sufficient to attain a high hardness value. Samples processed through Route A and Route C showed higher hardness values compared to Route B_C and B_A [32]. ECAE formed and consolidated WC-Co cermets from mechanically alloyed powder showed that hardness values were comparable to the conventionally processed cermets [28]. ECAE consolidated amorphous based copper powder showed increment in hardness values with increase in number of passes [34].

The microhardness values were slightly higher at higher strain rates for pure aluminium. Microhardness measurements on Al-1%Mg alloy revealed an increase in the hardness up to five passes through the die, but the values stabilized after higher number of passes [50]. The CP-Ti processed by ECAP as well as in the two-step processes (ECAP + cold extrusion) showed increase in micro-hardness values. In all the cases micro-hardness measured in longitudinal direction was higher than in the transverse direction [46, 101].

2.1.5.3 Fatigue Limit

The ECAP processed 5056 Al-Mg alloy material exhibited higher tensile and low-cyclic fatigue strength under constant stress amplitude [13]. ECAE processed Cu-Cr-Zr alloy showed significant improvement of tensile and high cycle fatigue properties, without sacrificing ductility and low cycle fatigue properties [98]. The fine-grained Ti processed by ECAP reveals considerable increase in fatigue life and fatigue limit under constant load testing when compared to that in the coarse-grain state [102].

2.1.5.4 Superplasticity

The phenomenon of superplastic deformation in metals refers to extreme extensibility with elongation usually between 100 and 1000 percent. To exhibit superplasticity, the metal should possess equiaxed grains of an optimum grain size (less than 10µm for high temperature superplasticity), which depends on the material system. Low temperature superplasticity refers to the occurrence of superplastic elongations at temperatures lower than those generally required for superplasticity. If grain size is reduced to submicrometer level, it is possible to achieve low temperature superplasticity. The occurrence of superplasticity at high

strain rates and low temperature is especially attractive in metal forming operations because of the consequent reductions in forming time and tool wear.

The cast alloy of Al-3%Mg-0.2%Sc subjected to superplastic deformation after ECAP showed very high tensile elongation [103]. Superplastic behavior at a high strain rate and relatively low temperatures was reported in a Cu-40% Zn alloy [104] by the application of severe plastic deformation at a temperature associated with phase transformation.

2.1.6 Physical Properties

Increase in magnetic anisotropy was reported in Armco iron due to the strain intensity of the ECAE process [10]. The ECAP processed $\text{Pr}_{20}\text{Fe}_{73}\text{B}_{5.5}\text{Cu}_{1.5}$ alloy showed coercivity greater than 1500 kA/m. This increase in coercivity is due to grain refinement and uniform microstructure obtained through SPD [15].

The obstacles needed for pinning magnetic flux in superconducting materials were easily obtained through ECAE process. The ECAE processed Nb-Ti alloy showed these defects in the shear plane, normal to the shear direction, similar to the single crystals [10]. ECAE processed and aged Cu-Cr-Zr alloy showed electrical conductivity, 81% of IACS [98].

The ultrafine-grained copper obtained through ECAE possesses showed notably better resistance to stress corrosion cracking (SCC) when compared to coarse-grain copper. [14].

2.1.7 Potential for Scaling the ECAE Process

The potential for scaling the ECAP for use with large samples was investigated by conducting tests on an aluminium alloy using cylinders with varying diameters. The results showed that refinement of the microstructure and subsequent mechanical properties after pressing were independent of the initial size of the sample and for the largest sample with a diameter of 40 mm, independent of the location. Measurements of applied load during ECAP showed the required load was related to the sample strength and, at least for well-lubricated samples, frictional effects between the sample and the die wall make relatively minor contribution to the measured load. The results demonstrated the feasibility for scaling ECAP to large

sizes for use in industrial applications. An example would be the production of reasonably large sheets of metal for use in superplastic forming operations by rolling after ECAP [93].

ECAE was attempted at room temperature to refine the grain sizes of six different aluminium alloys 1100, 2024, 3004, 5083, 6061 and 7075. Studies conducted to find out the effect of sample size on the microstructure and mechanical properties have suggested that the scaling up of the sample size is feasible without any significant change in grain size and mechanical properties [94].

2.1.8 ECAE of Intermetallics

Canned preforms of titanium aluminide Ti-44.5Al-2Cr-2Nb were hot worked via equal channel angular extrusion [105]. The non-uniform deformation during ECAE increased rapidly as the preheat temperature decreases. Multiple passes ECAE was used to breakdown and refine the structure of near-gamma titanium aluminide ingots.

ECAE is a promising method for producing sub micron sized equiaxed microstructures in titanium aluminides. Equiaxed fine-grained microstructure with grain sizes $< 2 \mu\text{m}$ were successfully produced in Ti44Al11Nb by ECAE. Severe plastic deformation below the eutectoid isotherm results in the conversion of gamma + alpha lamellae to equiaxed gamma + alpha₂ grains [106]. In addition, the equiaxed microstructure with grain sizes $< 2 \mu\text{m}$ in Ti – (42-52) atomic %Al and Ti-48Al-2Cr alloys were obtained by ECAE at 1000 and 1100 °C. Fine grained γ - Ti Al showed increase in room temperature ductility [107].

2.1.9 Potential Applications of ECAE

2.1.9.1 Ultra Fine Grain Materials and Superplastic Forming Sheets

ECAE has the capability to produce ultrafine grain sizes with homogeneous microstructure even in bulk materials. The large sheets for superplastic forming can be produced by rolling after ECAP.

2.1.9.2 Processing of Nanocomposite Wires for Pulse Magnet Applications

By combining ECAE with wire drawing, nanocomposites of Cu-Ag, which have both high strength and reasonable conductivity was produced [11]. Filamentary

structure was obtained in the ECA extruded Cu-18Nb. The highest strength level obtained was for Cu-Ag with ultimate tensile strength of 1070 MPa with a conductivity of 80 % of the International Annealed Copper Standard value.

2.1.9.3 ECAE of Gamma Titanium Aluminide Alloys

Based on gamma (TiAl) phase, the titanium aluminides are under intense development as candidates for components in light weight high performance jet engines. However, the usefulness of these alloys depends on the careful control of microstructure during manufacture, which can be achieved by ECAE by breaking down coarse as-cast structure without decreasing the size of billet. ECAE has been successfully applied to Ti-45.5Al-2Cr-2Nb alloy to get near lamellar microstructure with a grain size of 150 μ m [11].

2.1.9.4 In-situ composites

In situ composites were fabricated by intensive simple shear, by extruding repeatedly in the same direction, which transformed ductile second phase particles into aligned filaments. Results of experiments on Cu-18Nb and Cu-25Ag alloys demonstrated the ability of this method to fabricate filamentary composites from cast bulk products with no change in work piece cross-section [108].

2.1.10 ECAE Processing of Powders

Design of materials, densification of powders by retaining fine grain structures is possible through ECAE processing of powders. Generally, ECAE processing of powders is carried through powder in tube (PIT) technique at different temperatures. The following wide range of powders: soft metallic, alloys, amorphous, bulk metallic glasses and hard tungsten carbide powders, has been subjected to ECAE processing through PIT.

High temperature ECAE has been applied to WC-Co powders [12, 28], nanostructured titanium silicide [29]. Successful formation and compaction of WC-Co was also achieved after two ECAE passes at 1200°C and subsequent annealing in argon at 1400°C [12, 28].

ECAE processing of pure aluminum [30], aluminum powder alloys [31, 32], semi amorphous aluminum alloy [33], amorphous aluminum and copper powders [34,

35], zirconium based bulk metallic glasses [36] were carried at warm (100 - 500°C) and low (25 - 100°C) temperatures. Micro and nano copper powder [37], aluminum and copper alloy [38] were consolidated through room temperature ECAP consolidation.

Pure aluminum particles compacted through back pressure ECAE, showed enhanced mechanical properties with fine grains [30]. The microstructure of consolidates (2024 Al-3Fe-5Ni [3F5N] alloy) made by ECAP and hot extrusion, were compared. The alloy consolidated by ECAP showed much finer microstructure (micro-cellular and fine eutectic which exists in rapidly solidified powders) compared to hot extrusion [31]. The compaction studies [32] on 2024 Al-3mass%Fe-5mass%Ni (3F5N) alloy powder showed that ECAP was capable of obtaining compaction with almost 100% density and with very high hardness even after a single pass. The results of repetitive pressings through different routes A, C and B_C, showed that Route B_C is the most effective in producing a reasonably homogeneous microstructure including fine intermetallic particles and equiaxed sub-grains, which contribute to the superplastic-like deformation behavior at high strain rates.

ECAE consolidation is also a promising avenue for the fabrication of bulk metallic glasses, if exposure time in the high temperature, super cooled liquid regime can be reduced. Cu₅₀Ti₃₂Zr₁₂Ni₅Si₁ amorphous based copper powder was consolidated by warm ECAE and compared with consolidates made by conventional extrusion. The consolidation was done at above the glass transition temperature but below the crystallization temperature. The ECAE consolidates showed a super cooled liquid region similar to that of starting powder but with slightly higher glass transition and lower crystallization temperatures [35]. In addition the Al₈₅Ni₁₀Y_{2.5}La_{2.5} alloy powder produced by gas atomization was compacted using ECAE to about 99% of full density. Powder particles consisting of amorphous and FCC phases were heavily deformed during ECAE compaction and they were well sintered to each other [33].

2.2 MAGNESIUM BORIDE (MgB_2)

MgB_2 is a brittle intermetallic compound, showing a superconducting transition temperature of 39K from the resistivity and magnetization measurements [19]. The temperature is highest yet determined for a non-copper-oxide bulk superconductor. Even though the critical temperature of MgB_2 is three times lower than the mercury based high T_C superconductors (HTSC) cuprates (134 K), it has some additional advantages compared to cuprates and low temperature superconductors. Compared to low temperature superconductors like NbTi, Nb_3Sn , avoiding the usage of liquid He is the major advantage of MgB_2 . In addition, MgB_2 has a simple crystal structure, low material density, better mechanical behavior, lower cost, lower anisotropy, larger coherence lengths and high J_C values due to weak link free grain boundaries. High transport critical current densities of the order of 10^6 A/cm^2 have been obtained in the temperature range between 4.2 and 25 K in bulk samples. MgB_2 promises a higher operating temperature and a higher device speed than the present electronics based on Niobium. Furthermore, a T_C of 39K is high enough for use at elevated temperatures if a cryogen-free cooling system is readily available. For large-scale technological applications and electronic devices the main requirement is higher critical current densities (J_C). The J_C in polycrystalline high temperature superconducting (HTS) materials (perovskites) is highly sensitive to applied magnetic fields primarily due to the weakly coupled nature of the grain boundaries. In addition, polycrystalline MgB_2 samples showed intergranular critical current densities and large bulk magnetic flux pinning. Further improvement in J_C of this material can be achieved by the introduction of magnetic flux pinning centers by further control of the microstructure. For practical applications like magnets in levitated bearings, linear or rotating electric motors, shielding of alternate and low magnetic fields, current leads of cryogenic apparatus, fault current limiter for electrical grids are either inductive or resistive, and for processing methods like magnetron sputtering and laser ablation, MgB_2 is needed in the bulk form. For power transmission and electronic applications it is needed in the form of tapes/wires and thin films [109-115].

MgB_2 is mechanically hard and brittle, and is not amenable to be drawn into wires/tapes, and it is also challenging to prepare bulks of MgB_2 due to the volatility and reactivity of magnesium. The MgB_2 will decompose at relatively low

temperature of 1000 °C. To overcome all these problems, power in tube (PIT) method was successfully applied for sintering of bulk MgB_2 and for fabricating wires/tapes. The bulks and wires/tapes of MgB_2 are mostly produced through PIT technique by two different processes. One is an *in-situ* process in which constituent powders, such as Mg and B, are packed into a metallic tube and a MgB_2 phase is then formed inside the tube due to heat treatment. The other is *ex-situ* process in which commercially available or self-prepared MgB_2 powder is packed into a tube. In both cases after filling the powder in tube, further processes like sintering under high pressure and temperature, drawing and rolling will be done to obtain the final shapes like bulks and tapes/wires. The *ex-situ* processed MgB_2 bulks and tapes showed higher critical current densities [109,116,117]. The selection of sheathing/tube material is very important in terms of avoiding reaction between the tube and MgB_2 when subjected to high temperatures, and to obtain high packing density to improve grain coupling during fabrication of tapes/wires. In the process of selecting the best sheathing material several researchers proposed materials like Cu, Ag, Ni, Cu-Ni, Nb, Cu/NbZr, Ta, BN, stainless steel, Fe and carbon steel. Among these materials, tapes/wires produced with Fe and its alloys like carbon steel as a sheathing material and MgB_2 as a core, showed good improvement in properties with high packing density by avoiding reaction with MgB_2 [113 - 116, 118 - 124].

Density, porosity and microstructure of MgB_2 samples are very important factors for obtaining high transport critical current densities. Transport current densities are limited due to thermal stability of the conductors caused by the large filament size, phase impurities, inhomogeneity and especially a non-perfect densification of the superconductor. In all, the density and porosity of MgB_2 samples play a crucial role for transport currents [23,124,125]. To fabricate bulk MgB_2 with higher densities, most of the researchers used conventional & advanced P/M techniques like, high pressure sintering [24, 112, 126-128], hot pressing [129], magnetic compression [25], shock wave consolidation [26]. The mechanical properties of MgB_2 bulks prepared by all the above techniques are detailed in Table 2.1. With these techniques there is a limitation in the sample dimensions and possible shapes that can be produced. To decrease the pressure requirements and to increase the sample sizes, some authors used hot isostatic pressing (HIP), HIP with DMCUP (dense

Table 2.1 Mechanical properties of MgB₂ bulks prepared by different processing techniques.

Processing Technique	Density (g/cc) of MgB ₂ bulks	Hardness of MgB ₂ bulks	Elastic Modulus (GPa)	Poisson's ration
Hot isostatic pressing (HIP) with dense material cooled under pressure (DMCUP) [23]	2.67	10.4 GPa	272.5	0.181
Hot isostatic pressing (HIP) [27]	2.63 ± 0.02	----	---	---
Cold isostatic pressing [26]	2.05	72 (VHN)	---	---
<i>Shock wave consolidation</i>				
Under water shock consolidation [26]	2.41	270 (VHN)	---	---
Gun method [26]	2.39	230 (VHN)	----	---
Magnetic compression [25]	2.45	---	---	---
<i>High pressure sintering</i>				
At 700°C [24]	2.70	2153.3 (Kg/cm ²)	---	---
800°C [24]	2.71	4109.5 (Kg/cm ²)	---	---
1000°C [24]	2.88	1448.8 (Kg/cm ²)	---	---

material cooled under pressure) with a pressure of 200 MPa, maximum temperature of 1000 °C with a time of 200 minutes. The sample dimensions obtained are still limited (20 mm diameter and 10 mm height). However, HIP processed samples showed good superconducting properties [27, 130-132]. Techniques like HIP and hot pressing are feasible, but with limitation in the dimensions of the samples (needed for practical applications) to be produced, due to the practical difficulty in the use of very large vessels and dies when high pressures and temperatures are involved. In addition the prolonged temperatures in HIP and sintering time and temperature in conventional techniques may cause grain growth. Grain growth may cause the decrease of superconducting properties. There is a need to develop new methods to overcome these difficulties and to obtain higher density in large dimension samples with relatively low application of pressures and temperatures. In this research ECAE is used to compact MgB_2 powder into bulk to achieve theoretical densities with refined microstructures.

2.3 SHEAR PUNCH TEST

Shear Punch Test (SPT) is an efficient test technique for evaluating the mechanical properties when the material availability is limited. The mechanical properties, viz., yield and ultimate strength, and strain hardening exponent values can be calculated from the SPT data [133,134]. A linear correlation between the SPT data and tensile data has been reported by many investigators [133,134]. This technique has successfully been used over the past two decades to evaluate irradiated materials in the nuclear Industry. Though this technique was initially employed to metals and alloys, it has subsequently extended to other materials like composites [134], bio materials [135, 136], and nanocrystalline materials [137].

Typical stress-displacement curves reported during SPT of pure aluminium and plain carbon steel were shown in Figs. 2.4 & 2.5. Several stages can be distinguished on SPT curves shown in Figs. 2.4 & 2.5. These are: (a) an initial linear region, (b) a point of deviation, (c) a non-linear region and (d) an ultimate loading (stress) point. The correlations reported between the tensile and shear yield, and ultimate strengths were shown in Figs. 2.6 & 2.7. A linear correlation of $\sigma_Y = 1.77 \tau_Y$ between the tensile and shear yield strengths, $\sigma_{UTS} = 1.77 \tau_{USS}$ were reported [133].

Figure 2.4 Shear punch test curve for pure aluminium [133].

Figure 2.5 Shear punch test curve for plain carbon steel [133].

Figure 2.6 Correlation between shear and tensile yield strengths of different materials [133].

Figure 2.7 Correlation between ultimate shear and tensile strengths of different materials [133].

CHAPTER 3

FINITE ELEMENT ANALYSIS OF ECAE

3.1 LIMITATIONS OF THEORETICAL STUDIES

Most of the theoretical equations developed on ECAE are based on the upper bound method and slip line field theory [8 – 10, 43]. In both techniques all assumptions are based on rigid plastic material. However, in practice most of the materials are strain hardenable. The behavior of strain hardening material is different from rigid plastic material. Thus the theory cannot predict the realistic deformation behavior, punch pressure, stresses and strain distribution in the elastic/plastic deformation zone. Therefore, it is necessary to analyze the deformation behavior of work piece during ECAE process. This information is most important for designing the optimum die to obtain optimum properties in the processed materials.

3.2 FINITE ELEMENT ANALYSIS (FEA) AS MY CHOICE

The successful implementation of ECAE process to synthesize different materials and alloys to achieve the expected material properties relies on the design, control and optimization of the process. For this knowledge on the deformation pattern, stresses (loads), and heat transfer, as well as technical information associated with lubrication, material handling, die design and manufacture etc. is required. To solve numerically the governing equations finite element method has to be used compared to the traditional analytical methods, such as the upper bound analysis and slip line field theory. Further, consideration of complicated and important realistic factors (like strain hardening behavior of the material, frictional conditions and speed of the process) is more possible through finite element analysis. Thus FEA was chosen in current study to study the strain homogeneity (uniform strain distribution), and deformation behavior in ECAE.

3.3 MOTIVATION FOR CURRENT FINITE ELEMENT STUDIES

The strain achieved in the material through ECAE is very sensitive to the tool angles (ϕ and ψ) [8 - 10], strain hardening behavior of material [57, 62], frictional conditions [62, 63], speed [60] and temperature [66] of the process, all of which can have a large effect on the microstructure and strain homogeneity within the

processed billet. To study the influence of ϕ and ψ on strain induced through ECAE, researchers used finite element, physical modelling and real experiments. The experiments on aluminum alloy with different channel angles showed that the channel angle, ' ϕ ' being nearer or equal to 90° is essential in order to achieve ultrafine microstructure with equiaxed grains separated by high angle grain boundaries in minimum number of passes [45]. At the same time the physical and finite element modelling of ECAE with ($\phi = 90^\circ$ and $\psi = 0^\circ$) showed that non uniform deformation in the processed material, formation of dead metal zone, generation of excessive die stresses and severe stress concentration due to sharp outer corner [41, 57, 62, 90]. The non-uniform deformation is also observed with $\phi > 90^\circ$ [41, 63]. In addition to that FE simulations of ECAE process claimed that ($\phi = 90^\circ$ and $\psi = 30^\circ$ to 90°) produces non-uniform deformation in the material [52 - 54]. But plasticine experiments with $\phi = 90^\circ$ and $\psi = 28^\circ$ yielded uniform deformation in the material compared to $\phi = 90^\circ$ and $\psi = 0^\circ$ [88]. The predictions on the most important parameter ' ψ ' are quite contradictory. It is necessary to sort out discrepancy in ' ψ ' and to find the optimal outer corner angle, which can generate strain homogeneity in ECAE by avoiding detrimental affects, and with minimum punch pressure.

As per theoretical calculations of ECAE [8 - 10], the inner corner should be sharp. But with sharp inner corner, there is a chance where surface cracks could occur in low ductile and high strength samples and even in ductile samples during multiple ECAE passes. This sharp inner corner radius is also a location of severe stress concentration on the die surface which causes high load requirements. To avoid these effects the possible solution is to provide smooth inner corner radius. At the same time this smooth inner corner radius should not vary the deformation behaviour and strain homogeneity of the samples. Hence there is a need to find the optimum inner corner radius, which does not vary the strain homogeneity of the samples.

In practice different channel angles $\phi = 60$ to 160° were used to produce ultrafine grain microstructures in different materials, [45, 138] based on the material strength and ductility. Channel angle ' ϕ ' is also one of the major parameters which can vary the strain homogeneity in ECAE. Before design and fabrication of ECAE die, it is

important to know how the deformation behavior, strain homogeneity varies with different channel angles.

Severe strains induced by multiple passes via different routes of ECAE are responsible for achieving enhanced microstructure and mechanical properties in the deformed materials [46]. It shows that the strain homogeneity is also influenced by processing route. Thus it is necessary to know how the strain homogeneity varies with different processing routes of ECAE.

3.4 PROCEDURE FOR FINITE ELEMENT STUDIES

The commercial finite element code Abaqus/Explicit was used to carry out all simulations. To simplify the analysis to a two-dimensional problem, the symmetry plane through the centre of the sample was modeled by assuming plane strain conditions. Simulations were carried under the following conditions (shown in Table 3.1): ideal (elastic-perfectly plastic material with frictionless contact between the die and sample), realistic (elastic-strain hardening material with frictional contact between the die and sample). The die designed and fabricated will be used to extrude materials with different strengths: solid, tubular and powder materials. Hence it is reasonable to assume the strongest material solid, to design the die. Moreover, powder consolidation is carried out only by enclosing the powder in metallic tubes [32, 36]. Extent and uniformity of consolidation depends on the strain homogeneity in the deformed samples. This strain homogeneity is in turn influenced by tool angles, strain-hardening behavior of material and friction between the contacting surfaces while thickness of the die is determined by strength of both the materials. Thus, the design of die geometry for fulfilling the desired purpose requires consideration of strain hardening exponent and frictional coefficient of a metallic material. Solid sample/billet with dimensions $60 \times 10 \times 10$ (l x b x t) mm³ was considered for simulations. Die and punch were modeled with analytical rigid elements. The boundary conditions for dies are all movements in all directions are zero, where as for punch movement in z direction is allowed with a speed of 1 mm/sec. Von-Mises yield criterion is considered for billet. Billet material was modeled with four-node bilinear plane strain quadrilateral (CPE4R) elements [139]. To accommodate large strains during simulations the adaptive meshing (automatic remeshing) was used. Reduced integration and hourglass control were used in the

analysis. To attain isothermal conditions, simulations were performed with a speed of 1 mm/sec. Smoothness of loading is important in quasi-static analysis. In order to attain smoothness in loading, smooth step amplitude is chosen for loading the punch.

Table 3.1 Material Properties and frictional conditions of AA1100 considered for simulations [140, 141].

Ideal conditions	Realistic conditions
Elastic modulus = 69 GPa	Elastic modulus = 69 GPa
Poisson ratio = 0.33	Poisson ratio = 0.33
Density = 2710 kg/m ³	Density = 2710 kg/m ³
Yield strength = 34.483 MPa	Yield strength = 34.483 MPa
	Flow stress $\sigma_f = 173.79 \varepsilon^{0.304}$
	Friction coefficient = 0.05

To overcome the discrepancy on angle ' ψ ' and to determine the optimum angle which can generate optimal strain homogeneity in the processed billet, simulations were carried out with $\phi = 90^\circ$, $\psi = 0, 10$, and 20° under both ideal and realistic conditions.

To find the optimum inner corner radius, simulations were carried out with different inner corner radii (r) for the best outer corner angle selected from the above simulations. These simulations were carried under ideal conditions.

The influence of channel angle on strain homogeneity in ECAE was studied by conducting the simulations under realistic conditions for a range of channel angles from 60° to 150° , and with the best outer corner angle determined from the above simulations.

The influence of processing route on strain homogeneity in ECAE was studied by conducting the simulations under realistic conditions for two processing routes A & C, with tool angles $\phi = 90^\circ$, $\psi = 10^\circ$.

3.5 STUDIES ON STRAIN HOMOGENEITY IN ECAE BY FEA

3.5.1 Validation of ECAE FE Model

During the equal channel angular extrusion/pressing (ECAE/P), the equivalent plastic strain/effective strain (PEEQ) induced in the sample, given by Eq. 2.4, was developed from the slip-line theory along with the assumptions of ideal plastic material behavior and frictionless conditions [8]. To validate the current finite element model, simulations were carried out with tool angles: $\phi = 90^\circ$ and $\psi = 0^\circ$ by assuming the material behavior as elastic-perfectly plastic and frictionless contact between the die and sample.

Fig. 3.1 shows the PEEQ distribution obtained when sample is simulated with $\phi = 90^\circ$ and $\psi = 0^\circ$. Theoretically shear deformation takes place in thin layers at the crossing planes of the channels [10]. The same can be observed from the PEEQ contours in the deformation zone in Fig.3.1, thus uniform strain distribution was achieved in the entire sample. Fig. 3.2 shows the PEEQ variation across the width A-B (located in Fig 3.1) of the sample. PEEQ value obtained from the theoretical calculations as per Eq. 2.4 is also shown in the same figure for comparison. The PEEQ values obtained from FE model are almost nearer to the theoretical calculations. Similar observation was reported in earlier FEA studies of ECAE with perfectly plastic material [52, 53]. However the strain values near the outer corner are slightly lower and nearer to theoretical strain values in current study due to fine mesh. Hence, it shows that the developed FE model is close to the theoretical calculations.

Figure 3.1 PEEQ contours of the sample simulated with $\phi = 90^\circ$ and $\psi = 0^\circ$.

A

B

over estimates the stress and strain values in that region. The shape of the front portion of the deformed sample changes a lot and the steady state region of the deformed sample also less with coarse mesh.

Figure 3.3 Influence of number of elements (a) 600, (b) 2400, and (c) 9600 on the deformation behavior.

Fig.3.4 shows the equivalent plastic strain/effective strain (PEEQ) contours obtained with different number of elements. With increase in number of elements, the strain distribution becomes more uniform in the entire sample. To confirm this, variation of effective strain across the width A-B (located in Fig.3.4) of the sample was plotted in Fig.3.5. The strain values are almost uniform upto 40% width of the sample (from A) with all mesh sizes, whereas in the remaining 60% width it varies a lot. This variation is mainly due to corner gap/dead zone formation near the outer corner. To find the most suitable number of elements to carry out further simulations, it is compared with the theoretically calculated strain values. Strain variation with 2400, 9600 elements are almost uniform with theoretical calculations.

But the corner gap formed near the outer corner of the die with 2400 elements is bit higher compared to 9600 elements. In addition, with 9600 elements the shape of the PEEQ contours in the deformation zone matches with the theoretical shear deformation patterns [10]. Accordingly, further simulations in current study were carried out with 9600 elements to obtain pragmatic deformation behavior and strain distributions.

3.5.3 Influence of Outer Corner Angle

Outer corner angle ' ψ ' is one of the most important parameters which can influence the deformation behavior and strain homogeneity in ECAE. Thus, the simulations (shown in Table 3.2) were conducted to find the optimal outer corner angle which can induce strain homogeneity in ECAE processed materials with channel angle, $\phi = 90^\circ$. Simulations were carried under ideal conditions (elastic perfectly plastic material and frictionless contact between the die and sample) and realistic conditions (elastic-strain hardening material with frictional contact between the die and sample). Strain homogeneity can be inferred from the overall deformation behavior, shape of deformation zone and equivalent plastic strain (PEEQ)/effective strain distribution. The flow behavior of the sample in the die can be obtained from the overall deformation behavior of the sample/billet.

Table 3.2 List of finite element simulations performed to determine the optimal outer corner angle with $\phi = 90^\circ$.

Case	Outer corner angle (ψ) in degree	Material behavior (Elastic-Perfectly Plastic/ Elastic-Strain Hardening)	Friction coefficient (μ)
Ideal			
1	0	$n = 0.0$	0.0
2	10	$n = 0.0$	0.0
3	20	$n = 0.0$	0.0
Realistic			
4	0	$n = 0.304$	0.05
5	10	$n = 0.304$	0.05
6	20	$n = 0.304$	0.05

3.5.3.1 Overall Deformation Behavior of the Sample/Billet

This section explains how the sample deformation varies with different outer corner angles for $\phi = 90^\circ$ under both ideal and realistic conditions. The deformed meshes of the samples are shown at the end of deformation in Figs. 3.6 & 3.7. It depicts the overall deformation behavior of the sample, corner gap/dead zone formation and thus gives broad idea on the generation of stresses and strains during the deformation. The deformed mesh directly indicates the amount of shear deformation induced in the samples during ECAE. The deformation takes place in two steps with $\phi = 90^\circ$ in all cases. The front end of the sample touches the bottom of the die exit channel first followed by flowing. This can be clearly observed from different stages of the deformation shown in Figs. 3.8 & 3.9.

Fig. 3.6 shows the deformed mesh of the samples under ideal conditions. Mesh is not distorted severely with all outer corner angles under ideal conditions due to dead zone formation. The dead zone formed is continuous with all outer corner angles under ideal conditions and is present in all deformation steps shown in Fig. 3.8. With $\psi = 0^\circ$, under ideal conditions the dead zone formed is very small compared to $\psi = 10^\circ$ & 20° . Even with very small amount of dead zone formed with $\psi = 0^\circ$, the deformation is similar to ideal simple shear [10]. This shows that the small dead zone formed is due to rigid body rotation. Similar behavior is observed in earlier studies under ideal conditions with $\psi = 0^\circ$ [52, 53, 75]. With $\psi = 10^\circ$ & 20° dead zone formed is greater and this dead zone resulted in the generation of tensile stresses (shown in Fig. 3.10) in the deformation zone at the initial stages of deformation. The intensity of the tensile stresses increased with an increase in outer corner angle. No gaps were observed between the sample and at the top and bottom surfaces of the die exit channel due to lower outer corner angles in current study, whereas it was observed with higher outer corner angles [52, 53, 75]. The shape of the front end of the sample is more uniform with $\psi = 0^\circ$ & 10° , slightly bend upwards with $\psi = 20^\circ$.

Fig. 3.7 shows the deformed mesh of the samples under realistic conditions. With $\psi = 0^\circ$, the mesh is more severely distorted and the behavior is closer to simple shear as per theoretical expectations [10] than that with other outer corner angles. At the beginning of the extrusion the billet sticks on to the bottom of the exit channel die -

Figure 3.6 Deformed mesh of the samples simulated with different ECAE tool angles $\phi = 90^\circ$ and (a) $\psi = 0^\circ$, (b) $\psi = 10^\circ$, (c) $\psi = 20^\circ$ under ideal conditions ($n=0, \mu=0$) at the end of deformation.

Figure 3.7 Deformed mesh of the samples simulated with different ECAE tool angles $\phi = 90^\circ$ and (a) $\psi = 0^\circ$, (b) $\psi = 10^\circ$, (c) $\psi = 20^\circ$ under realistic conditions ($n=0.304, \mu=0.05$) at the end of deformation.

walls (observable in Fig. 3.9 with $\psi = 0^\circ$) due to friction and then starts flowing subsequently. The sticking of the sample to the die walls depends on the value of the friction coefficient. If the friction coefficient is high it may cause damage to the die surface. The influence of friction and sharp outer corner was represented in the form of the excessive deformation at the outer side of the deformed billet. The inability of the sample to flow along the sharp outer corner angle is represented in the form of severely damaged mesh. Similar non-uniform deformation is also observed at the start of the extrusion [62]. With $\psi = 10^\circ$ & 20° , the mesh deformation is not severe due to outer corner angles and dead zone formation.

In realistic conditions, with $\psi = 0^\circ$ the dead zone is formed between 15% and 30% of the deformation step (Fig. 3.7). The influence of dead zone is clearly observable in the form of undeformed mesh between the severely deformed regions. If the dead zone is continuous in the entire deformation as per the previous expectations [62, 75, 90] the mesh should not be deformed too severely beyond 30% of the deformation at the outer corner of the sample. This shows that the dead zone formation is not continuous throughout the deformation step with $\psi = 0^\circ$, under realistic conditions of current study. Similar behavior was observed in recent study [83]. With the increase in outer corner angle, $\psi = 10^\circ$ & 20° , dead zone formation is greater and is continuous in the entire deformation step unlike that of $\psi = 0^\circ$ (observable in Fig. 3.9). However, this dead zone is larger at 10% deformation, smaller at 60%, and at the end of the deformation steps. This dead zone resulted in the generation of tensile stresses in the samples (observable in Fig. 3.11) at the beginning of deformation in the deformation zone. With $\psi = 10^\circ$, the tensile stresses generated are confined in a very small region due to smaller dead zone formation. The intensity of the tensile stresses increased with an increase in outer corner angle. In practice this may result in the initiation or generation of cracks in the deformed samples. The increase in outer corner angle also exhibits non-uniform deformation at the outer side of the deformed samples and the deformation of mesh is not as severe as shown in Fig. 3.7 with $\psi = 0^\circ$. These effects together cause the deformation to deviate from the simple shear. This similar type of behavior was also observed in the perfectly plastic material with frictionless behavior, and strain hardening materials with friction and frictionless behavior with higher outer corner

Figure 3.8 Equivalent Plastic Strain (PEEQ) contours of the samples simulated with different ECAE tool angles $\phi = 90^\circ$ and (a) $\psi = 0^\circ$, (b) $\psi = 10^\circ$, (c) $\psi = 20^\circ$ under ideal conditions ($n=0$, $\mu=0$) at 10%, 60%, and end of deformation.

Figure 3.9 Equivalent Plastic Strain (PEEQ) contours of the samples simulated with different ECAE tool angles $\phi = 90^\circ$ and (a) $\psi = 0^\circ$, (b) $\psi = 10^\circ$, (c) $\psi = 20^\circ$ under realistic conditions ($n=0.304$, $\mu=0.05$) at 10%, 60%, and end of deformation.

angles [52 – 54, 83]. At the same time dead zone formation is unavoidable with strain hardening materials which is also observed in earlier study [54]. Thus, it is

Figure 3.10 Principal Stress (S22) contours in Pascal of the samples simulated with different ECAE tool angles $\phi = 90^\circ$ and (a) $\psi = 0^\circ$, (b) $\psi = 10^\circ$, (c) $\psi = 20^\circ$ under ideal conditions ($n=0$, $\mu=0$) in steady state deformation zone.

Figure 3.11 Principal Stress (S22) contours in Pascal of the samples simulated with different ECAE tool angles $\phi = 90^\circ$ and (a) $\psi = 0^\circ$, (b) $\psi = 10^\circ$, (c) $\psi = 20^\circ$ under realistic conditions ($n=0.304$, $\mu=0.05$) in steady state deformation zone.

better to allow low amount of dead zone formation, made possible with either sharp or lower outer corner angles.

The shape of the front end of the sample (Fig. 3.7) is not uniform in realistic conditions with all outer corner angles, whereas it was uniform under ideal conditions with $\psi = 0^\circ$ & 10° . The upward bend is observed near the front end with $\psi = 20^\circ$ in realistic conditions is very small compared to ideal conditions. The upward bend increases with increase in outer corner angle irrespective of material behavior [68, 75]. As the outer corner angle increases the front end of the sample starts to bend upwards, and generates non-uniform deformation in the front end of the sample. This bending causes wastage of substantial volume of material during multi-pass ECAE. With an increase in the outer corner angle, flow of the sample becomes smoother, avoiding generation of excessive die stresses and severe deformation of the mesh. Nevertheless, non-uniform deformation still exists at the outer side of the deformed samples, but the amount is reduced with sharp outer corner angle. The amount of non-uniform deformation is observable in the form of equivalent plastic strain (PEEQ) distribution.

3.5.3.2 Deformation zone/Plastic zone

The strain homogeneity/uniform strain distribution in the entire sample can be achieved if the deformation follows simple shear perfectly [10]. The simple shear behavior can be inferred from the shape of the deformation zone/plastic zone, which is identified from the effective stress distribution contour plots. The effective stress distribution obtained in the deformation zone with different tool angles under different conditions is shown in Figs. 3.12 & 3.13. This is shown in steady state region at 60% of the total deformation step in order to obtain the pragmatic deformation behavior. Based on the theory of the ECAE process [10] under ideal conditions, deformation is confined to thin layers at the crossing plane of channels to obtain simple shear deformation in the material.

In the current study with $\psi = 0^\circ$ under ideal conditions (Fig. 3.12) the deformation zone is narrow and close to simple shear. Whereas with an increase in outer corner angle, $\psi = 10^\circ$ & 20° , the deformation zone splits into two near the outer corner and created a region of no plastic deformation in between the split portion. This will be

the cause for non-uniformity in strain distribution with higher outer corner angles. Similar behavior is observed under ideal conditions with larger outer corner angles [75].

In current study under realistic conditions the shapes of deformation zones obtained are quite different from the earlier studies [10, 54, 57, 75]. With $\psi = 0^\circ$ (Fig. 3.13) the deformation zone is very wide and not in the form of an arc, the behavior is actually nearer to the theoretical predictions (clearly observable with deformed mesh in Fig. 3.7). The deformation zones under realistic conditions with $\psi = 10^\circ$ & 20° are relatively narrow compared to ideal conditions. In addition, with $\psi = 10^\circ$ no splitting of the plastic zone is observed near the outer corner as in ideal conditions. Accordingly, the strain distribution is quite uniform under realistic conditions compared to ideal conditions with $\psi = 10^\circ$. This shows that the material behavior and influence of friction will varies the shape of deformation zone. The similar phenomenon being observed in earlier studies [75].

Figure 3.12 Von Mises Stress/Effective stress (VMS) contours in Pascal of the samples simulated with different ECAE tool angles $\phi = 90^\circ$ and (a) $\psi = 0^\circ$, (b) $\psi = 10^\circ$, (c) $\psi = 20^\circ$ under ideal conditions ($n=0$, $\mu=0$) in steady state deformation zone.

Figure 3.13 Von Mises Stress/Effective stress (VMS) contours in Pascal of the samples simulated with different ECAE tool angles $\phi = 90^\circ$ and (a) $\psi = 0^\circ$, (b) $\psi = 10^\circ$, (c) $\psi = 20^\circ$ under realistic conditions ($n=0.304$, $\mu=0.05$) in steady state deformation zone.

From the above discussion one can conclude that smaller outer corner angle yields a smaller deformation zone. It also shows that the parameters like friction, strain hardening behavior of material and tool angles influence the shape of the deformation zone, all of which in turn affect the deformation behavior and strain homogeneity of the deformed samples.

3.5.3.3 Equivalent Plastic Strain/Effective Strain

The strain homogeneity and the amount of strain induced in the deformed samples were directly obtained from the effective strain (PEEQ) contours. Those are shown in Figs. 3.8 & 3.9 for different outer corner angles under ideal and realistic conditions. The PEEQ contours are shown at three different stages of deformation i.e. at 10%, 60%, and at the end of the deformation process to study the variation of deformation behavior and strain homogeneity in the entire deformation process.

Under ideal conditions with $\psi = 0^\circ$ (in Fig. 3.8) shear deformation concentrates perfectly in a diagonal plane encompasses by two corners (inner and outer), at all stages of deformation. Thus, the strain distribution is more uniform across the width of the sample, and the deformation behavior is similar to simple shear [10]. Similar

behavior is observed in most of the earlier studies [52, 53, 75]. With $\psi = 10^\circ$ & 20° (in Fig. 3.8) the shear deformation is also concentrated in the diagonal of two corners, but the plane is perfectly diagonal up to 85 & 80% of the diagonal respectively. A slight bend is observable in the remaining part of diagonal near the outer corner. This bend increases with increase in outer corner angle, thus the strain values along the outer corner are different from the inner corner and centre of the sample. Thus non-uniformity in strain distribution is observable with increase in outer corner angle. Similar behavior is observed at all stages of deformation. The difference of strain values increases with larger outer corner angles [52, 53, 75].

In realistic conditions the diagonal plane of shear deformation varied at all stages of deformation with all outer corner angles as observed in Fig. 3.9. With $\psi = 0^\circ$ the diagonal plane is perfect up to 72%, the remaining part is bent. Thus the effective strains obtained were uniform across 72% of the width of the sample while rest of the width was subjected to higher strains. In practice heavy stresses are needed to generate such strains that can cause damage to the die surface and score the surface of the deformed samples. In addition, it increases the load requirements. With $\psi = 10^\circ$ & 20° (in Fig. 3.9), the diagonal plane is perfect up to 85 and 72% respectively. The effective strain achieved with $\psi = 10^\circ$ was uniform across 85% of the width of the sample, the remaining 15% being subjected to non-uniform deformation. With $\psi = 20^\circ$ the effective strains were uniform across 72% of the width. However, the range of the strain variation in $\psi = 10^\circ$ was not as high as in the case of $\psi = 20^\circ$. This shows that strain values are more uniform with $\psi = 10^\circ$ compared to $\psi = 0^\circ$ & 20° under realistic conditions.

To observe the strain homogeneity in an easier way the PEEQ variation across width A-B (shown in Fig. 3.1), at the centre of the sample was plotted for both ideal and realistic cases with all outer corner angles in Fig. 3.14. PEEQ variation is more uniform across the width with $\psi = 0^\circ$ under ideal conditions, whereas it was uniform up to 72% of the width under realistic conditions. The remaining 28% width was subjected to very high strains. The range of difference in strain values between uniform and non-uniform regions are relatively large. It therefore shows the non-uniformity in strain distribution is larger with sharp outer corner angle under realistic conditions. With $\psi = 20^\circ$ under ideal conditions the PEEQ variation

is uniform upto 80% of the width of the sample, whereas in realistic case it is uniform up to 72% of the width. In both cases the strain values are lower in the remaining portions of the width. With $\psi = 10^\circ$, PEEQ variation is uniform across the 85% of the width under both ideal and realistic conditions. The remaining 15% width is subjected to lower strain values in both cases. However, the strain values in this region under realistic conditions are slightly higher compared to ideal conditions. This higher strain values with realistic conditions are due to friction. Friction reduces the corner gap formation when high strains were achieved in that region. In addition the range of strain variation is also very small comparing $\psi = 0$ and 20° .

Figure 3.14 PEEQ variations across the width A-B of the deformed samples under both ideal and realistic conditions for different outer corner angles with channel angle of 90° . (PSI indicates the outer corner angle ' ψ ').

This shows that the strain homogeneity is more of a possibility with $\psi = 10^\circ$ under both ideal and realistic conditions. Hence, it can be deduced that $\psi = 10^\circ$ is the best angle to obtain optimum strain homogeneity in the deformed samples.

3.5.3.4 Peak Punch Force and Maximum Equivalent Plastic Strain

Fig. 3.15 shows the maximum equivalent plastic strain achieved and peak punch force needed for ECAE under both ideal and realistic conditions with different outer

corner angles. Peak punch force needed of extrusion is a prime factor to be considered for design of sound ECAE die.

In ideal case peak punch force decreased gradually from $\psi = 0$ to 20° . But the maximum PEEQ achieved is almost equal with $\psi = 0$ & 10° , but lower with $\psi = 20^\circ$. In addition the non-uniformity of strain distribution is also high with $\psi = 20^\circ$. The force reduction is mainly due to the easy flow of the sample with increase in outer corner angle, and lack of friction.

In realistic case the peak punch force needed for ECAE in the following descending order of $\psi = 0, 20$, and 10° . But the maximum PEEQ decreased with increase of outer corner angle.

Figure 3.15 Peak punch force and maximum PEEQ under both ideal and realistic conditions for different outer corner angles with channel angle of 90° .

With $\psi = 0^\circ$ peak punch force and max PEEQ are high. This amount of higher force is achieved mainly due to generation of high strains in the sample, which are due to lack of free flow of the sample caused by sharp outer corner angle and friction. High non-uniformity in strain distribution and high punch force requirement are due to $\psi = 0$ & 20° in realistic case. Therefore, with $\psi = 10^\circ$, the punch force requirement will be lower and strain distribution more uniform in realistic case.

Thus the best combination of lower peak punch force and maximum PEEQ is possible with $\psi = 10^\circ$ under both ideal and realistic cases.

The simulations conducted in this section for different outer corner angles with channel angle of 90° under both ideal and realistic conditions showed that the outer corner angle, $\psi = 10^\circ$ is optimal for achieving strain homogeneity, maximum PEEQ, and low punch force requirements by avoiding the detrimental effects. In addition the shape of the front end of the deformed samples are also relatively uniform with $\psi = 10^\circ$.

3.5.4 Influence of Inner Corner Radius

The sharp inner corner, shown in Fig. 3.16, of the ECAE die causes severe stress concentration and thus can damage the surface of samples during processing. In addition, FE analysis cannot be completed due to the penetration of the sample into the rigid die in the simulations as shown in Fig. 3.17. To avoid this penetration near inner corner, it is necessary to use finite fillet radius in all the simulations. However, the choice of fillet radius should not vary the deformation behavior and strain homogeneity of the deformed materials. As discussed earlier, FE simulations under ideal and realistic conditions showed that tool angles $\phi = 90^\circ$ & $\psi = 10^\circ$ develop optimal strain homogeneity in the deformed materials. Accordingly, optimal fillet radius selection simulations were performed with the same tool angles under ideal conditions. The inner corner radii, $r = 0.05W$, $0.1W$, and $0.15W$ (where 'W' is the width of the sample in mm) were used for the simulations.

Figure 3.16 Location of inner corner radius (r) of ECAE dies.

Fig. 3.18 shows the Von Mises stress/effective stress (VMS) contours in the deformation zone with different inner corner radii. These contours were shown when deformation achieved steady state (at $\sim 60\%$ of deformation). When $r = 0.05W$, the deformation zone is quite narrow and is nearer to simple shear behavior. With $r = 0.1W$ and $0.15W$, the deformation zone would split into two regions and expand. In addition the dead zone formation is increased with increase of inner corner radius. All these effects will cause the deformation to deviate from simple shear, introducing non-uniformity in strain distribution.

Figure 3.17 Simulation uncompleted for inner corner radius, $r = 0$.

Fig. 3.19 shows the PEEQ contours obtained with different inner corner radii at the end of deformation. With an increase in the inner corner radius, the effective strain near the inner corner increases as compared to the region near the outer corner, and it developed non-uniformity in strain distribution. To obtain the strain homogeneity in quantitative terms PEEQ variation across the width A-B (shown in Fig. 3.1) was plotted in Fig. 3.20. With an increase in inner corner radius, the effective strain increases in the upper 70% of the width of the sample. The remaining 30% is not subjected to the influence of inner corner radius. This shows that the inner corner radius will influence the strain distribution only in the upper 70% width of the sample. When $r = 0.05W$, the strain variation is close to theoretical calculations, whereas with high 'r' the strain difference from the theoretical and simulated strain values is quite significant. Thus the results show that lower inner corner radius does not vary the strain homogeneity achieved with optimal tool angles.

Figure 3.18 PEEQ variations across the width A-B of the deformed samples under both ideal and realistic conditions for different outer corner angles with channel angle of 90°. (PSI indicates the outer corner angle ' ψ ').

Figure 3.19 Equivalent Plastic Strain (PEEQ) contours of the samples simulated with different inner corner radii (a) $r = 0.05W$, (b) $r = 0.1W$, and (c) $r = 0.15W$ (where, W = width of the billet) ECAE dies at the end of deformation.

Figure 3.20 PEEQ variations across the width A-B of the deformed samples for different inner corner radii ECAE dies.

Fig. 3.21 shows the punch force-displacement curves for different inner corner radii. The punch force curves trend is almost similar irrespective of the inner corner radius. However, the punch force needed for extrusion is increased with increase in

Figure 3.21 Force-displacements curves with different inner corner radii ECAE dies.

inner corner radius. As the inner corner radius increases, the flow of the sample becomes complicated whereas the force required for extrusion increases.

Fig. 3.22 shows the influence of inner corner radius on peak punch force and maximum PEEQ. With an increase in inner corner radius, the peak punch force, and maximum PEEQ would increase. However, the increase of PEEQ is not uniform across the width of the sample. Hence the result shows that lower inner corner radius generates uniform simple shear deformation behavior, with strain homogeneity.

Figure 3.22 Peak punch force and maximum PEEQ for different inner corner radii ECAE dies.

3.5.5 Influence of channel angle

Channel angle is also one of the most important parameters which can vary the strain homogeneity in ECAE. But the amount of strain induced is mainly dependent on the channel angle. In practice different channel angles from 60 to 160° were used to produce ultrafine grain microstructures in different materials [45, 121] based on the material strength and ductility. In section 3.5.3, FE simulations under ideal and realistic conditions showed that the tool angles $\phi = 90^\circ$ & $\psi = 10^\circ$ develops strain homogeneity in the deformed materials by avoiding the detrimental

effects. The same tool angles also showed the best combination of lower peak punch force and maximum PEEQ. It is important to know how the deformation behavior, strain homogeneity varies with other channel angles compared to channel angle of 90° before designing the die. Simulations were carried for a range of channel angles from 60° to 150° by using constant outer corner angle of 10° under realistic conditions.

To observe the deformation behavior and strain homogeneity during the ECAE, three locations of interests: P1, P2 and P3 (Fig. 3.23) were chosen across the sample width at the middle of the sample. When the sample is in the deformation zone, P1 will be nearer to the outer corner of the die, P3 will be nearer to inner corner of the die, and P2 will be at the centre of the sample width.

Figure 3.23 ECAE die with $\phi = 90^\circ$, $\psi = 10^\circ$, and points P1, P2, P3 are the locations of interest for strain homogeneity studies.

3.5.5.1 Deformation Behavior

Deformation behavior during the ECAE can be extracted from the equivalent plastic strain/effective strain (PEEQ) distributions during the total deformation step. Fig.3.24 shows the PEEQ at the three points (P1, P2, and P3) against time (t) for different channel angles. When $t = 0$, load was applied; sample enters the deformation zone at $t \sim 80$; and at $t \sim 95$ it exits from the deformation zone. For $\phi <$

Figure 3.24 Equivalent plastic strain/effective strain (PEEQ) at different points (P1, P2, P3) during the ECAE deformation with different channel angles (a) $\phi = 60^\circ$, (b) $\phi = 75^\circ$, (c) $\phi = 90^\circ$, (d) $\phi = 105^\circ$, (e) $\phi = 120^\circ$, (f) $\phi = 135^\circ$, and (g) $\phi = 150^\circ$. (In graphs Phi indicates the channel angle ' ϕ ').

90° the deformation takes place in three steps (shown in Fig. 3.25), whereas for $\phi \geq 90^\circ$, the deformation takes place in two steps (shown in Figs. 3.26 & 3.27). In the case of $\phi < 90^\circ$, the sample sticks to the bottom of the die, fills the entire die, and only then the deformation starts, which is observed in Fig. 3.24. With $\phi \geq 90^\circ$, initially the sample sticks to the die and deformation starts immediately thereafter. Even with different channel angles, start and end timings of deformation at the three points are almost comparable. However, this is quite different from earlier reported results [63, 64], and it is postulated that it could be due to the selection of lower outer corner angle in the current study. This shows that there is this possibility of reducing the delay in starting and ending of the deformation with lower outer corner angles. Due to this, the differences in strains achieved at different points are lower.

Figure 3.25 Deformation steps with tool angles $\phi = 60^\circ$ & $\psi = 10^\circ$ (a) step 1 – sample sticks to the bottom of the die (b) step 2 – sample fills the entire deformation zone, and (c) step 3 – deformation proceeds.

Figure 3.26 Deformation steps with tool angles $\phi = 90^\circ$ & $\psi = 10^\circ$ (a) step 1 – sample sticks to the bottom of the die, and (b) step 2 – deformation proceeds.

Figure 3.27 Deformation steps with tool angles $\phi = 105^\circ$ & $\psi = 10^\circ$ (a) step 1 – sample sticks to the bottom of the die, and (b) step 2 - deformation proceeds.

Corner gap/dead zone formation is the major factor for non-uniform strain distribution in ECAE of strain hardening materials with $\phi \geq 90^\circ$ (observable in Figs. 3.26 & 3.27), and the same issue is discussed in most of the earlier FE studies of ECAE [57, 62, 75]. To prevent this, researchers had to opt for high friction coefficients, and the introduction of outer corner angle. But this dead zone problem was easily overcome by acute channel angles (observable in Fig. 3.25). In addition, when $\phi = 90^\circ$, the corner gap formed is very small (observable in Figs. 3.26) due to the lower outer corner angle and the influence of friction.

3.5.5.2 Equivalent Plastic Strain/Effective Strain

Table 3.3 shows the theoretical [8 - 10] (neglecting the influence of friction and strain hardening), and finite element simulated (considering the influence of friction and strain hardening) effective strain values for different channel angles at different points. For $\phi = 60^\circ$ & 75° , strain achieved at point P1 (along the outer corner of the die) is quite high compared to the points P2 and P3 (at the centre and inner corner of the die). For the case of $\phi = 90^\circ$ & 150° , strain achieved is maximum at point P2 compared to points P1 & P3. For the case of $\phi = 105^\circ$, 120° and 135° , strain achieved is maximum at point P3, compared to points P1 & P2. The difference between the strains achieved at different points is high for $\phi = 60^\circ$, and low for $\phi = 90^\circ$. To get uniform properties in the deformed materials, strain homogeneity is preferable. Strain homogeneity is unattainable in the ECAE of strain hardening materials while the presence of friction further causes non-uniform strain distribution. Hence, the minimum differences in strain values (at three points) are preferable which is possible with $\phi = 90^\circ$ as shown by current results. With lower channel angles $\phi < 90^\circ$, the higher strains achieved at point P1 are due to the inability of the material to flow freely due to die geometry, and absence of a dead zone. With higher channel angles, $\phi \geq 90^\circ$, the lower strains achieved at point P1 are due to the development of a small dead zone.

3.5.5.3 Punch Force

Fig. 3.28 shows the punch force-displacement curves obtained for different channel angles. These curves have some fluctuations, which are unavoidable in explicit analysis. Peak punch force indicates the force required for the start of extrusion, and

Table 3.3 Theoretical [8 - 10] and FE simulation strain values for different channel angles at different points.

Channel angle (ϕ) in deg	Effective strain (ϵ) as per [2,5]	Effective strain (ϵ) at P1	Effective strain (ϵ) at P2	Effective strain (ϵ) at P3
60	1.82	4.38	1.47	1.55
75	1.41	2.52	1.29	1.37
90	1.09	0.98	1.13	1.07
105	0.98	0.64	0.88	0.89
120	0.65	0.49	0.67	0.71
135	0.59	0.30	0.47	0.59
150	0.30	0.23	0.30	0.29

Figure 3.28 Punch pressure-displacement curves with different ECAE channel angles. (Phi in figure indicates the channel angle ' ϕ ').

is the most important factor to be considered in designing a sound ECAE die. With the increase in channel angle, flow of the sample becomes easier and the force required for the extrusion decreases. The deformation steps can also be correlated with these force-stroke curves. For the case of $\phi < 90^\circ$ the persistence of peak force, across certain displacement, is due to the filling of the sample in the die before the onset of extrusion. While, for $\phi \geq 90^\circ$, the extrusion is accompanied by a reduction in punch force gradually. For the case of $\phi = 135^\circ$ & 150° the peak force was observed at a displacement of 20mm during the extrusion.

From a broader perspective, it can be understood that the deformation is more complicated with acute channel angles compared to $\phi \geq 90^\circ$, and so are the force requirements. The deformation took place in three steps for acute channel angles, $\phi = 60^\circ$ and 75° , in comparison to only two steps for $\phi \geq 90^\circ$. Formation of corner gap, which is often the major drawback in the case of $\phi \geq 90^\circ$ channel angle, was not encountered for the acute channel angles. With acute channel angles, the higher strains along the outer corner angle would not damage the die surface due to the compressive nature of the stresses. There is a possibility to reduce these high strains by increasing the outer corner angle. However, an increase in the outer corner angle would reduced strain values and strain heterogeneity. With $\phi = 90^\circ$ differences between the effective strain values at different points are minimal compared to all other channel angles. Thus the results shows that strain homogeneity in ECAE is possible with $\phi = 90^\circ$.

3.5.6 Influence of Processing Route

FE simulations in earlier sections 3.5.3 & 3.5.5 showed that the tool angles $\phi = 90^\circ$, $\psi = 10^\circ$ will develops strain homogeneity in the deformed materials. In practice, enhanced microstructure and mechanical properties of the deformed materials is possible through the severe strains induced by multiple passes via different routes of ECAE [46]. Thus, it is important to know how the strain homogeneity varies with different processing routes. Simulations were carried for two processing routes: Route A & Route C, with tool angles $\phi = 90^\circ$, $\psi = 10^\circ$ under realistic conditions. A sample length of 80mm was considered for these simulations.

3.5.6.1 Deformation Behavior through different routes

The FE models used for multipass ECAE simulations by Routes A & C with sample-deformed mesh was shown in Fig. 3.29. The die geometry shown in Fig. 3.29 has two turns in two directions to accommodate the two passes through two different routes. The sample moved along the first turn of the die indicating the completion of Pass 1. The same sample takes the second turn of the die as it entered into Pass 2. During the second turn it is possible to define two routes, Route A (no rotation of sample after each pass) and Route C (involves 180° rotation of sample after each pass), as shown respectively in Figs. 3.29b & d. Figs. 3.29a & c show part of the sample moved along the first turn of die after completing Pass 1 of ECAE.

Figure 3.29 FE models used for multipass ECAE simulations. The deformed mesh of the samples after first turn indicates the completion of first pass and after second turn indicates the completion of second pass. Deformed mesh after (a) Pass 1 of Route A dies, (b) Pass 1 and Pass 2 of Route A dies, (c) Pass 1 of Route C dies, and (d) Pass1 and Pass 2 of Route C dies.

3.5.6.2 Equivalent Plastic strain/Effective Strain

Fig. 3.30 shows the equivalent plastic strain/effective strain contours obtained with different routes in multipass ECAE simulations. Fig. 3.30a & b shows the strain contours after Pass 1, whereas Figs. 3.30c & d show the strain contours after multiple passes, i.e., Pass 1 and Pass 2 with Route A, Pass1 and Pass2 with Route C respectively. The main differences between these two ECAE multi passes as observed from Fig. 3.30 are corner gap formation and strain homogeneity

(uniformity of strain distribution). The corner gap formed during Pass 1 of ECAE is observable in Fig. 3.30a. It generates non-uniform strain distribution along the outer side of the sample which is unavoidable with strain hardening materials [57]. The corner gap, observed during the first pass in current study, is lower [81] due to the increase of outer corner angle to 10° and also the influence of friction between the die and sample. When the sample enters Pass 2, the corner gap disappeared near the second turn due to the influence of the outer corner angle, friction, die geometry and reduced strain hardening rate of the material. Once the sample crosses the second turn the corner gap near the first turn also disappeared due to the creation of backpressure caused by the die geometry. The effect of backpressure creates a state of hydrostatic stress in the deformation zone which will be very much useful for ECAE of low ductility materials.

Figure 3.30 Equivalent Plastic Strain (PEEQ) contours obtained from (a) & (b) Pass1, (c) Pass1 & Pass 2A , (d) Pass1 & Pass 2C of ECAE.

Fig. 3.31 shows the variation of effective strain across the width A-B (from Fig. 3.30) for different passes of the sample. The effect of corner gap formed during the Pass 1 is clearly observable in Fig. 3.31 in the form of low strain values nearer to width 'B' of the sample while the same effect was not observable in Passes 2A & 2C due to the disappearance of the corner gap. In Pass 2A the effective strain across the width A-B is almost uniform, but in Pass 2C it is high for 60% of the width of the sample. These high strain values in Pass 2C are due to the change in the shear direction and the effect of friction. The deformation behaviour is thus more severe and non-uniform in Route C compared to Route A. Non-uniform deformation was also observed with Route C in earlier studies [68, 80, 81] with different ECAE tool angles. Fig. 3.31 also shows that in Pass 2A the effective strain values were twice of Pass 1 as per Eq.2.4 whereas the strain values of Pass 2C were very much higher as compared to Pass 1. This shows that the smooth deformation behaviour and strain homogeneity during multipass ECAE of the strain hardening materials is possible with Route A compared to Route C. Thus, the results infer that Route A generates more uniform properties in the material than Route C.

Figure 3.31 Equivalent Plastic Strain (PEEQ) variation for different ECAE passes under ideal and realistic conditions.

3.5.6.3 Punch Force

Fig. 3.32 shows the punch force - displacement curves of two passes via two routes of ECAE. The force displacement curves shown in Fig. 3.32 have two peaks, first peak indicates the force requirement for first pass, and the second peak indicates the force requirement for second pass. Force-displacement curves trends are similar for both processing routes of ECAE and the force requirement during the second pass is about two times compared to the first pass via both routes. The increase of load during the second pass is due to work hardening of the materials after first pass. The similar behavior is observed in earlier studies [80, 81]. The peak punch force is slightly higher in Route A compared to Route C. The lower peak punch force recorded in Route C is due to change of shear direction.

Figure 3.32 Force – displacement curves for different ECAE passes.

Finite element simulations of multipass ECAE with Route A and Route C showed that smooth and un interrupted deformation behaviour is possible with Route A. Strain homogeneity of the samples can be achieved with Route A compared to Route C. The peak punch force is also slight higher with Route A compared to Route C.

CHAPTER 4

MATERIALS AND METHODS

4.1 DESIGN OF ECAE DIE AND ESTABLISHMENT OF ECAE SET UP

To process the materials through ECAE, a die is required. As the research is first of its kind in this lab, establishing the ECAE set up (design of the die, punch and clamps) is a major part of this research. In ECAE die design the most important parameters are the tool angles: die/channel angle ' ϕ ', outer corner angle ' ψ ', and the thickness of die. Optimal tool angles which can generate strain homogeneity in ECAE were found from the finite element studies of ECAE discussed in the earlier chapter. Accordingly, ECAE die (shown in Fig. 4.1) is fabricated with the tool angles: $\phi = 90^\circ$ & $\psi = 10^\circ$. In addition the stresses required for calculation of die thickness were also obtained from the FE simulations of ECAE. Sample/Billet diameter and length are restricted to 10 and 60 mm respectively due to the limitations of maximum loading capacity of the machine. The entry channel length of 70 mm was considered for die fabrication. For easy removal of the deformed billet design was made of split configuration. Dowel pins (shown in Fig. 4.1) are located in one piece of the split portion die, for easy assembling. ECAE die and sub-press equipment (punch & clamps) drawings were prepared by AutoCAD, and these are fabricated by external vendors with H11 heat treated tool steel. The chemi-

Figure 4.1 ECAE split dies fabricated with tool angles $\phi = 90^\circ$ & $\psi = 10^\circ$.

-cal composition of H11 tool steel is shown in Table 4.1, and the die is hardened and tempered to HRC 63. Split pieces of ECAE die is hold in position by seven set screws. ECAE die and its setup is shown in Figs. 4.1 to 4.3.

Table 4.1 Chemical composition of H11 Tool steel.

Element	C	Mn	Si	Cr	V	Mo	Others
Composition (by weight %)	0.37	0.4	1.0	5.0	0.4	1.3	91.53

Figure 4.2 (a) ECAE die clamped between the rams of Instron machine at the start of extrusion (b) Close view of ECAE die setup at the end of extrusion.

Figure 4.3 ECAE experimental setup on Instron machine along with data acquisition system.

4.2 SELECTION OF MATERIALS

4.2.1 Magnesium Boride (MgB_2)

MgB_2 is selected for current study. MgB_2 is a brittle intermetallic compound available in powder form and becomes superconductive at a temperature of 39 K. It has wide applications in superconducting field. Commercially available MgB_2 powder was purchased from Alfa Aesar has hexagonal crystallinity. Powder has wide range of particles, 0.3 to 70 μm , with no definite shape.

Properties of MgB_2 :

- HCP crystal structure
- Low material density (theoretical density = 2.625 g/cc)
- High temperature metallic superconductor
- Large coherence length
- High critical current density (J_C)
- Low anisotropy
- Low cost.

4.2.2 Tube Materials

To process MgB_2 through ECAE, powder in tube (PIT) technique will be used. Thus, the selection of the tube material is very important, and it should not react with the powder while working above room temperatures. As per literature pure iron and its alloys does not react with MgB_2 even when subjected to high temperature sintering [115-118, 120-126]. However in current study three different tubes: Low carbon steel (LCS), Pure Iron, and Pure copper are used to process the MgB_2 through ECAE.

4.2.2.1 Low Carbon Steel

Low carbon steel (LCS) AISI 1015 rods of 12.7mm diameter and lengths of 1000 mm are provided by Nat Steel, Singapore. The composition of LCS is given in the Table 4.2.

Properties of Low carbon steel (AISI 1015):

- BCC crystal structure
- High ductility and high tensile strength
- Low cost

Table 4.2 Chemical composition of LCS.

Element	C	Si	S	P	Mn	Others	Fe
Composition (by weight %)	0.16	0.138	0.027	0.023	0.45	0.873	98.329

4.2.2.2 Pure Iron

Iron (Fe) tubes of 99.5% purity with an outer diameter = 10mm, inner diameter = 7 mm, and lengths of 1000 mm are purchased from Good fellow Cambridge Limited.

Properties of Pure Iron:

- BCC crystal structure
- High ductility and medium tensile strength
- High cost

4.2.2.3 Pure Copper

Copper (Cu) rods of 98% purity with diameter = 10 mm, and lengths of 1000 mm are purchased from local vendors.

Properties of Pure Copper:

- FCC crystal structure
- High ductility and low tensile strength
- Low cost

4.3 PREPARATION OF POWDER IN TUBES

4.3.1 Preparation of Tubes & Plugs

LCS, Cu rods, and Fe tubes are sliced into lengths of 65 mm and subjected to annealing in a vacuum tube furnace. The time and temperature used for vacuum annealing of different materials is shown in Table 4.3. After annealing, tubes and plugs (front and rear) are prepared from the rods by turning and drilling in a lathe. Sharp edges of the tubes and plugs are grinded by using bench grinder. Plugs made from LCS rod are used for sealing the ends of Fe tube.

Table 4.3 Annealing conditions for Tube Materials.

Materials	Annealing Conditions	
	Temperature (°C)	Time (hrs)
LCS	950	4
Fe	600	1
Cu	600	2

Tubes of outer diameter $\phi 9.9$ mm and inner diameter of 7 mm are prepared from LCS and Cu rods by using drill bits of 2, 5 and 7 mm diameter, and drilled to a depth of 45 mm. Front and rear plugs of 7 mm diameter, and lengths of 20 & 10 mm respectively are prepared from the rods. Diagrams of the tubes (along with

dimensions) sealed by the front plug, and prepared from the solid rod are shown in Figs. 4.4 & 4.5. The dimensions of the tube & plugs are decided based on the initial trail runs with different tube thicknesses and front plug lengths.

Figure 4.4 Tube sealed by front plug (All dimensions are in mm).

Figure 4.5 Tube prepared from the solid rod (All dimensions are in mm).

4.3.2 Powder Packing

The tube, sealed with both front and rear plugs are subjected to ultrasonic cleaning with acetone before powders are packed into them. Packing die and punches, fabricated in-house, are used to fill the powder into tubes. The set-up used for powder packing is shown in Fig. 4.6. Powder is packed into the tubes layer by layer by pressing with a punch under ambient conditions upto a depth of 35 mm. After that the other end of the tube is sealed by rear plug.

Figure 4.6 Schematic diagram shows the Packing of Powder into the tube by using Packing Die & Punch.

4.4 ECAE PROCESSING OF POWDER IN TUBES

4.4.1 Single Pass ECAE

In single pass ECAE, powder filled tubes are extruded once through ECAE die.

Single pass ECAE is conducted according to the following steps:

- The die channels (Fig. 4.1) are coated with molybdenum disulfide (MoS_2) lubricant and the die clamped by set screws.
- The powder filled tube is also coated with MoS_2 , and is placed in the entry-channel of ECAE die.
- The ECAE die, along with powder filled tube, is kept on the bottom clamp, which was hold by bottom ram of Instron machine.
- The Punch, coated with MoS_2 , is placed, on the top of the powder filled tube, in the entry-channel of ECAE die.
- Top clamp is placed on the top of the punch, and it is hold by the top ram.

- ECAE die, along with powder filled tube and punch, is hold in between the two rams of the Instron 8516 servo hydraulic tester as shown in Fig. 4.2. This Instron machine has a load cell capacity of 100 kN.
- Load is applied through the bottom ram with a speed of 2 mm/min. Thus, the entire die set is moved against the punch. During loading, the load-displacement curve is recorded by Series IX data acquisition system connected to the Instron through a personal computer as shown in Fig. 4.3. Loading is continued upto 50 mm displacement. The remaining 10 mm length of the tube is extruded by placing another solid dummy sample of 10 mm length into the entry-channel of the die. At the end of 60 mm displacement, the entire tube will be extruded into the exit-channel of the die.
- The extruded tube was taken out from the exit-channel by un-screwing the seven set screws of ECAE die.

4.4.2 Multipass ECAE

In case of multipass ECAE, powder filled tubes are processed a number of times through the same ECAE die. During the multiple passes the tubes are rotated around its longitudinal axis between the adjacent passes, creating different routes of ECAE. Accordingly, tubes are processed through four basic processing routes: Route A (no rotation), Route C (180° rotation), Route B_A (90° clockwise and counter clockwise rotation) and Route B_C (90° clockwise rotation) in between the passes. The four basic processing routes are shown in Fig. 2.1.

Steps similar to single pass ECAE are followed in case of multipass ECAE. In addition, the extruded tube outer diameter is slightly turned before the next ECAE pass, for easy entry into the entry-channel of the ECAE die. In between the passes, tubes are subjected to stress relief annealing to restore the ductility of the tube material. The stress relief annealing is carried in vacuum at 700 °C for 25 minutes for LCS, and 500 °C for 30 minutes for pure iron tubes.

4.5 CHARACTERIZATION OF MgB₂ POWDER AND ITS COMPACTS

As received MgB₂ powder is characterized to determine the shape and size of the powder particles. The powder filled tubes before and after ECAE are subjected to

X-Ray image studies to analyze the powder distribution in the tubes during ECAE. The MgB_2 compacts are sliced from the ECAE processed tubes, and used for characterization of microstructure and deformation properties.

4.5.1 Particle Analyzer

Fritsch Particle Sizer ANALYSTTE 22 was used to measure the particle size of commercially purchased MgB_2 powder. A total of three runs were conducted for each sample.

4.5.2 X-Ray Images

ANDREX Portable X-Ray unit (control unit: SMART 583, X-Ray tube head: SMART 225) shown in Fig. 4.7 is used to obtain the X-Ray images of the powder filled tubes before and after ECAE. The powder filled tubes are kept below the tubular X-Ray unit on top of the dark enveloped film, over the lead sheet. The voltage and current needed for exposure was decided based on number of initial trials from 70 – 120 V by keeping a constant current of 3 mA. Exposure time of 1 min is used for all the tubes. After X-Ray exposure, films are developed in the dark

Figure 4.7 X-Ray unit used for taking the X-Ray Images of Powder Filled Tubes.

room by using standard film developing procedure. Images of the developed films are observed by placing on the top of the light source. Digital pictures were captured from the observed images.

4.5.3 X-Ray Diffraction (XRD)

SHIMADZU X-Ray Diffractometer Lab XRD-6000 of Max: 50 KV, 50 mA was used for X-Ray diffraction studies. As received MgB_2 powder and its compacts after ECAE were subjected to XRD studies to determine phase constitution. The location of the compact used for XRD studies was shown in Fig. 4.8. This compact is sliced in the transverse plane (plane perpendicular to the length of the tube). All scans are conducted with a scan speed of 0.2 deg/min. The following measurement conditions: copper target, 40 KV, 30 mA, divergence and scatter slit of 1° , and receiving slit of 0.3 mm are used for all the scans.

4.5.4 Microstructure

Microstructural studies are carried in the flow and transverse planes of the MgB_2 compacts by JSM-5410LV Scanning Microscope, and JSM-6340F Field Emission Scanning Electron Microscope (FESEM) respectively. Flow plane is the plane parallel to the extrusion direction, and transverse plane is the plane perpendicular to the length of the tube. Locations of the compacts used for transverse and flow plane studies are shown in Figs. 4.8 & 4.9. As received MgB_2 powder particles shape is also characterized by using the above microscopes.

For transverse plane studies, MgB_2 compact is sliced from the canning material (Fig. 4.8) by using the ISOMET low speed precision cutter. The sliced MgB_2 compact is not subjected to any grinding and polishing steps (inorder to identify the voids and cracks, particle size, and agglomerates) and used for microstructural studies.

For flow plane studies the MgB_2 compact, along with tube, is sliced across the flow plane (Fig. 4.9) by using the same precision cutter. The sliced portion is subjected to grinding with 180, 300, 800, 1000 grit papers. Polishing was carried on rough, and fine polishing clothes without using the diamond paste. The polished flow plane cross-section is used to observe the shear sliding and microstructure.

Figure 4.8 Location of the Transverse Plane MgB₂ compact sliced used for Density, XRD and Microstructural studies by removing the surrounding tube.

Figure 4.9 Location of the flow Plane MgB₂ compact sliced along with tube, and used for Hardness, and Microstructural Studies.

4.5.5 Density

MIRAGE electronic densimeter (MD-200S) shown in Fig. 4.10 is used to measure the density of the compacts. It has a density resolution of 0.001, and the equipment capacity is 200grams. It works based on the Archimedes principle.

Density measurements are carried on the compact sliced in the transverse plane of the tube. The location of the MgB₂ compact used for density measurements is shown in Fig. 4.8. Measurements were carried by removing the surrounding tube.

Location and thickness of the compacts sliced for density measurements is maintained uniform for all the compacts.

Sample under Measurement

Figure 4.10 Electronic Densimeter used for measuring the density of the compacts.

4.5.6 Deformation Properties

4.5.6.1 Hardness

The schematic of the Nanotester [144] (Macro Materials, UK) used for hardness measurements is shown in Fig. 4.11. This instrument has a facility to indent for a range of loads: Nanotest: 10 to 500 mN and Micro test: 2 to 20N. Both of these tests can be carried out with the options of depth controlled indentation, load controlled indentation, and multiple indentations with increasing load. This instrument gives the load-displacement data throughout the indentation (both loading and unloading) process. From this data, the need of imaging to determine the contact radius is obviated and mechanical properties can be investigated. Hardness and modulus of the compacts can be calculated from the loading curve and unloading curves respectively.

Hardness is calculated according to the following equation:

$$H = \frac{P_{\max}}{A} \quad (4.1)$$

where, $P_{\max}$ is the peak indentation load and A is the projected area of the hardness impression.

$$A = 24.56 h_p^2 \quad (4.2)$$

where, h_p is the plastic depth

Figure 4.11 Schematic of the Nano Tester used for depth-sensing indentation measurements [144].

Hardness profiles are taken in both the transverse and flow plane sections of the compacts shown in Fig. 4.12. These compacts are grinded and polished before subjected to hardness measurements. The flow plane hardness profile is taken across the width A-B (Location 'A' is nearer to inner corner of the die, and the Location 'B' is nearer to the outer corner of the die). The transverse plane hardness profiles are taken in two directions: X-X and Y-Y. In X-X direction, indentations are taken from left to right of the compact. In Y-Y direction, indentations are taken from top to bottom of the compact. Top of the compact is the location nearer to the inner corner, and the bottom of the compact is the location nearer to the outer corner of the ECAE die. Table 4.4 shows the conditions of hardness measurements. Berkovich indenter is used for current studies.

Figure 4.12 Flow and Transverse Plane cross-sections of the MgB_2 compacts, and directions used for hardness studies.

Table 4.4 Conditions for hardness measurements.

Indentation Mode	Load controlled
Maximum & Minimum Load	20N
Depth Gain	15%
Loading & Unloading rate	50 MN/sec
Dwell Period	10sec
Indenter	Berkovich

4.5.6.2 Shear Punch Test (SPT)

Shear Punch Test (SPT) is an efficient test technique for evaluating the mechanical properties when material availability is limited. SPT is similar to the blanking operation, where a flat solid punch shears the sheet material through a die at a constant speed. The curve obtained during the shearing operation has many similarities to tensile test curves, e.g., an initial linear elastic region, yield point, a plastic deformation region, and an ultimate load.

The setup used for current SPT studies is shown in Fig. 4.13a, and the schematic of SPT is shown in Fig. 4.13b. Both the punch and die were made of hardened steel.

Figure 4.13 (a) Shear Punch Test set up. The die portion shows clamping fixture for the sample with the die fixed underneath, (b) Schematic for shear punch test showing a shear zone confined to the annular clearance zone. This assumes simple shear deformation.

The punch is attached to a load cell with a capacity of 4500 N. LVDT with a travel range of 3.2 mm measures the displacement of the punch. The load-displacement data are acquired through a data acquisition system 'PDAQ 55'. All shear punch tests were performed at room temperature using a screw driven ATS machine with a constant cross head speed of 4.2×10^{-3} mm/sec.

Before SPT, compact is sliced in the transverse plane of the tube, and subjected to grinding and polishing by using 400 to 2400 grit papers. Compacts prepared for SPT has a wide range of thickness from 150 to 500 μm . On single compact four to five tests carried at different locations. From the load-displacement curves of SPT, shear stress-displacement is calculated.

Shear stress ' τ ' is calculated from the following relation

$$\tau = \frac{P}{\pi dt} \quad (4.3)$$

where, P is the applied load, t is specimen thickness, d is the average of punch and die diameter.

The peak point of the shear stress-displacement curves gives the ultimate shear strength, which indicates the maximum strength of the compact.

CHAPTER 5

COMPARISON BETWEEN FINITE ELEMENT ANALYSIS AND EXPERIMENTAL RESULTS

The main aim of this chapter is to validate the finite element analysis results with experimental results, and to establish the reliability of FEA results. In addition, load and effective strain values calculated from the FE simulations are compared with the experimental load and hardness values.

Results obtained from FEA and experiments were discussed in sections 5.1 & 5.2, respectively. Comparison was made between FEA and experimental results in section 5.3.

5.1 FINITE ELEMENT ANALYSIS OF ECAE WITH COPPER

FE simulations of single pass ECAE was carried out with tool angles $\phi = 90^\circ$ and $\psi = 10^\circ$ by considering the stress-strain relationship properties of pure copper obtained from the tensile test. These simulations were carried out with a coefficient of friction of 0.05. The deformed mesh and effective strain contours were shown in Figs. 5.1 & 5.2, respectively. The deformation is relatively smooth and follows simple shear perfectly [10], which can be observed from the deformed mesh. The deformed mesh along the outer corner is not deformed unlike the regions at the centre and the inner corner. Thus the effective strain achieved along the outer corner is small compared to the other regions. To reveal the variation of effective strain, it was plotted across the width, A-B, (located in Fig. 5.2) as shown in Fig. 5.3. The effective strain distribution is more uniform up to 90% of the width and the remaining 10% width shows lower strain values due to the chosen outer corner angle.

Figure 5.1 Deformed mesh of the copper sample at the end of deformation with $\phi = 90^\circ$ and $\psi = 10^\circ$.

Figure 5.2 Effective strain contours of the copper at the end of deformation with $\phi = 90^\circ$ and $\psi = 10^\circ$.

Figure 5.3 Effective strain variation of the copper across the width A-B. (Location of A-B is shown in Fig. 5.2).

Fig. 5.4 shows the load-displacement curve obtained during the ECAE simulations. It has shown some fluctuations, which are un-avoidable in explicit analysis. During the deformation, the front end of the sample sticks to the bottom of the die, and then

the flow starts. The same can be observed from the first peak load at the start of deformation. Once the deformation starts, the load decreased gradually.

Figure 5.4 Punch load-displacement curve of single pass ECAE of copper calculated from FEA.

5.2 ECAE OF COPPER

Pure copper rod of $\phi 10\text{mm}$ was cut into lengths of 60mm, and vacuum annealed at 600 °C for two hours. Annealed sample was subjected to single pass ECAE with the die setup shown in Fig. 4.1. Sample was coated with molybdenum disulfide (MoS_2) lubricant before ECAE. The extruded and annealed samples are shown in Fig. 5.5. The load-displacement curve recorded during ECAE is shown in Fig. 5.6. Micro hardness (by using Micro hardness tester MXT 70) profiles taken along the flow plane are shown in Fig.5.7. After ECAE, sample for hardness measurements was prepared through the following three steps (shown in Fig. 5.5). First, 5mm disc at the centre of the sample was sliced in the transverse plane; next the same disc was sliced into two pieces along the flow plane, F-F. Finally, the sliced piece was polished and used for hardness measurements. Hardness profile was taken across the width, A-B, with a distance of $\sim 0.5\text{ mm}$ between the indents. Hardness measurements were carried out with a load = 100gf, and dwell time = 10sec.

Figure 5.5 Copper samples before and after single pass ECAE.

Figure 5.6 Force-displacement recorded during single pass ECAE of pure copper along with different stages of deformation.

Fig. 5.6 shows the force-displacement curve recorded during ECAE. It shows that deformation during the ECAE will be taking place in different stages. In the first stage, sample fills the entire die by upsetting, which can be observed from the force-displacement curve reaching the peak load position. In the second stage, as the material enters the exit-channel of the die elastic recovery takes place because of which the force lowers gradually as the deformation progresses. In the third stage force increases monotonously as the ram displacement continues. This is due to the

increase in the frictional resistance with the increase in the contact area between the sample in the exit-channel and the inner surface of the die. The decrease in the force at the end of the deformation (fourth stage) is due to the reduction of triaxial stress state as the rear surface of the bulk material nears the die diagonal.

Fig. 5.7 shows the hardness profiles in the flow plane of annealed and ECA extruded samples. ECA extruded sample shows the hardness values ~ 2.5 times the annealed samples. Thus it shows that the strength enhancement was achieved during ECAE. In the annealed sample, the hardness values are more uniform except at the surfaces, which are nearly twice of that of the main bulk. This high hardness near the surface is due to surface hardening caused by machining. However, after ECAE the sample displayed only small differences in hardness values between the surfaces and the bulk. The hardness profile of extruded copper sample in this study is more uniform than that of aluminum [145, 146], and titanium [76]. This is mainly due to the lower outer corner angle of the ECAE die used in current study.

Figure 5.7 Hardness Profiles in the Flow Plane of annealed and single pass ECA Extruded copper samples.

5.3 COMPARISON OF FEA RESULTS WITH EXPERIMENTAL RESULTS

Comparison was made for two aspects between the FEA and experimental results. First aspect is the strain homogeneity, next is the peak load needed for extrusion.

To compare the FEA strain homogeneity with experimental values, hardness measurements were made across the width A-B of the sample, as discussed in section 5.2. The hardness profile and effective strain variation across the width A-B were shown in Fig. 5.8. The effective strain consists of large number of data points owing to the fine mesh adopted in the simulations, whereas the experimental hardness measurements were taken only at twenty locations to avoid the strain hardening effect between the indents. The trend of both curves was found to be similar. The hardness and effective strain values are nearly uniform up to 90% of the width A-B. The remaining 10% width has lower strains in FEA, and lower hardness values in experimental. The uniform hardness and strain distribution were achieved in the current study due to the usage of lower outer corner angle ECAE die, and the results also show better correlation between FEA strain homogeneity and the hardness measurements, compared to earlier studies [76, 87].

Figure 5.8 Effective strain and Vickers hardness distributions across the width (A-B) of the copper after single pass ECAE.

The second aspect of comparison was made between FEA and experimental results through peak force and force-displacement curves. The force-displacement curves obtained from FEA and experimental are shown in Fig.5.9. The force-displacement curve calculated from FEA has some fluctuations, which are un-avoidable in explicit analysis. From Fig.5.9 it can be seen that the force-displacement curve obtained from the experiment is different from that of the FEA. In the case of FEA, frictional coefficient assumed can be lower than the real case of experimental ECAE and thus the resultant compressive stresses acting on the sample decrease the shear stresses that cause yielding in the material. Accordingly, the force required for the ECAE deformation is more than that determined from the FEA.

Figure 5.9 Force-displacement curves recorded during the single pass ECAE, experiments and finite element simulations, of copper.

To correlate the peak load from FEA and experimental results, simulations were carried by assuming different friction coefficient values. The influence of friction coefficient on peak load was shown in Fig. 5.10. This curve shows that when coefficient of friction is 0.07, the peak load calculated from FEA is closed to the experimental peak load. This clearly shows that the assumed friction coefficient,

0.05, is too low. This shows that by considering the proper coefficient of friction value, it is possible to calculate the exact peak load needed for extrusion from FEA, which will be the most important factor for designing a sound ECAE die.

Figure 5.10 Peak Load calculated for single pass ECAE of copper from FEA for different frictional coefficients.

In conclusion it was found that from FEA strain homogeneity has good conformity with the hardness measurements, along with good correlation between the experimental and FEA peak load calculations. Exact peak load calculation is possible through FEA with various coefficients of friction. Thus, from the above discussions, it can be concluded that FEA results are validated by the experimental results.

CHAPTER 6

STUDIES ON SINGLE PASS ECAE PROCESSING OF POWDER-IN-TUBES

ECAE processing was carried out on MgB_2 and copper powders, filled in a tube casing and sealed with front and rear plugs of the same material as tube. Such a tube, filled with powder, is hereafter addressed as powder-in-tube (PIT). Section 6.1 covers the basic understanding made on the mechanism of ECAE processing of PIT. Section 6.2 explores the role of tube materials on the ECAE processing of MgB_2 powder. Section 6.3 discusses on the compaction of MgB_2 powder through ECAE. Particle refinement in MgB_2 powder and processing of copper powder are discussed in sections 6.4 and 6.5, respectively. Correlation between the processing mechanism and force-displacement curves of ECAE of PIT is explained in section 6.6.

ECAE processing of MgB_2 PITs was carried by using three different tube materials: pure iron, pure copper and low carbon steel (LCS). MgB_2 powder was filled into these tubes to reach an initial relative density of ~ 0.6 . PITs were subjected to single pass ECAE at room temperature. After ECAE, these tubes were argon sintered at 750°C for an hour followed by furnace cooling, and used for density, microstructure and hardness measurements.

Single pass ECAE processed MgB_2 PITs are shown in Fig. 6.1. None of the extruded tubes showed any cracks on the tube surface. In order to observe further details of compacted MgB_2 powder, these tubes were sliced in transverse plane at the middle of the sample. The cross-sectional images of the ECAE processed MgB_2 PITs are shown in Fig. 6.2.

6.1 MECHANISM OF ECAE PROCESSING OF PIT

The process mechanism of ECAE of PIT has never been studied while both FEA and experimental investigations has been carried out on the bulk material processing of ECAE [45 - 87]. It was found as a result of our studies that ECAE processing of powders is an interesting process, wherein the densification of powders and particle refinement occur simultaneously. Mechanism of processing in ECAE of PITs was understood and discussed in the current studies. The types of

stresses generated during the ECAE processing of bulk materials have been thoroughly reported in previous works [8, 41, 43]. Besides these stresses, additional stresses are defined in the current efforts in order to achieve a clear understanding on the ECAE of PITs. Definitions of these stresses are enlisted in the following paragraph by virtue of their nature and are delineated clearly in Fig.6.3a.

Figure 6.1 MgB_2 PITs, processed with different tubes, after single pass ECAE.

Figure 6.2 Cross-section of MgB_2 PITs, processed with different tubes, after single pass ECAE.

The types are defined in such a way that any type of stress is generated due to the combined effects of some or all of the above types of stresses. The phenomenological analyses were primarily done with the help of pictorial details (Figs. 6.3b & 6.4) obtained from the flow planes of the processed samples.

Type 0: Primary stresses acting on the PIT through the punch

Type 1: Reaction stresses acting on powder and tube resulting from the lateral constraints from the die

Type 2: Normal compacting stresses acting on the powder along the entry-channel, above the die diagonal

Type 3: Principal shear-sliding stresses acting in tube and powder along the die diagonal plane

Type 4: Resolved stresses, from the principal shear-sliding stress along the diagonal plane, acting parallel to the exit-channel on the processed segment in the exit channel.

Type 5: Frictional stresses in powder along the principal shear plane opposing the shear-sliding of the powder

Type 6: Frictional and milling stresses between the particles and on the inner surface of the tube

Type 7: Frictional stresses, acting in the entry-channel on the tube outer surface, after the onset of sample arrival into the exit-channel

Type 8: Frictional stresses, acting in the exit-channel on the tube outer surface, generated after the onset of sample arrival into the exit-channel

Type 9: Normal compacting stresses acting in the exit-channel of the die

6.1.1 Rear Plug and Upsetting

Compaction of the powder starts (Type 0) from this stage onwards as the rear plug starts moving in to the tube. In the next stage of the ECAE process, tube material changes in shape to some extent and starts filling (Type 1) the entry-channel through upsetting. Further compaction of the powder occurs (due to Type 1 and Type 2) in conjunction with this filling mechanism. This continues until the whole space in the entry-channel is filled, thereby leaving no room for buckling of the tube. Subsequent to the rear plug movement and upsetting, load starts increasing primarily due to the resistance offered by the front plug in undergoing ECAE

simple-shear extrusion (Type 3) along the diagonal shear plane (Fig. 6.3a). During this increase in load, compaction of the powder continues. This is due to the steady increase in compacting stress on account of resistance to plastic deformation offered by the front plug and tube material.

(a)

(b)

Figure 6.3 (a) Schematic diagram indicates types of stress acting during ECAE of PITs, and (b) Schematic diagram shows the shear sliding phenomenon.

Figure 6.4 Digital photograph of single pass ECAE processed MgB₂ PIT of Fe tube (sliced into two in the flow plane) and scanning electron micrographs, of the highlighted area, shows the parallel depression lines in the powder compact and wavy patterns at the interface between the tube and powder.

6.1.2 Front Plug Deformation

Fig. 6.5 shows the MgB_2 PIT, with Fe tube, before and after single pass ECAE. The deformed PIT in Fig. 6.5 shows that the lower section of the front plug (near outer corner of the die) deformed before the upper section (near inner corner of the die). This is due to the constraint imposed on the material closer to the inner corner by virtue of higher triaxial state of compressive stresses than at the outer corner as a result of the normal reaction stresses (Type 1) above the inner corner angle. As the length of the tube above the inner corner decreases, the triaxial state of stress in the zone above the die diagonal decreases. On account of this load requirement for the deformation decreases for the front plug in the final stage of the complete arrival into the exit-channel.

Figure 6.5 X-Ray Images of MgB_2 PIT of Fe tube before and after single pass ECAE shows the wavy patterns at the interface between the tube and powder interface.

6.1.3 Shear-Sliding of MgB_2

After the front plug and the surrounding tube have completely entered the exit-channel of the die, it is the turn of the hard brittle powder to move along the channels in accordance with the constraints imposed by the die. A number of parallel depression-lines across the powder appeared in the processed samples as can be seen from the SEM images in Fig. 6.4. Unlike the case of front plug, MgB_2 powder does not undergo shear deformation along the die diagonal due to its low ductility. Instead, the powder undergoes shear-sliding by separating, into

parallelograms of powder compacts, off the entry-channel at regular intervals of ram-displacement. After the separation, each of these compacts moves along the exit-channel by pushing the front segment forward into the exit-channel. This is due to the resolved stress (Type 4), acting parallel to the exit-channel, dominating the resistance offered by the tube, and the frictional stress (Type 8) in the exit-channel. Each of the shear-sliding events represents one depression-line in the processed sample (Figs. 6.3b & 6.4). The distance between each of such shear-slides is dependent on the volume of powder between the front plug and the die diagonal.

6.1.4 Wavy Patterns and Localized-Deformation

In addition to the shear-sliding, development of wavy patterns can also be seen in the, Figs. 6.4 & 6.5, exit-channel within the thickness of the tube. It can be seen from the figures that these wavy patterns are related with the shear-sliding behavior of the powder core. The depression-lines, caused by shear-sliding, originated at the thinnest point of the tube. Each of the shear-sliding event starts from the peak of the wavy pattern of the tube. A peak in the wavy pattern always represents the region of lowest tube thickness from where tube material flowed to the adjacent regions. Due to this localized plastic deformation there is a spike in load during the process.

Alternating bands of locally deformed and undeformed bands can be seen in the ECAE processed bulk sample shown in Fig. 6.6 [41], which can be visualized as a series of parallel bands. In the case of ECAE processing of bulk solid materials, geometric restrictions imposed by the rigid die are complied sufficiently by the sample through a series of localized-deformations. Each pair of these deformed and undeformed bands undergoes successful arrival in to the exit-channel by virtue of the changes in geometry & orientation. In the case of hollow tube material, a similar localized-deformation occurs by forming wavy patterns as can be seen (Fig. 6.7) for the cases of both MgB_2 and Cu powders in LCS tubes.

It should be understood that the localized-deformation is analogous to the necking behavior in the case of a pure tensile deformation process [145] of a ductile material subjected to large strains. However, there is a striking difference between the localized-deformation behavior of the tensile loading and the ECAE process. In the case of tensile deformation process while the strain increases owing to the localized-deformation at the region of necking, in the case of ECAE, severe strains

are imparted to the alternating regions, each for only a brief period of the whole process. The ECAE strains are generated within the in bands only during the brief period of intersection with the principal die diagonal plane [41, 146]. In other words, whole of the huge strain is induced in a short span of time for every band during the ECAE process. This is the primary reason for relatively higher strain rates in the case of ECAE process for a fixed speed of the ram which in turn increases the localized-deformations in the case of ECAE process.

Figure 6.6 Macrostructures for copper after three passes shows alternate bands of locally deformed and undeformed bands [41] (a) steady flow, $V=0.25$ mm/sec (b) non-steady flow, $V=25$ mm/sec.

Figure 6.7 Wavy patterns in the LCS tube with MgB_2 and Copper compacts.

Localized-deformation occurs in the materials depending on the strain, strain rate and the processing temperature [41,146,147]. Preferential activation of slip systems

in some grains over the others, disregard of the uniformity in the externally applied load, is the reason for the localized-deformation. In other words, greater the variation of the local hardness distribution across the length of the sample, greater is the tendency for the localized-deformation. Such preferential activation of slip systems occurs for low strains, high strain rates and high homologous working temperatures, for a given material. In the case of increase in strain, yield strength of the material within the localized region increases due to strain hardening, as a result of which the no. of regions undergoing deformation increases thereby hindering the commencement of localized-deformation. In the case of increase in strain rate and working temperature, preferential activation of slip systems increases and thus the variation of the microscopic yield strength across the volume of the material increases. Accordingly, the tendency for localized deformation increases. This is factually supported by pronounced localized-deformation behavior in the case of hot ECAE carried out at a range of temperatures as can be seen in Fig. 6.8.

Figure 6.8 Single pass ECAE processed MgB₂ PITs with LCS tube showing enhanced localized-deformation with higher processing temperatures.

Thus, as a converse to the discussion made in the above paragraph, it can be inferred that low strain rates, low temperatures, high strain hardening hinder the localized deformation and help for the achievement of optimal strain and

densification homogeneity. However, other factors like, inter-particle friction, friction between the tube surface and the die and difference in yield strengths in composite samples like hard core and soft tube also influence the homogeneity of deformation in ECAE and thus are to be collectively dealt with for the optimal process results.

6.2 TUBE MATERIAL IN ECAE OF MgB_2 PIT

The magnitude of principal shear stress is dependant on the instantaneous load requirement of the ECAE. As was mentioned previously, tubes with higher yield strengths require higher loads and thus generate higher normal compacting stresses (Type 2) which in turn increase the magnitude of shear stress (Type 3). This suggests that greater the yield strength of the tube & plug materials, greater is the maximum density achievable in each of the parallelogram of compacts for a given ECAE setup. However, various other factors (discussed in section 6.3) like distribution of stress and strain in the PIT also contribute to the average compaction achieved at the end of single pass ECAE. The peak load requirement for ECAE processing of PITs, with different tube materials, increases with increasing yield strength of the tube material. This can be seen from the force-displacement curves shown in section 6.6.

6.2.1 Phase Constitution of MgB_2 Compacts

XRD patterns of the MgB_2 compacts consolidated with different tube materials are shown in Fig. 6.9 along with as received MgB_2 powder. As received MgB_2 powder does not show any additional peaks other than MgB_2 . Whereas, the compacts processed with three different tube materials showed additional MgO phase. MgO formation is due to the oxidation of MgB_2 with the entrapped air in the PITs. MgB_2 powder was filled into the tubes under ordinary atmosphere, without vacuum degassing. Thus, the XRD patterns show no contamination in MgB_2 compacts other than MgO formation (even after high stresses generated during ECAE, and sintering). In addition, the peaks obtained are very smooth and no noise is observed unlike shock wave consolidated MgB_2 bulks [26]. Thus, the MgB_2 compacts consolidated from ECAE maintaining its crystallinity. This clearly shows that tube material does not have any influence on the crystallinity of the MgB_2 compacts.

Figure 6.9 XRD Patterns of the MgB_2 Powder, and MgB_2 compacts after ECAE processing with different tubes.

6.2.2 Microstructure of MgB_2 Compacts

Fig.6.10 shows the polished flow plane micrographs of the MgB_2 compacts processed by ECAE with different tube materials. These micrographs are taken at the inner corner (a), (c), & (e), outer corner (b), (d) & (f). Among these micrographs (a) & (b) belong to LCS, (c) & (d) belong to Fe, (e) & (f) belong to Cu tube processed MgB_2 compacts. No chemical reaction occurred at the interfaces with the various tube materials even with high stresses generated during ECAE, and sintering. However, a layer of reaction products are observable, between MgB_2 and LCS, Cu tube materials, in the tapes and wires produced by PIT and sintering [148, 149].

6.3 COMPACTION OF MgB_2 POWDER THROUGH ECAE

From Table 6.1, it can be seen that the degree of compaction is more for the case of LCS tube than for Fe tube, due to the greater yield strength of the former. However, in the case of copper tube material, which has lower yield strength than Fe, the degree of compaction is more than that of Fe. Thus, it can be seen that the increment in the densities did not follow the differences in initial filling densities

Figure 6.10 Microstructure of MgB₂ compacts near the inner corner (a, c, e), and outer corner (b, d, f) of the ECAE die in the flow plane after ECAE processing with (a) & (b) LCS, (c) & (d) Fe, (e) & (f) Cu tubes.

nor the yield strengths of the tube materials. Compaction efficiency depends on many factors and can not be readily correlated with any one parameter per se. Disrespect to the initial compaction induced during the powder filling, densifications achieved in the compacts is dependent on the ECAE resistance caused by the front plug, inhomogeneity in inter-particle friction, frictional stresses between the extruded segment and the exit-channel, frictional stresses between tube and the powder, and the ECAE resistance of the tube. Variations in initial compaction do not influence the compaction efficiency. This is because of the comparable degree of compaction achieved gradually in all the tubes before the onset of ECAE deformation across the die diagonal. The continuous compaction of the powder during the ECAE is intermittently interrupted by the sudden movement (through shear-sliding) of a segment of particles into the exit-channel from the entry-channel. This is due to the fact that normal compacting stresses (Type 2) cease to increase thereafter. After a compact arrives at the exit-channel, further compaction (Type 9) occurs in it due to the resistance offered to the compact by the frictional stresses (Type 8) between the exit-channel and the tube.

Table 6.1 Initial filling density of PITs and MgB₂ compact density of respective PITs after single pass ECAE.

Tube Material	Initial Powder fill density (g/cc)	Relative Density (Initial)	Density of the Compact (g/cc)	Relative Density (Final)
LCS	1.72	0.65	2.09	0.80
Fe	1.53	0.58	1.77	0.67
Cu	1.76	0.67	2.10	0.80

Apart from the role of yield strength of the tube material, stress homogeneity across the volume of the MgB₂ powder core also contributes to the average densification, achieved in the powder. The greater the stress homogeneity, the more uniform is the compaction process, thus contributing to optimal densification. It should be recalled that frictional stresses (Type 7 and 8) on the tube and strain hardening behavior of the tube material cause (section 3.5.3) inhomogeneity in stress.

Figs 6.11 to 6.13 show the hardness profiles of MgB_2 compacts processed by LCS, Fe and Cu tubes, respectively, in the transverse and flow planes. Hardness profiles were taken in the flow and transverse planes of the MgB_2 compacts processed with different tubes. Each profile constitutes twelve indentations, each 0.4 mm apart. The flow plane hardness profile was taken across the width A-B (Figure 4.6). The transverse plane hardness profile was taken in the X-X direction (Figure 4.6). It can be seen from the hardness profiles that the inhomogeneity of microstructure resulting from the ECAE process is there in both the transverse cross section and flow planes. It has to be noted that the variations in degree of compaction taking place along the PIT along various axes depend on the uniformity in the stress distribution, which in turn is affected by phenomena like localized-deformation and inhomogeneity in various frictional stresses.

The inhomogeneity in the transverse cross section can be majorly attributed to the friction between the powder and the tube while the inhomogeneity in the flow plane should be due to the inter-particle friction itself. Compacting stresses imparted into the powder by the punch decrease as the distance from the punch increases. This is due to the hindrance caused by the inter-particle friction. Thus, the maximum

Figure 6.11 Hardness profiles in flow and transverse planes of the ECAE processed MgB_2 compact with LCS tube.

Figure 6.12 Hardness profiles in flow and transverse planes of the ECAE processed MgB_2 compact with Fe tube.

Figure 6.13 Hardness profiles in flow and transverse planes of the ECAE processed MgB_2 compact with Cu tube.

compaction efficiency, achievable along the die diagonal, increases as the ECAE progresses. Accordingly, it is worth mentioning that the degree of compaction follows a trend similar to that of the load variation with respect to the ram displacement throughout the ECAE process.

6.4 PARTICLE REFINEMENT IN MgB_2 POWDER

In addition to the consolidation, refinement of the MgB_2 powder particle also occurs during ECAE consolidation. The particle refinement occurs mainly due to the breakage of powder during the shear-sliding at the die diagonal and due to the milling stresses (Type 6). Further, the efficiency of particle breakage increases for more densified powder compacts. Fig. 6.14 shows a decrease in average particle size after a single pass of ECAE with respect to the starting powder (particles from 0.3 to 70 μm , with no definite shape). As received powder particle size was analyzed by Frisch Particle Size ANALYSTTE22. Thus, greater the compaction along the die diagonal the more is the number of particles broken. However, the individual contributions of milling phenomenon and shear sliding phenomenon in the refinement process are not clearly known.

Figure 6.14 Micrographs of (a) as received MgB_2 powder and (b) MgB_2 compact, not subjected to any heat treatment, after single pass ECAE.

6.5 PROCESSING OF COPPER POWDER

Single pass ECAE processing of copper (99.4% purity) PIT is carried by using LCS tube at room temperature. After ECAE, tube is sliced and used for density, microstructure and hardness measurements.

After single pass ECAE, the relative density of the copper compact is 98%. It was seen that for the case of copper powder compacting and processing steps are different from that of MgB_2 . After passing through the shear plane, the copper powder compact (Fig. 6.15b) enters into the exit-channel, and does not split into individual parallelograms as MgB_2 (Fig. 6.15a). This is mainly due to the low yield strength and high ductility of the powder particles. Due to the low yield strength of the Cu powder, the shear stresses generated are sufficient for the plastic deformation of powder compact. The powder undergoes deformation due to the mechanical interlocking thereby increasing the interlocking further. This cyclic phenomenon causes the compact to densify close to the theoretical density, before the powder enters the exit-channel. Thereafter, the deformation of the compact occurs like that of bulk material.

Figure 6.15 Micrographs at the centre of the flow plane of the single pass ECAE processed (a) MgB_2 and (b) Copper Compacts with LCS tube.

Fig. 6.16 shows the hardness profiles of copper compact in the transverse and flow planes. Hardness profiles of both the MgB_2 and copper compacts showed variation in hardness in two planes. Thus, the inhomogeneity in the compaction and microstructure is induced by the ECAE process. However, when compared to MgB_2 , copper compact showed greater homogeneity of microstructure as can be seen from the relatively low variation within the hardness profile of the latter. This greater homogeneity in the case of copper powder is due to the greater densification induced in copper powder through the ECAE process.

Figure 6.16 Hardness profiles in flow and transverse planes of the ECAE processed copper compact with LCS tube.

6.6 FORCE-DISPLACEMENT CORRELATIONS

6.6.1 Force-Displacement of PITs

Analysis of force-displacement curves can be used to further support and validate the understanding made on the mechanism of ECAE processing of PIT.

Fig. 6.17 shows the force-displacement curves obtained during ECAE of the MgB_2 PITs. This force-displacement curve can be divided into seven stages as indicated in Fig. 6.17. Initially, load gradually increases corresponding to the increase in powder compaction while the rear plug moves inside the tube (stage 1). After whole of the plug has moved into the tube, load increases (stage 2) due to the initial compaction of the powder. Upsetting of the tube and further compaction takes place during the stage 3. Here, inner space of the entry-channel is completely filled through upsetting of the tube at the localized places where the normal reaction stresses are relatively low. The filling process can occur only by overcoming varying amounts of local frictional forces between the tube surface and the inner surface of the die. This is the reason why complete filling up of the entry-channel takes place across a range of gradually increasing load. After the filling has completely taken place, load continues to increase, corresponding to the elastic compressive strain, until the

Figure 6.17 Force-displacement curves recorded during single pass ECAE processing of MgB_2 powder with different tubes. Different stages, I to VII, of deformation are also indicated.

shear stresses in the diagonal plane reach the yield strength of the front plug and tube material. With the onset of the shear deformation of the front plug, the PIT starts arriving at the exit-channel, where the leading surface of the PIT does not experience reactive compressive stresses. At this point the compressive elastic strain starts recovering through the front face of the PIT in the exit-channel. This corresponds to the net lowering of the load from its peak value. This load lowering completely occurs over a range of ram displacement (stage 4) while beyond stage 4 only deformation of the front plug takes place and extends into stages 5 and 6, as explained subsequently. Soon after the elastic recovery, the continuous increase in the frictional forces at the exit-channel with the increase in the contact area increases the load requirements (stage 5). However, beyond stage 5, when the horizontal rear surface of the front plug is close to the inner corner a state of plane stress condition is experienced due to the disconnection at the powder/plug interface. Thus, the stress required for the successful arrival of the front plug material into the exit-channel decreases as the plane stress condition increases. This stage continues until the complete arrival of the front plug into the exit channel beyond which only powder experiences the vertical load. Stage 6 corresponds to the change in the load requirements for the shear-sliding to occur. In stage 7 load increases gradually due

to the gradual increase in the contact area between the tube surface and the exit-channel. This suggests that the load requirements for the plastic deformation of the front plug are higher than that of the initial shear-sliding events. The serrations in the load curve in this stage correspond to the intermittent shear-sliding. Shear-sliding and wavy-pattern in the tube collectively contribute to the brief increase in load serrations. It should be noted that the shear-sliding of the powder occurs only after inter-particle frictional forces are overcome by the load and the load drops momentarily after each shear-sliding event. The load-displacement curve can give a clear idea of the distribution of the density of the powder along the longitudinal axis of the PIT.

6.6.2 Frictional Forces in the Exit-Channel

Frictional forces arise due to the contact of the deformed material with the surface of the exit-channel. These forces are increased due to the phenomenon of localized-deformation. The resistance caused to the movement of the tube due to the wavy patterns has to be overcome by deformation of the crests of the wavy patterns in compliance with the rigid die constraints. Magnitude of the resistance to the tube movement depends on the resistance to the crest-deformation by the sample tube structure, yield strength of the tube material and the area of tube surface constrained by the rigid die. This is the reason for the greater frictional resistance offered by the iron-based tubes than the copper tube where all are with the same thickness and contact area. The increase in frictional force thus increases with the yield strength of the tube material for a fixed thickness of the tube. The negligible increase in the load requirements for the case of a softer tube, material of copper, whilst predominant frictional forces for the case of LCS tube can be seen from the stage 7 of the force-displacement curves in Fig. 6.17. This is because of greater increase in friction, associated with localized deformation, for higher yield strength material LCS than copper.

6.7 ULTIMATE SHEAR STRENGTH OF SINGLE PASS ECEA PROCESSED MgB_2 COMPACTS

The ultimate shear strength (USS) of MgB_2 compact is calculated from the shear stress-displacement curves of shear punch test (SPT). It indicates the strength of the

compact at different depths. It is possible to estimate the extent of compaction from this test.

The stress-displacement curves obtained during SPT of MgB_2 compact (single pass ECAE processed with copper tube) are shown in Fig. 6.18. These four curves indicate the stress-displacement variation of the four tests performed at different locations in the transverse cross-section of the sample. These curves are plotted after correcting the punch compliance. Fig. 6.19a, b, & c shows the micrographs of the MgB_2 compacts, consolidated by ECAE with different tube materials, after SPT. Fig. 6.19d shows the transverse cross-section of the LCS tube consolidated MgB_2 compact used for SPT, and it also indicates the locations subjected to SPT. The micrographs show that there is no breaking at the circumference of SPT region with LCS and Cu consolidated MgB_2 compacts (Fig. 6.19a & c) unlike Fe tube (Fig. 6.19b).

Figure 6.18 Stress-displacement curves obtained during Shear Punch Testing of ECAE consolidated MgB_2 compact processed with copper tube.

The USS values obtained from SPT of MgB_2 compacts, compacted by ECAE with different tube materials, are shown in Table 6.2. The USS value is high with the LCS tube consolidated MgB_2 compact, whereas there is only a slight difference for USS for Fe and Cu tube consolidated MgB_2 compacts. The variation of USS values

in powder compacts is due to poor sampling nature, which is due to enormous inhomogeneity in the ECAE of PITs. Thus, SPT results show that high strength of the compact is achieved with LCS tube consolidated MgB_2 , and the compaction is more uniform in the following descending order of tube materials: Cu, Fe and LCS.

Figure 6.19 Micrographs of the MgB_2 compacts after Shear Punch Test. These compacts were ECAE consolidated by different tubes (a) LCS, (b) Iron, (c) Copper, and (d) Typical MgB_2 compact used for shear punch testing.

The average USS and average hardness in the transverse plane of MgB_2 compacts processed with different tube materials was shown in Table 6.3. The trends of both USS and hardness of MgB_2 compacts processed with different tube materials are very much similar, and the USS and hardness values are decreased in the following order of tube materials: LCS, Cu and Fe. In all compacts the range of hardness is low, but the range of USS is high, which are due to the locations of the sample chosen for the study. The hardness values are taken in X-X and Y-Y directions in the transverse plane, whereas the USS values are taken at different locations in the

transverse cross section of the sample. In addition, the poor sampling nature also caused this large variation in mechanical properties. It can be observed that the hardness and USS values from ECAE processing of MgB_2 would be more uniform with Cu tube. However, the USS and hardness of the compact are lower. In contrast high USS and hardness are achieved in LCS tube consolidated MgB_2 compact.

Table 6.2 Ultimate Shear Strength (USS) of MgB_2 compacts obtained from Shear Punch Test (SPT).

Tube Material	Sample	Ultimate Shear Strength (MPa)				Avg. USS (MPa)
		Test 1	Test 2	Test 3	Test 4	
LCS	1	93.08	64.81	71.28	114.90	177.64 ± 45.44
	2	173.98	177.22	128.21	---	
Fe	1	57.76	78.61	123.20	---	62.68 ± 34.10
	2	49.33	32.58	34.58	---	
Cu	1	63.31	67.39	41.30	---	63.96 ± 12.05
	2	59.97	81.27	65.20	69.27	

Table 6.3 Average Ultimate Shear Strength (USS) and Average Hardness of MgB_2 compacts processed with different tubes.

Tube Material	Average Ultimate Shear Strength (MPa)	Average Hardness (GPa)	
		Transverse Plane, X-X	Transverse Plane, Y-Y
LCS	177.64 ± 45.44	2.10 ± 0.40	2.10 ± 0.31
Fe	62.68 ± 34.10	1.40 ± 0.31	1.47 ± 0.27
Cu	63.96 ± 12.05	1.49 ± 0.25	1.38 ± 0.27

CHAPTER 7

STUDIES ON MULTIPASS ECAE PROCESSING OF POWDER IN TUBE

This chapter discusses the influence of multiple passes on ECAE processing of MgB_2 PITs. The multiple ECAE passes make it possible to rotate the tubes around its longitudinal axis between adjacent passes, creating different ECAE routes. Accordingly, tubes were processed via four basic routes upto four passes, in order to study the influence of processing route on ECAE processing of MgB_2 PITs. Thus, the influence of processing route on ECAE processing of MgB_2 PITs was discussed in section 7.1. Section 7.2 explains the influence of number of passes on ECAE processing of MgB_2 PITs.

7.1 INFLUENCE OF PROCESSING ROUTE

To study the influence of processing route on ECAE processing of MgB_2 powder filled tubes, four LCS tubes were filled with MgB_2 powder with an initial relative density of ~ 0.6 . (Details regarding the tube preparation, dimensions and powder filling were discussed in section 4.3). These PITs were subjected to ECAE passes at room temperature via Route A (no rotation), Route C (180° rotation), Route B_A (90° clockwise and counter clockwise rotation), and Route B_C (90° clockwise rotation) between the passes. All the PITs were subjected to stress relief annealing under vacuum at 700°C for 25 minutes in between the passes. After four passes, these PITs were sintered at 750°C for an hour under argon atmosphere.

The influence of processing route on ECAE processing of MgB_2 was studied in terms of density, phase constitution, microstructure and deformation properties of the MgB_2 compacts obtained from different processing routes.

Fig. 7.1 shows the PITs after four ECAE passes and sintering. These pictures show the surface of the tubes from two directions. All these PITs have been extruded well and did not show any cracks on the tube surface even after four passes. However, these PITs do not show the details of MgB_2 . In order to observe the details of MgB_2 , these PITs were sliced in transverse plane at the centre of the sample. Sliced cross-sections are shown in Fig. 7.2. Cross-section of the MgB_2 compact is almost

circular with Routes C & B_C, whereas the cross-section is deviated from circular with Routes A & B_A. Samples of 5 mm thickness were sliced in transverse plane from these PITs (same location for all the samples) and used for property characterization.

Figure 7.1 Different views of MgB₂ PITs with LCS tube after four passes via different routes of ECAE.

Figure 7.2 Cross section of the MgB₂ compacts processed from different routes of ECAE.

7.1.1 On MgB₂ Compacts

7.1.1.1 Density

The initial filling density and density of the compacts, and their relative densities were shown in Table 7.1 after four ECAE passes with different routes. The variation of relative densities before and after ECAE was plotted in Fig. 7.3. Density enhancement is achieved in the following descending order of processing routes: A, B_A, C & B_C. The continuous severe strain achieved via Route A is responsible for effective bonding of the particles, accordingly highest density achieved with Route A. Whereas, in all other processing routes debonding of the particles may take place due to rotation of the samples, and therefore the density of the compacts are lower. The density achieved via Route A is comparable with the advanced consolidation techniques used for consolidation of MgB₂ bulks [25, 26]. In addition the load requirement for ECAE passes are very small compared to advanced powder consolidation techniques.

The peak load recorded during the powder zone deformation for different ECAE passes and routes was shown in Fig. 7.4. The peak load recorded is about the highest for Route A in all passes, whereas it is lower for Route C. The peak load of Route B_A and B_C fluctuated quite substantially in between the passes. Due to stress reversal in each pass, the load recorded with Route C is smaller. Whereas, with Routes B_A and B_C rotation of the tube in between the passes is not consistent, and there are more fluctuations in the loads.

Table 7.1 Densities of the MgB₂ compacts processed with different routes of ECAE.

Pass	Initial Powder fill density (g/cc)	Relative Density (Initial)	Density of the Compact (g/cc)	Relative Density (Final)
4A	1.65	0.63	2.41	0.92
4C	1.67	0.63	2.20	0.84
4B _A	1.58	0.60	2.21	0.84
4B _C	1.69	0.64	2.08	0.79

Figure 7.3 Relative Density of the MgB_2 compacts processed with different routes of ECAE.

Figure 7.4 Peak Punch Load recorded during ECAE processing of MgB_2 PITs via different routes.

7.1.1.2 Phase Constitution

To study the influence of processing route on MgB_2 compact crystal structure, X-Ray diffraction studies were carried on the transverse cross-section of the compacts obtained from the four processing routes. Fig. 7.5 shows the XRD patterns of the compacts obtained from the ECAE passes of 4A, 4C, 4B_A and 4B_C along with the initial MgB_2 powder. The compacts processed with all the processing routes showed additional MgO phase, which was not observable in commercial MgB_2 powder pattern. Even after four ECAE passes, there is no additional phase formed in the compacts other than MgO. The MgO formation is mainly due to filling of the powder into the tubes under ambient conditions. This clearly shows that the processing route of ECAE does not have any influence on crystal structure of the MgB_2 compacts.

Figure 7.5 XRD Patterns of the MgB_2 Powder and its compacts processed by different routes of ECAE.

7.1.1.3 Microstructure

To study the influence of processing route on MgB_2 compaction and grain refinement, microstructural studies were carried in the both transverse and flow planes of MgB_2 compacts obtained from four processing routes, and after four passes.

Fig. 7.6 shows the polished flow plane micrographs of the MgB_2 compacts processed by four processing routes. These micrographs were taken from the centre of the sample flow plane. Micro cracks and voids are observable with 4A and 4B_A compacts, whereas the compacts of 4C, 4B_C showed only micro cracks. But the intensity of the cracks is higher for 4B_A compact. No details regarding grain refinement is observable from these micrographs.

Figure 7.6 Flow Plane Micrographs of the MgB_2 compacts processed by different routes of ECAE.

Figs. 7.7 & 7.8 show the transverse plane micrographs taken from the centre and edge of the compacts. The micrographs taken from the centre of the MgB_2 compacts (Figs. 7.7 b & d, 7.8 b & d) showed observable micro cracks and voids. Whereas, the micrographs taken from the edge of the compacts (Figs. 7.7 a & c, 7.8 a & c) does not show any cracks and voids. This clearly indicates that the compact density is higher at the edge, compared to the centre.

Grain refinement occurred in all the compacts processed with the various routes. It is not possible to estimate the exact grain size due to the combination of agglomerates and fine particles. However, comparison with the scale bar shows that the grains are refined to ultrafine size in all the compacts. The micrographs of the compacts of 4A (Fig. 7.7a) and 4B_A (Fig. 7.8a) shows observable finer grains compared to 4C (Fig. 7.7c) and 4B_C (Fig. 7.8c).

Figure 7.7 Transverse Plane Micrographs taken at the edge (a) & (c), and at the centre (b) & (d) of the MgB₂ compacts processed by ECAE four passes via Route A (a) & (b), Route C (C) & (d).

Figure 7.8 Transverse Plane Micrographs taken at the edge (a) & (c), and at the centre (b) & (d) of the MgB_2 compacts processed by ECAE four passes via Route B_A (a) & (b), Route B_C (C) & (d).

7.1.2 On Deformation Properties of MgB_2 Compacts

The influence of processing route on deformation properties of the MgB_2 compacts was studied in terms of hardness and ultimate shear strength.

7.1.2.1 Hardness

Hardness profiles were taken for all the samples in both the transverse and flow planes. Figs. 7.9 & 7.10 shows the hardness at three locations in the transverse plane X-X and Y-Y directions, from left to right corner, and top to bottom of the compact respectively. In transverse plane, X-X direction hardness values at three points are higher for the compact processed by Route A, and lower for Route B_C. In addition the hardness values are also more uniform with Route A compared to all other routes. Even in transverse plane Y-Y direction, the hardness values at three

points are highest for Route A, and lowest with Route B_C. However, in the transverse plane hardness is more uniform for Route C followed by Route A and Route B_A.

Figure 7.9 Hardness of the MgB₂ compact at different locations in the Transverse Plane X-X direction from left to right.

Figure 7.10 Hardness of the MgB₂ compact at different locations in the Transverse Plane Y-Y direction from top to bottom.

Fig. 7.11 shows the hardness values at three points from inner corner to the outer corner of the sample in flow plane, for all the samples. In the flow plane hardness values are almost uniform for all the samples except Route B_A. For Route B_A, the hardness at the centre of the flow plane showed significantly lower value as compared to the inner and outer corners of the compact. Thus non-uniformity in hardness is more evident for Route B_A. The same can be observed from the flow plane micrographs shown in Fig. 7.6. The flow plane of the compact processed with Route B_A showed substantial micro cracks and voids, thus the low hardness values resulted.

Figure 7.11 Hardness of the MgB₂ compact across the width at different locations in the Flow Plane.

Table 7.2 shows the average hardness values of the MgB₂ compacts consolidated by different processing routes of ECAE. These hardness values are the average of twelve indentations in that particular direction. The average hardness is higher, and more uniform in all the three directions with Route A, and lowest and non-uniform with Route B_C. In the transverse plane, the hardness is more uniform for Route B_A, and non-uniform for Route B_C. Thus, the results show that the hardness is homogeneous in the following descending order of routes: A, C, B_A and B_C. The highest hardness in Route A also is due to finer grains and high density of the compact. The density values also mirrored the hardness values for the compacts in

the same sequence of processing routes. In addition the hardness values are quite comparable with hardness of the MgB_2 compacts consolidated by advanced powder consolidation techniques [22 – 24, 26].

Table 7.2 **Average Hardness of MgB_2 compacts processed with different routes of ECAE.**

ECAE Route & Passes	Average Hardness (GPa)		
	Flow Plane	Transverse Plane, X-X	Transverse Plane, Y-Y
4A	4.75 ± 1.09	4.37 ± 1.16	4.58 ± 1.74
4C	4.56 ± 0.8	3.55 ± 1.65	4.06 ± 2.00
4B_A	2.91 ± 1.29	3.61 ± 1.11	3.53 ± 1.13
4B_C	4.15 ± 0.77	1.37 ± 0.76	2.29 ± 1.06

7.1.2.2 Ultimate Shear Strength

The ultimate shear strength (USS) indicates the strength of the MgB_2 compact. A typical stress-displacement curve recorded during shear punch testing (SPT) of the MgB_2 compact, consolidated by four ECAE passes was shown in Fig. 7.12. Table 7.3 shows the USS values obtained from the different tests of 4C and 4B_A compacts. In each sample, four SPT tests were carried at different locations in the transverse direction of circular compact. The average USS is high with 4B_A compared to 4C, and the variation of average USS is also higher with 4B_A. Table 7.4 shows the average USS along with average hardness, in two directions of the transverse plane, for 4C and 4B_A compacts. The high USS value with 4B_A is due to fine grains, and more uniformity of the hardness in X-X and Y-Y directions of the transverse plane compared to Route C.

Figure 7.12 Typical Stress – displacement curve of the Shear Punch Testing of MgB₂ compact processed by four ECAE passes.

Table 7.3 Ultimate Shear Strength (USS) values of MgB₂ compacts obtained from Shear Punch Test (SPT).

ECAE Route & Passes	Sample	Ultimate Shear Strength (MPa)				Avg. USS (MPa)
		Test 1	Test 2	Test 3	Test 4	
4C	1	336.28	206.82	332.29	---	332.66 ± 62.1
	2	360.47	389.8	387.1	315.79	
4B_A	1	419.82	472.76	328.55	341.63	390.69 ± 67.95

Table 7.4 Average USS and Hardness of MgB₂ compacts processed with different routes of ECAE.

ECAE Route & Passes	Average USS (MPa)	Average Hardness (GPa)	
		Transverse Plane, X-X	Transverse Plane, Y-Y
4C	332.66 ± 62.1	3.55 ± 1.65	4.06 ± 2.00
4B_A	390.69 ± 67.9	3.61 ± 1.11	3.53 ± 1.13

7.2 INFLUENCE OF NUMBER OF PASSES

To study the influence of number of passes on ECAE processing of MgB_2 PITs, six pure iron tubes (details of tube dimensions, preparation were discussed in section 4.3) are filled with MgB_2 powder. Initial relative density of ~ 0.6 was maintained for all the samples. These PITs were subjected from one to six ECAE passes via Route C at room temperature. In between the passes these PITs were subjected to stress relief annealing under vacuum at 500°C for 30 minutes. After ECAE, PITs were sintered at 750°C for an hour under argon atmosphere.

Before choosing the processing route for this study MgB_2 PITs of Fe tube were processed via four basic processing routes of ECAE like in earlier section. Fig. 7.13 shows the X-Ray images of the PITs after different ECAE passes and routes. These PITs are able flow smoothly only with Route C up to four ECAE passes, whereas with all other routes either tube or plug is damaged. The damage of the tube or plug influences the density of the compact and can cause the contamination to the powder. In order to overcome these problems associated with Fe tube MgB_2 PITs, Route C is chosen to study the influence of number of passes.

Figure 7.13 X-Ray Images of MgB_2 PITs with Fe tube after different ECAE routes and passes.

The influence of number of passes on ECAE processing of MgB_2 PITs was studied in terms of density, phase constitution, microstructure and deformation properties of the MgB_2 compacts obtained from different passes.

Fig.7.14 shows the X-Ray images of the PITs after different passes of ECAE and sintering. A reference PIT which was not subjected to ECAE is also included in the same figure. After ECAE, wavy patterns are formed at the interface between the tube and powder, and thin depression lines are observed at equal intervals with an inclination of about 51° to the longitudinal axis of the tube in the entire powder zone. These lines indicate the transfer of powder from entry channel to exit channel of the ECAE die.

The brighter shade in the X-ray images represents greater transmittance of the X-rays and thus indirectly correlates with the gross thickness of the metal, x-rays have passed through. It has to be further noted that for each of the pass newer set of wavy patterns smear across the width of the tube. While the latest set of patterns appear predominantly in the X-ray image, cumulative influence of previous sets makes the background, within the interior of the image, brighter. This is the reason why brighter appearance of image interior, for higher passes, should not be mistaken with the increase in density of the powder.

The shape of the powder zone would revert back to the original position after every two passes via Route C. With the increase of multiple ECAE passes, the thickness of the tube decreased, which is due to the turning of tube diameter slightly after each pass, in order for easy entrance of the sample into entry channel of ECAE die. Thus after four passes, the tube diameter reduced, and a small crack on the tube surface is observable with fifth and sixth pass processed tubes. In addition large crack initiated in the fifth pass in front plug, and the plug is broken into two in the sixth pass. Cracking of the front plug is due to lack of sufficient ductility and strain reversal of Route C. The damage of front plug and tube surface will influence the density and deformation properties of the MgB_2 compacts.

To observe the cross-section of the MgB_2 compact, these PITs were sliced along the transverse plane at the centre of the tube. The sliced cross-sections are shown in Fig. 7.15. The cross-sections are almost circular irrespective of the pass number.

Figure 7.14 X-Ray Images of MgB₂ PITs with Fe tube after different ECAE passes.

Figure 7.15 Cross section of the MgB₂ compacts processed from different passes of ECAE.

7.2.1 On MgB₂ Compacts

7.2.1.1 Density

The initial filling density, density of the MgB₂ compact and their relative densities obtained from different ECAE passes are shown in Table 7.5. Fig. 7.16 shows the variation of density of the compact with the increase in ECAE pass number. Density of the compact is gradually increased with the increase in ECAE passes. Density increased from 60 to 90 % after six passes. Density of the compact is also influenced by the load response during the ECAE. In order to study the role of the load in density enhancement, the peak punch load recorded during deformation of the powder zone was also plotted in the Fig.7.16. The load required for deformation is gradually increased with the increase in pass number. During the sixth pass the load is slightly reduced compared to fifth, which is mainly due to the reduced tube thickness.

The density achieved after six passes is comparable with the density of the MgB₂ bulks prepared by advanced powder processing techniques [25, 26]. In addition the energy requirements for ECAE is very small compared to the advanced powder consolidation techniques.

Table 7.5 Densities of the MgB₂ compacts processed from different passes of ECAE.

Pass	Initial Powder fill density (g/cc)	Relative Density (Initial)	Density of the Compact (g/cc)	Relative Density (Final)
1	1.53	0.58	1.77	0.67
2C	1.59	0.60	1.87	0.71
3C	1.48	0.56	1.93	0.73
4C	1.51	0.57	1.96	0.75
5C	1.73	0.66	2.25	0.85
6C	1.57	0.60	2.29	0.87

Figure 7.16 Peak Punch Load and Relative Density of the MgB_2 compacts processed by different passes via Route C.

7.2.1.2 Phase Constitution

To study the influence of number of ECAE passes on MgB_2 compact crystal structure, X-Ray diffraction studies were carried on the transverse cross-section of the MgB_2 compacts obtained from the different passes. Fig. 7.17 shows the XRD patterns of the compacts obtained from the ECAE passes of 1 to 6C along with the MgB_2 powder. The compacts processed with all the passes showed additional MgO phase, which was not observable in MgB_2 powder pattern. Even after six ECAE passes, there is no additional phase formed in the MgB_2 compacts, other than MgO . The MgO formation is mainly due to filling of the powder into the tubes under ambient conditions. This clearly shows that the number of ECAE passes does not have any influence on crystal structure of the MgB_2 compacts.

Figure 7.17 XRD Patterns of the MgB₂ Powder and its compacts processed by different ECAE passes.

7.2.1.3 Microstructure

Microstructural studies were carried out in both the transverse and flow planes of MgB₂ compacts, obtained from the different ECAE passes, to observe voids, cracks and grain refinement.

Fig.7.18 shows the transverse plane micrographs taken from the centre (a), (c), (e), (g) and (i), and from the edge (b), (d), (f), (h) and (j) of the MgB₂ compact, after different ECAE passes. Grain refinement is enhanced with the increase of ECAE passes, which can be clearly observable from these micrographs. However, as before, it is very difficult to measure the exact grain size due to combination of agglomerates and fine particles. With the increase in ECAE passes, compaction of the powders is enhanced. The same can be observed from the micrographs of four and six passes (Fig. 7.18h & i) in the form of bigger agglomerates. Voids and cracks are observable in the micrographs taken from the centre of compacts compared to the edge. Voids and cracks are more clearly observable with the compacts of fourth and sixth passes (Fig. 7.18g & i).

(a)

(b)

(c)

(d)

(e)

(f)

Continued.....

Figure 7.18 Transverse Plane Micrographs taken at centre (a), (c), (e), (g) & (i) and at the edge (b), (d), (f), (h) & (j) of the MgB_2 compacts processed by Pass 1 (a) & (b), Pass 2C (c) & (d), Pass 3C (e) & (f), Pass 4C (g) & (h), Pass 6C (i) & (j).

Fig. 7.19 shows the micrographs taken from the polished flow plane MgB_2 compacts of different ECAE passes. The same flow plane compacts used for hardness and microstructure characterization. The compacts of second, third and sixth (Figs. 7.19 b, c & f) passes show the indentations in the same micrographs. Micro-cracks are observable from all the samples in the flow plane. More the cracks are observed for the fifth and sixth pass (Fig. 7.19e & f) samples. Damage of the tube surface for the fifth and sixth pass tubes observed from the Fig. 7.14 are responsible for these high intensity micro cracks. Compacts processed by second, fourth and sixth (Figs. 7.19 b, d & f) passes shows good compaction compared to one, three and five passes (Figs. 7.19 a, c & e).

Figure 7.19 Micrographs taken at centre of the Flow Plane of the MgB₂ compact processed by (a) Pass 1, (b) Pass 2C, (c) Pass 3C, (d) Pass 4C, (e) Pass 5C, and (f) Pass 6C.

7.2.2 On Deformation Properties of MgB₂ Compacts

The deformation properties of the MgB₂ compacts processed by number of ECAE passes were studied in terms of hardness and ultimate shear strength.

7.2.2.1 Hardness

Hardness profiles were taken in both the transverse and flow planes of the MgB₂ compacts processed by multiple ECAE passes.

Figs. 7.20 & 7.21 shows the hardness at three points from left to right, and from top to bottom of the compact in the transverse plane X-X and Y-Y directions respectively. Hardness at these three points is gradually increased with the increase of pass number in both directions of the transverse plane. However, the hardness is higher in the Y-Y direction as compared to the X-X direction for all passes. In the X-X direction, the fourth pass hardness is more non-uniform while the sixth pass hardness is uniform, whereas the situation is reverse in the Y-Y direction.

Fig. 7.22 shows the hardness at three points along the flow plane. Hardness at these three points is higher with all the passes in the flow plane compared to the X-X direction of transverse plane. Along the flow plane, hardness is almost uniform upto four passes near the inner corner, whereas it is non-uniform near the centre and outer corner. Enhanced hardness at the three points is observable with the fifth and sixth passes compared to first four passes. But the uniform hardness is observable with sixth pass.

Table 7.6 shows the average hardness values of the MgB₂ compacts in both the transverse and flow planes along with the standard deviation. This hardness is the average of twelve indentations in that particular direction. These hardness values along with variation are plotted in Fig. 7.23 for different number of ECAE passes. The average hardness gradually increased with the increase in pass number. This is mainly due to finer grains and enhanced compaction. Upto three ECAE passes the average hardness is uniform in the two planes and three directions. Whereas, from the fourth pass onwards the hardness is increased but non-uniformity of hardness in the three directions also increased. The variation of hardness values are more substantial for the fifth and sixth passes compared to first four passes. This larger

variation of hardness is mainly due to the formation of cracks on the tube surfaces and breaking of the front plug.

Figure 7.20 Hardness of the MgB₂ compact at different locations in the Transverse Plane X-X direction from left to right.

Figure 7.21 Hardness of the MgB₂ compact at different locations in the Transverse Plane Y-Y direction from top to bottom.

Figure 7.22 Hardness of the MgB₂ compact across the width at different locations in the Flow Plane.

Table 7.6 Average Hardness of MgB₂ compacts processed from different passes of ECAE.

ECAE Passes	Average Hardness (GPa)		
	Flow Plane	Transverse Plane, X-X	Transverse Plane, Y-Y
1	1.63 ± 0.44	1.40 ± 0.31	1.47 ± 0.27
2C	1.71 ± 0.26	1.56 ± 0.44	1.77 ± 0.42
3C	1.48 ± 0.37	1.61 ± 0.25	1.64 ± 0.30
4C	1.86 ± 0.19	2.25 ± 0.48	2.01 ± 0.49
5C	2.41 ± 0.41	1.69 ± 0.39	2.14 ± 0.42
6C	2.08 ± 0.39	1.62 ± 0.56	1.92 ± 0.61

Figure 7.23 Average Hardness of the MgB_2 compacts in different planes and directions.

7.2.2.2 Ultimate Shear Strength

The USS is calculated from the shear stress-displacement curves of SPT. It indicates the extent of the compaction. Four tests were carried at different locations in the transverse plane of each compact. The typical shear stress-displacement curves obtained from four tests in the sixth pass processed MgB_2 compact is shown in Fig. 7.24. The tested compact is also shown in Figure 7.24. The USS values obtained from different tests of the compacts processed with different ECAE passes are shown in Table 7.7. The variation of average USS for different number of ECAE passes is plotted in Fig. 7.25.

In the first pass compact, the average USS is small and USS obtained from the different tests also showed a large difference. The same was observed from the hardness values at the three points in the transverse plane as shown in Figs. 7.20 & 7.21. This is mainly due to a lack of sufficient compaction as observed from the density results (Table 7.5). The fourth pass processed compact has a average hardness twice of that of the first pass processed compact. With the increase in pass number the USS values are increased, due to finer grains and enhanced compaction obtained from the multiple ECAE passes. However, the variation is also higher with

Figure 7.24 Stress – displacement curve of the Shear Punch Testing of MgB_2 compact processed by Pass 6C.

Table 7.7 Ultimate Shear Strength (USS) values of MgB_2 compacts obtained from Shear Punch Test (SPT).

ECAE Passes	Sample	Ultimate Shear Strength (MPa)				Avg. USS (MPa)
		Test 1	Test 2	Test 3	Test 4	
1	1	57.76	78.61	123.19	---	86.52 ± 33.43
2C	1	94.17	92.83	109.44	---	98.82 ± 9.23
4C	1	187.93	157.37	140.99	171.9	164.55 ± 20.06
5C	1	170.25	143.06	174.91	---	162.74 ± 17.2
6C	1	172.99	122.76	171.36	190.17	169.65 ± 19.05

Figure 7.25 Average USS of the MgB_2 compacts obtained from different ECAE Passes.

high number of ECAE passes compared to the second pass. The USS value in the fourth pass compact is twice greater than the second pass compact. Even though the USS values are lower with second pass as compared to the fourth, fifth and sixth passes, the difference is quite small.

CHAPTER 8

CONCLUSIONS AND FUTURE WORK

8.1 CONCLUSIONS

To achieve the objectives of present research the main requirements are design and fabrication of ECAE dies and sub-press equipment. The stress-strain histories needed for designing the ECAE die and sub-press equipments were obtained by conducting finite element simulations of the ECAE process using Abaqus/Explicit. The optimal ECAE tool angles, which can generate strain homogeneity in deformed materials, were explored from the simulations.

- ECAE simulations were carried out with different outer corner angles $\psi = 0, 10,$ and 20° under both ideal (elastic-perfectly plastic material with frictionless contact between the die and sample) and realistic (elastic-strain hardening material with frictional contact between the die and sample) conditions. At a channel angle of 90° the results showed that for both conditions, $\psi = 10^\circ$ is the optimal angle for achieving strain homogeneity, maximum PEEQ, and low punch force requirements while avoiding the detrimental effects.
- ECAE simulations were carried out for a range of channel angles, from 90° to 150° with an increment of 15° for a fixed outer corner angle, $\psi = 10^\circ$, results have shown that strain homogeneity is possible with a channel angle of 90° .
- In the case of multipass ECAE, the influence of processing route on strain homogeneity was studied by conducting simulations with routes A & C for tool angles: $\phi = 90^\circ$ and $\psi = 10^\circ$. Smooth and uninterrupted deformation behaviour is shown by Route A. The peak punch force is also slightly higher with Route A as compared to Route C.
- To validate FEA and the experimental results, simulations and experiments were carried out with bulk copper. Results shows that FEA strain homogeneity has good conformity with the hardness measurements, along with good

correlation between the experimental and FEA peak load calculations. Different stages of force-displacement curve of ECAE of bulk copper were explained.

In the next phase of the pursuit, efforts were directed to investigate the single pass ECAE processing of MgB_2 and copper powders with powder in tubes (PIT). Tube materials such as LCS (Low carbon steel), Fe (Iron) & Cu (Copper) were used. Extensive examination, analysis and consolidation were carried out during the experiments which resulted in thorough understanding in the processing behavior of the powders in tubes. From the analysis of the various stresses, the process mechanism of ECAE of powder in tubes (PITs) has been understood as follows.

- In the case of MgB_2 powder, the process was found to be predominantly inhomogeneous due to various frictional forces and the phenomena of localized deformation in the tube and shear-sliding. The process mechanism was observed to be accompanied by compaction of the powder and particle refinement.
- Different stages of force-displacement curve was discussed and correlated with the process mechanism.
- Powder compaction was found to be dependent on various factors, rather than solely dependent on the yield strengths of the tube materials. Homogeneity of stress and strain which are influenced by various frictional forces that governs the efficiency of powder compaction.
- In the processing of powders MgB_2 and copper were found to have different mechanisms in process and densifications.
- Grain refinement is occurred in powder compacts mainly due to shear-sliding at the die diagonal plane and also due to the milling stresses.

In continuation to the above focused works, multipass ECAE processing of MgB_2 PITs were carried out by using LCS and Fe tubes. The influence of processing route on powder compaction efficiency was studied by MgB_2 PITs with LCS tube. The

influence of number of ECAE passes on powder compaction efficiency was studied by MgB_2 PITs with Fe tube.

- MgB_2 PITs with LCS tube, processed via different processing routes of ECAE up to four passes, showed the density enhancement of powder compacts in the following descending order of processing routes: Route A, Route B_A, Route C, and Route B_C.
- MgB_2 PITs with Fe tube, processed via Route C up to six ECAE passes, showed the density enhancement with the increase in pass number.
- No other phases, other than MgO, are observed in MgB_2 compacts even after large mechanical working and annealing.
- Grain refinement is occurred in all the compacts processed with different processing routes of ECAE. However, the exact contribution of the each processing route on grain refinement is in-distinguishable due to combination of fine particles and agglomerates in the powder compacts.

8.2 FUTURE WORK

The following recommendations are suggested for the future work:

- The influence of ECAE process on superconducting properties of MgB_2 compacts is to be studied by conducting magnetization and critical current density measurements. Magnetization and critical current density of the MgB_2 compacts can be measured through superconducting quantum interference device (SQUIDS) and four probe techniques respectively. These results will determine the usefulness of ECAE in the field of superconductivity.
- Preliminary studies on multipass ECAE processing of MgB_2 PITs have been carried out. More detailed studies are needed to study the influence of ECAE processing route on MgB_2 grain refinement and texture. ECAE processing routes have major influence on the texture of the deformed materials. In

addition, in composite PITs the texture will be different at different locations of the flow plane, and at interfaces between the powder and tube. Texture also influences the critical current density of superconductors. Texture studies on existing multipass ECAE processed MgB_2 PITs can be carried by using X-Ray diffraction with pole figure attachment or electron back scatter diffraction (EBSD).

- Finite element modeling of ECAE of PITs can be carried to find the stress-strain histories and powder compaction efficiency. Modeling of ECAE powder compaction can be carried by using Abaqus/Explicit. The data needed for modeling the powder behavior can be obtained through direct shear test, tri-axial test, and hydrostatic test on powder materials. The results obtained from the simulations will be helpful to design the powder compaction experiments.
- Three dimensional finite element modeling of the multipass ECAE can be carried by using Abaqus/Explicit to study the influence of processing routes on stress-strain variations, which will reduce lot of experimental time in deciding the most optimal processing route for particular material.
- Flow localization observed during the high temperature ECAE processing of PITs can be modeled by developing proper constitutive model. The constitutive model should consider the damage, and flow localization phenomena of the ECAE process.
- The processing mechanism derived from the current study will be helpful to optimize powder compaction through ECAE process.
- The designed and fabricated ECAE set up can be used to process solid, tubular and powder materials. More interesting studies on microstructure and mechanical property enhancement through ECAE in wide variety of materials from pure metals, alloys, composites, and polymers can be carried by using the same die.

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APPENDIX

Figure A.1 X-Ray Images of the MgB₂ PITs with LCS tube after different ECAE passes via Route C.

Figure A.2 Force – displacement curves recorded during first pass ECAE of MgB_2 PITs with LCS tube.

Figure A.3 Force – displacement curves recorded during second pass ECAE of MgB_2 PITs with LCS tube.

Figure A.4 Force – displacement curves recorded during third pass ECAE of MgB_2 PITs with LCS tube.

Figure A.5 Force – displacement curves recorded during fourth pass ECAE of MgB_2 PITs with LCS tube.

Figure A.6 Force-displacement curves recorded for different ECAE passes via Route C of MgB_2 PITs with Fe tube.

Figure A.7 Mg - B Phase Diagram [Binary alloy phase diagrams, ASM hand book 1998, ASM International, Material Park, Ohio].

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