MECHANICAL CHARACTERIZATION OF Al- 6061 STRENGTHENED BY ECAP

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degree of Doctor of Philosophy in Mechanical Engineering

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Abstract

A study was carried out to investigate the effects of equal channel angular pressing (ECAP) on mechanical properties and microstructures of Aluminum alloy Al-6061. Teflon and Nylon was also investigated to visualization deformation because of their high elastic plastic behaviour to validate the mathematical model. The severe plastic deformation (SPD) techniques were used to increase strength through grain refinement. The ECAP process has improved upon the mechanical properties of light metal alloys like Al-6061. In this process the specimen size remain unchanged and the required mechanical properties are increased by SPD technique.

The design and development of ECAP experimentation was done on specimens and equipment used to do with suitable measurements and with safety precautions. The experiments were carried out on Teflon, Nylon, and Aluminum 6061. The die and ECAP fixture were designed and fabricated in the Mechanical Engineering Department at University of Engineering & Technology Taxila. A purpose built hydraulic press having capacity of 100 tons with special fixture was used to squeeze the material through ECAP die. In situ heating of die with specimen was employed for smooth flow of material through the die. The temperature was maintained at upper critical temperature (i.e. 450 °C) for two hours for obtaining homogeneous temperature. A total number of eight experiments were performed using the ECAP process on this experimental setup.
The shear strain mathematical model for ECAP was developed considering the elastic recovery of materials after angular extrusion. In addition to shear strain mathematical model, the load required to push the material through ECAP die was calculated to achieve results. The numerical simulation through ABAQUSTM 6.10.1 was performed to validate the mathematical model. The mathematical model of shear strain and orientation in axis was validated successfully with the 3% error along major axis and 2% along minor axis of ellipse. A series of tests including tensile, Vickers hardness, micro hardness, and three points bend fatigue (crack mouth opening displacement) test were performed for mechanical characterisation. In addition, metallographic and fractographic analyses were performed for microstructure verification.

The 3-point bend fatigue tests were performed on as-received and ECAP specimens. The specimens were made according to ASTM-E647 standard from Al-6061 alloy. The fatigue crack growth (FCG) behaviour of as-received and ECAP was investigated and compared against different stress ratios. Different plots were drawn between different geometric parameters and found that fatigue crack growth was slower in ECAP specimens as compared to as-received specimens. This slow rate of FCG was mainly due to the increase in strength by grain refinement introducing severe plastic deformation.

The mechanical and microstructure analyses validated grain refinement through ECAP process. A 25% increase in Vickers hardness, 15% improvement in yield strength and 35% enhancement in ultimate tensile strength was recorded.
Declaration

I certify that research work titled “Mechanical Characterisation of Al-6061 Strengthened by ECAP” is my own work. The work has not, in whole or in part, been presented elsewhere for assessment. Where material has been used from other sources it has been properly acknowledged / referred.

(Engr. Nazeer Ahmad Anjum)

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...to the loving memories of my FATHER and MOTHER.
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Nomenclature

a  Crack length
a/W  Crack length to specimen width ratio
AISI  American Iron and Steel Institute
ASM  American society for metals
ASTM  American society for testing and materials
B  Specimen grip section length
CMOD  Crack mouth opening displacement
COD  Crack opening displacement
CSYS  Coordinate system
da/dN  Fatigue crack growth rate
E  Modulus of elasticity
ECAP  Equal channel angular pressing
EDM  Electric discharge machining
EPFM  Elastic plastic fracture mechanics
F  Force
FCGR  Fatigue crack growth rate
FE  Finite element
FEA  Finite element analysis
f(a/W)  Geometric correction factor
G  Shear Modulus

GPa  Giga Pascal

H  Specimen height

Hz  Hertz

K  Stress intensity factor

K*  Crack driving force parameter

Kmax  Maximum stress intensity factor

kN  Kilo Newton

L  Length of specimen

LEFM  Linear elastic fracture mechanics

m  Slope of Paris law

μ  Surface friction between die walls and material

NY  Nylon

MPa  Mega Pascal

MTS  Material testing system

P  Applied load

R  Radius of fillet

RPP  Rigid-perfectly plastic

Rx, Ry, Rz  Rotation of specimen ends along x, y and z axis

S  Span length

SEM  Scanning electron microscope
TF  Teflon
Ux  Displacement along x-axis
Uy  Displacement along y-axis
V_{2N}  Normal component of the resultant velocity at zone 2
V_T  Tangential velocity component
W  Width of specimen inside gauge length
\bar{W}_i  Deformation energy during ECAP
\bar{W}_f  Friction energy
\bar{W}_e  Equivalent energy
\Delta K  Stress intensity range
\varepsilon  Strain
\nu  Poisson’s Ratio
\sigma_u  Ultimate tensile stress
\sigma_y  Yield stress
\theta, \phi, or \Phi  Die inner angle
\psi  Die Corner angle
\zeta  Recovery angle
1 Introduction

This chapter gives the introduction of equal channel angular pressing (ECAP) used to strengthen engineering materials for industries such as aerospace and automobiles. This research work concerns the study of ECAP effects on materials mechanical properties, designing and manufacturing of ECAP die to carryout experiments. There are different techniques used to increase the strength of materials through grain refinement by introducing shear strain in materials.

Grain size can be regarded as a key microstructural factor affecting nearly all aspects of the physical and mechanical behaviour of polycrystalline metals as well as their chemical and biochemical response to the surrounding media. Hence, control over grain size has long been recognized as a way to design materials with desired properties. Most of the mentioned properties benefit greatly from grain size reduction. As the race for better materials performance is never ending, attempts to develop sustainable techniques for microstructure refinement continue. A possible avenue for microstructure refinement of metals is the use of severe plastic deformation (SPD), a principle that is as old as metalworking itself. Recent essays [1, 2] tell a fascinating story of the art of ancient sword making through SPD. The modern-day history of SPD technology has its beginnings in the seminal work by Bridgman [3] who developed the scientific grounds and techniques for materials processing through a combination of high hydrostatic pressure and shear deformation, which today are at the core
of SPD methods [4]. Bridgman [3] effectively introduced the defining characteristics of SPD processing in the early 1950s. In a strict sense generally accepted in the materials engineering community, an SPD process is currently defined as “any method of metal forming under an extensive hydrostatic pressure that may be used to impose a very high strain on a bulk solid without the introduction of any significant change in the overall dimensions of the sample and having the ability to produce exceptional grain refinement” [4, 5].

Ultra-fine grained materials are currently of great scientific interest due to their unusual mechanical and physical properties. It is possible to develop the ultrafine grained structures in metallic materials by a process of intensive plastic strain [6, 7], very high plastic strains are required at ambient temperature [8]. It is advantageous to produce such materials relatively cheaply in bulk form. Grain refinement technique has long been utilized to strengthen alloys and metals. This can be attained through the use of traditional metal forming techniques, rolling, forging, and extrusion, sometimes followed by a post-processing heat treatment, but grain refinement is limited by the reduction in cross-sectional area that occurs during processing [9, 10]. While large values of strain are capable of being applied, the finished product will be in the form of very thin foils or wires that have limited applications [11].

To overcome these problems new severe plastic deformation techniques were developed, such as high pressure torsion (HPT) [12, 13], multi-directional forging (MDF) [14, 15], cyclic extrusion compression (CEC) [16], accumulative roll bonding (ARB [17] dissimilar channel angular pressing (DCAP) [18] , equal channel angular pressing (ECAP) [19] etc. Among these techniques of metal forming ultrafine grained materials, severe plastic deformation (SPD) techniques,
such as ECAP and torsion straining, are probably the most propitious. The ECAP technique was introduced by V. M. Segal, a Soviet Union scientist in 1972 [20], and it uses the principle of repeated shear deformation to refine the grain size in metallic materials. ECAP induced SPD in materials without reducing the cross-sectional area of the specimens [8]. ECAP is capable of producing significant grain refinement in bulk samples down to sub-micron levels that can greatly improve the tensile and fatigue strength of a material while maintaining reasonable levels of ductility [21].

The material deformation during the ECAP is affected by the nonlinearity of metallic materials, the geometry of mold, the temperature, the friction between the billet and the mold, and the shape of plungers [22, 23]. The early researches were executed only for basic mold design parameters such as the internal angle ($\phi$); between the two mold channels and corner curvature angle ($\Psi$) [24] as shown in Figure 2.6. According to Segal [20], the deformation behaviour during ECAP was thought to be homogeneous in shear plane within the majority of the sample.

The ECAP has a number of advantages [25]: first, very large deformation strain can be obtained after repeated passes without changing the shape of specimens. Second, very uniform and homogeneous deformation can be applicable throughout the cross section of the specimen. Third, no residual porosity is found in the deformed specimens. Fourth, since the size of the specimens is only limited by the size of the die and the pressing facility, it is possible to produce massive samples. Fifth, the areas exposed to tensile stresses are limited during deformation.
The interest in ECAP technique has dramatically increased due to its wide range of applications. ECAP has been employed to produce ultrafine and nanostructured materials. ECAP is also able to produce superplastic materials and to fabricate composites [26, 27].

Many systems of alloy have been successfully developed by ECAP technique. ECAP showed to be a very promising technique in those above mentioned applications. A possibility to produce a material with nano-scaled structure by equal channel angular pressing technique was clearly stated. ECAP technique is applicable to a variety of metals and alloys with a wide range of using purposes [28].

This study has also undertaken material characterization by evaluation of mechanical properties and microstructure processed through ECAP. Microstructures and mechanical properties of Al-6061 have been well-investigated and enabled to achieve very high values. The fatigue life was characterized of ECAP specimens so that material engineers can make an assessment of the usability of such strengthened materials.

There are two approaches used to strengthening the material known as bottom up approach and top down approach.

- **Bottom-up approach:** in this approach different alloying elements are used to change mechanical properties of the material.
- **Top-down approach:** in this technique different material deformation techniques are used to increase the strength of material such as conventional (rolling, extrusion and forging etc.) and advanced severe plastic deformation (SPD).
There are two important reasons in conventional metal deforming techniques that hinder the grain refinement. The first reason is that severe plastic deformation is not easily developed. Secondly no shear strain occurs in these conventional techniques. Therefore ultrafine grained (UFG) structure could not be achieved through these severe plastic deformation (SPD) techniques. Some new and better techniques have been developed which are known as severe plastic deformation (SPD) techniques in the early 1970’s. These techniques are used to change the shape of billet as well as increase the strength of material as shown in Figure 1.1. In Early 1990s, Valiev [29] and his co-workers also introduced SPD method to enhance the strength of the materials by converting coarse grain into UFG [20].

Figure 1.1: Severe plastic deformation techniques

Some new SPD techniques are:

- High pressure torsion (HPT) [12],
- Multi-directional forging (MDF)[14],
Cyclic extrusion compression (CEC) [16],
Accumulative roll bonding (ARB) [30],
Equal channel angular processing (ECAP) [31] and
Dissimilar channel angular pressing (DCAP) [18] to name a few.

Laser shock peening is another SPD technique used to strengthen the materials by applying compressive load or pressure which develops residual stress that enhances the fatigue crack initiation life [19, 32].

Among these SPD techniques equal channel angular pressing (ECAP), is the most dominant technique, used to increase strength of light alloys. ECAP was introduced by Segal [33, 34] and his co-workers in 1970s. A layout diagram about the ECAP development through the years is given in Figure 1.2.
Industries such as aerospace, automobile are continuously looking for high strength light alloys. This reduces the fuel consumption and transportation costs as well as increasing safety of equipment and occupants. Materials undergoing dynamic repeated loading require high fatigue strength. The main reason to develop a high strength light alloys, is to make them stronger, harder, resist to failure, to reduce wear rate, to resist against impact load and also to increase the fatigue life of the components.

There are different methods that can be used to enhance the strength of the materials, by recrystallizing and increasing dislocation through cold working, by strain hardening and solution hardening. There are many other metal deforming methods mainly used to change shape and a little bit grains refinement such as drawing, rolling, forging, extrusion, casting etc. The grain structure could not be refined up to nano-scale by the conventional metal working techniques.

Conventional material strengthening techniques like rolling, forging and extrusion don’t bring high plastic strain that is very essential to refine the grains but SPD techniques do that effectively. The UFG (ultra-fine grained) materials are having very small grains size, equi-axed microstructures with high fraction of grain boundaries, mis-orientation of axis, and high dislocation density. The UFG structure is having very good mechanical properties like hardness, fatigue life, high ultimate and yield strengths as shown in Figure 1.3[35].
The above figure shows that material strength increases with the increase of number of passes but up to a certain passes when processed through ECAP die. Among all other SPD techniques, the ECAP technique is the most effective and important SPD technique to convert coarse grains into refined one. The material can be processed through ECAP without changing the initial geometry and cross-section. Also it can process huge size of stocks. There is no change in the cross-section of the material pressed through ECAP so the process can be repeated many times to enhance strength.

There are many benefits of ECAP processing technique. Following are the main reasons:
1. To produce such kinds of materials that can be used for a wide range of application in automobiles and aerospace industries, this technique can be applied on large sizes of billets [36].

2. This technique is very simple to perform on nearly all kinds of alloys, except that the manufacturing of die is a complicated one [37, 38].

3. The equipment such as a press, furnace, is almost available in most of the laboratories.

4. This technique can be applied on the materials having different crystal structures. It can also be applied on precipitation-hardened alloys to inter-metallic and metal–matrix composites [39, 40].

5. Due to a very high strain and a continuous processing, a homogeneous property can be achieved in the materials [38].

6. This material strengthening technique can be applied on very large samples and also ECAP method can be designed for commercial metal-processing. By using ECAP methods the material lost as a scrap is negligible so is a cost saving method. The ECAP technique shows many of the potential benefits such as, the yield strength of the material increases up to 34% [35], the scrap can be minimized up to 50%, and the energy can be saved up to 40% [41, 42].

Each SPD technique has its own characteristics based on material used, processing parameters such as, die and billet shape, die angles, pressing speed, temperature, pressure, friction and other environmental conditions. The objective of all these SPDs is to improve the mechanical properties. Among them, ECAP is a particular technique by which the shape and cross-sectional area of the billet
remains same. Other SPD techniques improve material properties by changing its shape.

1.1 Problem statement

There are number of methods available for material strengthening. Each method produces specific material characteristics. The ECAP is a material strengthening technique by which the strength of the material is increased by severe shear plastic deformation. The aim is to establish an experimental technique capable of performing ECAP on 20x20 mm square specimens as well as experimentally established the effects of ECAP on the shear deformation of Al 6061 alloy. A mathematical model of ECAP is modified to include the effects of elastic recovery in high recovery materials. Most of the researches are available to investigate the effect of pressure and temperature on the material properties after ECAP for different die angles such as 110°, 115°, 120°. The die angle 90° is rarely investigated without lubrication due to sharp angle which needs extra high magnitude of pressure, lubrication and at very high ram speed. For the purpose a high pressure hydraulic press (single-stage) was designed and fabricated.

To visualize the rearrangement pattern of the grains after passing through ECAP die, an engraved circular marking technique is introduced in this research work using Teflon and Nylon materials. The specimens were split into two parts having cross-section of 10x20 mm square and engraved circular slots were filled with lead shots. Then the two segments of the specimens were put together into ECAP die inlet channel and processed. The well circular slots were converted into an elliptical shape then major and minor axes were determined following the orientation in axis. Shear strains produced in the specimens were also evaluated.
Material characterization of the ECAP material was performed by tensile testing, micro-hardness, 3-point bending fatigue testing and were compared with that of the as-purchase material.

1.2 Objectives of this study

The main aim of this study is to investigate the effects of ECAP on mechanical properties and microstructures of an Aluminum alloy 6061. The fatigue strength, tensile strength and hardness of the as-received and ECAP material are determined and compared. Microscopy was performed to evaluate the effect of achieved microstructure by ECAP process on the material strength. The other objectives of the study are presented below,

1. Identification and analysis of ECAP and other grain refinement SPD techniques, through an extensive literature review.
2. Development of mathematical model of ECAP.
3. Experimental validation of model and experimental setup development. Determination of the deformation of the grain structure through ECAP. For this purpose ECAP of the polymeric materials (Nylon and Teflon) was carried out. Small metallic balls were filled in engraved circular slot to assess the deformation behaviour of the material after ECAP. Image analysis was used to observe the change in shape and orientation of these engraved circular slots. Change in the shape of the engraved circular slot are determined in terms of enclosed area, angles and shear strains, and are compared with the simulation results
4. Experimentation on ECAP Al alloy
5. Validation of the results of current study with the results of numerical simulation performed by FEA software ABAQUS™ 6.10 on Teflon and Nylon materials.

6. Characterization of tensile and microhardness.

1.3 Study methodology

The aim of this section is to describe how the research activities were planed and the procedure or methodology followed to accomplish this research work. In order to carry out this research work progressively and efficiently, the problem was instigated, analyzed, and formulated. Theoretical background of ECAP was studied thoroughly. Design and development of ECAP experimentation was done with suitable measurements and with safety precautions. The sequence of the steps to carry out research activities is shown in Figure 1.4.
Figure 1.4: The work flow diagram
1.4 Thesis organization

The procedure and sequence of this research work has been described and discussed chapter-wise.

Chapter No. 1 is the introduction about history of Equal Channel Angular Pressing development, its importance and applications. Also some other material processing/strengthening techniques are also discussed.

Chapter No. 2 is about literature review in which the work done by other researchers in the field of material strengthening is discussed and compared to this research.

In Chapter No. 3, die designing and load calculations are performed. ECAP die consisting of two channels intersecting each other at 90 degree, is divided into three different zones to determine velocity in these zones. A mathematical model is developed to calculate shear strain in deformed Teflon and Nylon specimens.

Chapter No. 4 is about the experimental setup, procedures and methodologies. In this chapter die and plunger design is performed. Properties of testing materials are also discussed here. The preparation of specimens for tensile, hardness and fatigue testing are also discussed in detail.

In Chapter No. 5 Numerical Analysis is performed by FEA software ABQUS™ 6.10 on Teflon and Nylon to determine shear strains and to compare these with experimental results and image analysis results. A close conformity is found.

Chapter No. 6 is about results and discussion. The results obtained by fatigue test, tensile test and hardness tests are presented and are discussed by comparing with those of numerical simulation and literature.
Chapter No. 7 is about conclusions and future recommendations.

1.5 Summary

Equal channel angular pressing technique, along with other severe plastic deformation techniques is discussed in detail. The effect of ECAP on material strength, its grain size, grain boundaries and orientation in the axis are elaborated. Research problem along with outline and methodology of research is given in this chapter. The research background is discussed that consist of stages of the material strengthening methods and pros/cons of each technique. The experimental flow chart and thesis organization is also described.
CHAPTER 2

2 Literature Review

Introduction

In this chapter basics of SPD techniques and their effects on material strength through grain refinement are described and the use of these techniques in industrial sector is presented. The significance of ECAP and its effects on the material mechanical properties such as grain refinement, fatigue crack growth resistance, fracture toughness, tensile strength and hardness have been highlighted. The shear strain developed in the billet after ECAP is discussed. Numerical simulations for the study of stress and strain distributions in the ECAP billet are reviewed and different ECAP factors influencing mechanical properties, such as die angles, temperature, pressure, plunger speed, and back pressure are discussed. The work flow diagram is shown in Figure 2.1.
2.1 Severe Plastic Deformation (SPD)

In Severe plastic deformation (SPD) ultra-fine grained structure (UFG) is achieved when processed through ECAP [74]. The grain structure produced by
SPD is also called nanostructure because the grain size is within the range of 100-1000 nm [35].

2.1.1 Grain refinement effects
Material's mechanical properties can be improved by converting coarse grains into refined ones [75]. The grain refinement processes have been carried out for many decades. Severe plastic deformation process is employed to develop very high plastic strain in materials for grain refinement [4]. The researchers always remained determined to identify new SPD techniques for strengthening the materials to minimize the failure rate [31, 49, 76, 77]. The SPD is applied on light alloys to achieve high strength to weight ratio, such as Aluminum, magnesium and copper alloys. These alloys have vast applications in aerospace and automotive industries [40]. In some of the advanced SPD techniques such as twist extrusion and ECAP, strengthening of materials is achieved without changing the shape of work-piece [29, 78].

Traditional material processing methods such as forging, extrusion, rolling, and drawing have been used in which the cross-section and size of the material is changed [79]. However, all these methods cannot develop severe plastic deformation in the materials, necessary to refine grains. Probably, it is due to the fact that conventional metal forming methods are unable to develop sufficient straining and high angle orientations in the metals [80-83]. To obtain high orientation angles and high strength light alloys, the new SPD techniques are realized to overcome these problems. As a result submicron grains, also known as UFG materials, are produced using SPD techniques [84, 85].
Ultra-Fine Grained materials are the polycrystal materials whose grains are equi-axed and very small in size. These polycrystal materials have good mechanical properties and high strength [14, 17, 86, 87].

Grain size of nano-crystalline materials is equal to or less than 100 nm. Zhu et al. introduced two approaches i.e., bottom-up and top-down, to create nano-crystalline materials [5, 63]. The ‘bottom up’ technique is used to assemble individual atoms or to develop nano-scale building segments such as deposition of electrons, condensation of inert gas, physical and chemical authentication. Top-down approach is applied to refine the coarse grains of materials using different material deformation techniques known as SPD [88-90].

These SPD techniques enhance ultimate and yield strength of material two to three time. When aged artificially at 180°C after ECAP, ductility of the material was improved but the strength was decreased [39, 91, 92].

In some of the SPD techniques cross-section and shape of the billet does not change, such as High Pressure Torsion (HPT), ECAP, Multi-axial Compression/Forging(MAC/F), and Accumulative Roll Bonding (ARB) [64, 93-95]. Among the above mentioned techniques, the ECAP process introduces severe plastic deformation on thick materials, whereas thin sheets are processed by repetitive corrugation and strengthening (RCS), accumulative roll bonding (ARB) and con-shearing techniques [64, 96, 97].

Highly fine grained structures or nano-scale structures can only be obtained with the help of advanced high plastic techniques. The fine grained structures and the size of grains depend upon many deformation parameters such as processing route, the method adopted, die angles, magnitude of the shear strain, type of
material, composition, temperature and pressure, etc. [98-103]. The grain size of material processed through ECAP falls between 300 to 400 nm [104-106].

When severe plastic deformation is performed on the materials, mechanical properties are improved but thermal strength becomes very poor because of high dislocation density [26, 52, 95, 107, 108]. ECAP is the most effective technique among all the SPD techniques developed over past twenty years [51, 109].

2.1.2 Basic requirement of SPD

There are some basic requirements of SPD methods that should be kept in mind to create UFG structures, these are as follows [18, 20, 110].

- For grain refinement up to nanoscale, there should be large orientation in angles.
- There should be high shear strain to create a uniform nanostructure throughout the specimen.
- When SPD processes performed on the samples, the cracks or other mechanical damage should not be developed.

These requirements cannot be achieved through conventional material deformation techniques [35, 97].

2.2 SPD techniques in industrial context

For the last twenty-five years in the history of materials, a wide range of investigations have been carried out to develop materials of high strength by many research groups [74, 111]. Researchers have developed new SPD techniques, and also ample literature is available on these techniques which include: high pressure torsion (HPT), accumulative roll bonding (ARB), asymmetric rolling (AR), constrained groove pressing (CGP), equal channel
angular pressing (ECAP), dissimilar channel angular pressing (DCAP), multidirectional forging (MDF), repetitive corrugation and straightening (RCS), and cone-cone method (CCM). The detailed description of these techniques is given in the following sections.

2.2.1 High pressure torsion (HPT)

HPT was introduced by Percy Bridgman in 1946, in which material is kept between two anvils [112, 113]. This process is used for grain refinement and improvement of strength and hardness of material by introducing severe plastic deformation. A very high strain is developed that causes the ultrafine grains and strengthens the materials due to non-homogeneous deformation. Compressive load is applied on the material with the help of upper anvil, while the lower anvil is rotated at desired speed, creating torsional forces as shown in Figure 2.2.

![Schematic diagram of HPT](image)

Figure 2.2: Schematic diagram of HTP [62]
The material without losing its dimensions, remained under a severe shear state between the two anvils. The hardness generated due to HPT is higher than the other SPD techniques and grain refinement was achieved up to nano-meters [25, 114].

2.2.2 Accumulative roll bonding (ARB)
ARB is very simple process which does not require special equipment like other SPD techniques [30]. However, the surfaces to be joined using this technique must be well cleaned before passing through the rolling mills to achieve a good quality of bonding [80, 115]. It is a sheet metal forming process in which two sheets of the same material are piled together as shown in Figure 2.3.

![Figure 2.3: ARB deformation process [115]](image)

These piles of sheet are heated below their phase transformation temperature and then passed through the rolling mills so that the sheets are tied up. After one pass the sheet is cut into two halves, then these two halves are piled up together and in this way the process is carried out several times to achieve the required properties of the material [115].
2.2.3 Asymmetric rolling
In this technique, the rolling mills are designed in such a way that one roller has higher speed than the other roller. Rollers speed can be controlled independently using speed controller as well as by using rollers of different sizes as shown in Figure 2.4. The sheet being rolled between the rollers, experiences high frictional forces opposite in direction on both sides [116]. Due to normal compressive load, sever shear stresses are developed. The cross-section and shape of the material change when passed through rollers. The micro-structure of the processed material is alike other SPD techniques [117, 118].

![Figure 2.4: Process of asymmetric rolling [119].](image)

2.2.4 Equal channel angular pressing (ECAP) practices
The ECAP method is used to change the physical properties of the material by introducing severe plastic deformation when pressed through ECAP die. The material grain size converted into ultrafine grained by introducing severe plastic deformation. This severe plastic deformation results shear straining in the material as mechanical properties and microstructures directly affected by this, so
grains refinement take place. The severe plastic deformation mainly depends upon the following parameters:

- Die geometry consisting die angle, die corner angle and channel sizes.
- Materials properties such as hardening behaviour and strength.
- ECAP process variables such as lubrication, ram speed and temperature.

The die geometry consists of a uniform cross section area, the die angle “ϕ” and corner angle “Ψ”. These two angles play a vital role to develop severe plastic shear strain that is very important for the grain refinement, when specimen is pressed through ECAP die. These two angles are selected on the bases of material properties such as hardness and strength.

Shearing takes place when material is passed through the closed bend channels of the die. The grains are elongated along longitudinal axis with different angle orientations. When material passes through shear plane, the grains are subdivided causing a refined grain structure.

The grains structure before and after ECAP can be observed in the Figure 2.5. In figure (a) the cup and cones are very large with large cavities whereas in figure (b) the grain size becomes very small with packed grains.
(a) As-received  
(b) ECAP

Figure 2.5: Structure of grain of Al 6061 alloy

In short, severe straining; change in orientation and identical shear deformation results strong and refined grain structure [4]. This technique is employed to refine the microstructure through shear deformation without adding alloying elements [8, 33]. The building block of ECAP consisting of a die, a plunger and billet is shown in Figure 2.6. The ECAP process can be repeated several times on a single specimen without change of cross-section [47].
The grain refinement and the geometric characteristics depend on pressure, temperature and environmental conditions [20]. The formation of plastic zone on ECAP billet at the intersecting of two channels is shown Figure 2.7.

Figure 2.7: Distribution of effective strain in the deformed sample during single ECAP pass [10]

The ECAP billet is divided into different sections, the front section is named head deformation zone (HDZ), next is steady-state deformation zone (S-SDZ), main
deformation zone (MDZ) where two die channels intersect each other at 90 degree, and groove deformation zone (GDZ) [10]. The heterogeneous strain, which depends on wall friction and matrix geometry, can be observed between upper and lower surface of the billet. The head deformation zone is having non-uniform strain whereas a uniform strain distribution can be observed in S-SDZ. The MDZ is the main shear deformation zone which is located at the intersection of the two channels.

Many different material processing/forming techniques are being employed to improve or to attain desired mechanical properties since the history of material science. In this connection, rolling mills can be traced back into 1590, used to deform the material into desired shape. Their main concern was to change the material to get the desired shape rather than improve upon its properties. With the advancement in material science, different metal reforming methods were developed. These techniques are known as traditional techniques i.e. rolling, forging and extrusion. These techniques in the past were used to change the shape of the billet and to convert into useful product.

ECAP is a metal forming process in which material properties are changed by mechanical treatment without any change in shape and cross-section. In this process a billet is passed through an angular die channel by applying load with the help of plunger. Under some circumstances hot extrusion is performed for easy flow of billet through die [43]. External heating and friction increase surface temperature of billet below critical limit. When billet comes outside after the process, the temperature falls down suddenly which results thermal stresses in the material. These thermal stresses may cause surface cracking when their value exceeds the tensile strength of material [48].
2.2.5 Signification of ECAP method

ECAP was introduced by Segal et al. in 1970 [33, 49]. It is a new technique which is employed to improve mechanical properties, such as yield strength, ultimate tensile strength, toughness, hardness, etc. The grain refinement is the main reason to improve the mechanical properties of the materials which is caused by severe plastic deformation [50-52]. This process has following three distinct features:

1. Capability to introduce ultrafine grains due to severe deformation.
2. Process is simple as compared to other SPD techniques.
3. Process can be applied on the materials for mass production.

Simple shear is the leading phenomenon of ECAP to introduce high strain in the material. Therefore, the most important feature of ECAP is to achieve UFG structure and special textures [6, 53, 54].

Using the ECAP die, the billet is pressed thought two channels intersecting each other at 90° that consisted of two angles known as die angle ‘Φ’ and the corner angle ‘Ψ’ as shown in the Figure 2.8. [27, 28, 55]. These two angles of the ECAP die have a significant role in grain refinement and extrusion of the billet. The die corner angle facilitates a smooth flow of billet during extrusion. The typical value of the die angle is between 90° to 120°, whereas the corner angle is in the rage of 0° to 30° [56-58]. The billet experiences a severe shear deformation at the intersecting of the die channels as shown by dotted lines in Figure 2.8 [59-61].
Figure 2.8: Schematic illustration for the flow of material through equal channel angular pressing [19].

If corner angle $\Psi$ is kept equal to zero, then shear plane is stationary, whereas if non-zero angles are used then the shear plane gradually rotates along the shape of the corner. Plastic flow of the material does not depend upon the corner angle $\Psi$, its objective function is only to provide a round path as shown Figure 2.9 (b).
The key feature of this corner angle is that the shear plane gradually moves from the upper dashed line to the lower dashed line as shown in Figure 2.8. The important and unique feature of ECAP, which makes it superior from other deformation processes, is that in this process the deformation is very homogenous and also it takes place in the locality of the shear plane. Also the shear strain is zero in the thickness direction, so the deformation in only two dimensional and therefore involves nearly simple plane shear strain [17, 43]. The main difference between ECAP die from conventional extrusion dies is that the cross-section, shape and dimension remain the same at the inlet and outlet channels of ECAP. In conventional extrusion dies, the inlet angle and outlet angle is different and because of that, the cross-section, shape and dimensions are different as shown in Figure 2.10. Due to this difference, the deformation of materials performed by conventional extrusion is only once, whereas by ECAP, the process can be repeated many times. So the total effective strain due to conventional method is equal to single pass whereas by ECAP the accumulated
strain is equal to sum of strains per pass [62]. Hence total strain after ECAP is evaluated using equation 1.1.

\[ \varepsilon_{Total} = \varepsilon_1 + \varepsilon_2 + \ldots + \varepsilon_n = N.\varepsilon_1 \]  

(2.1)  

(Total strain in conventional extrusion is, \( \varepsilon_{total} = \varepsilon_1 \))

![Conventional extrusion die with nomenclature](image)

Figure 2.10: Conventional extrusion die with nomenclature [63].

Very high shear strain and hydrostatic pressure play a vital role to deform the billet at the intersection of the die channels. In ECAP process, if the fillet radius \( R \) is kept equal to zero at the outer corner of the die, the work piece experiences a simple shear at this intersection [61], cracks developed in the material as shown in Figure 2.11, indicating the importance outer radius that should be provided to avoid such cracking.

![ECAP specimen after single pass](image)

Figure 2.11: ECAP specimen after single pass.
When material is pressed through these die channels, the shear strain thus induced for frictionless deformation is derived by Segal et al. [8, 17, 58, 64]. For ideal conditions i.e., when $\Psi = 0$, the shear strain is given by the following equation [65, 66];

$$\gamma = 2 \cot \frac{\varphi}{2}$$  

(2.2)

Under actual conditions, this equation is modified accordingly considering the actual corner angle ($\Psi$). The shear strain in the billet after single pass can be calculated as [8, 47, 67];

$$\gamma = 2 \cot \left( \frac{\varphi}{2} + \frac{\psi}{2} \right) + \Psi \csc \left( \frac{\varphi}{2} + \frac{\psi}{2} \right)$$  

(2.3)

The Von Mises effective strain that includes die corner angle $\Psi$ (for single pass), can be determined by using the following equation developed by Iwahashi et al. [47].

$$\varepsilon_{eff} = \frac{2 \cot \left( \frac{\varphi}{2} + \frac{\psi}{2} \right) + \Psi \csc \left( \frac{\varphi}{2} + \frac{\psi}{2} \right)}{\sqrt{3}}$$  

(2.4)

The above equation was modified for N number of passes by Wu et al. [68] as:

$$\varepsilon_{eff} = \frac{N}{\sqrt{3}} \left( 2 \cot \left( \frac{\varphi}{2} + \frac{\psi}{2} \right) + \Psi \csc \left( \frac{\varphi}{2} + \frac{\psi}{2} \right) \right)$$  

(2.5)

Nakashima and co-workers had explained the effect of die and corner angles by performing different experiments by changing these angles [8]. Their research showed that increase in die angle and corner angle decreases the equivalent strain developed after single pass. The corner angle decreases the value of the equivalent strain by providing inhomogeneous plastic flow in the material. Also the die angle has more effect of introducing the strain during ECAP process as
compared to corner angle. The ECAP process has the ability to develop different microstructures and textures [69, 70]. Moreover, with the help of ECAP process, it is possible to control the microstructural development, which is not possible by conventional methods. Segal et al. explained that different microstructures can be attained by applying multiple extrusions when billet is rotated between consecutive passes [71]. Researchers have demonstrated the principle of billet rotation and their experimental results showed that the microstructural characteristics mainly depend on the precise processing conditions during ECAP [6, 47].

Higher values of plastic strain can be achieved if the billet is rotated and pressed through ECAP die for multiple passes. Furukawa et al. [6] and Jiang et al. [25] have explained four different processing routes for ECAP and designated these routes as A, B_A, B_C, and C as shown in Figure 2.12.

During the route A, the billet is pressed in the ECAP die in several consecutive cycles without rotation of the billet. For route B, the billet is rotated at 90° along its longitudinal axis in between the cycles. This route can be subdivided into two different routes, namely route B_A, in which rotations occur in alternate directions and route B_C, in which rotations occur in one direction only. In route C billet is rotated at 180° in between the cycles. As these rotations of the billet generate different slip systems, this results in development of different microstructures when processed through ECAP die [72, 73].
2.3 Severe plastic deformation SPD theories

<table>
<thead>
<tr>
<th>Year</th>
<th>Name of researcher</th>
<th>Technique</th>
<th>Description</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>1977</td>
<td>V. M. Segal [43]</td>
<td>ECAP</td>
<td>Shear strain depends on friction uniformity in channels and die angles.</td>
<td>ECAP method is very convenient technique used to examine the correlation between structure development and shear strain. Severe plastic deformation causes grains refinement.</td>
</tr>
<tr>
<td>1979</td>
<td>J. Richert et al. [44]</td>
<td>Reciprocating Extrusion Compressio n (REC)</td>
<td>Very high force is required that needs an expensive press and special tools.</td>
<td>In this technique, metal passes in a sinusoidal path in which extrusions and compression dies are fixed. Severe repeated plastic deformation results improvement in mechanical properties.</td>
</tr>
<tr>
<td>1989</td>
<td>Valiev at al.</td>
<td>High</td>
<td>The coin shaped specimen</td>
<td>High hydrostatic pressure applied</td>
</tr>
<tr>
<td>Year</td>
<td>Authors</td>
<td>Process Description</td>
<td>Details</td>
<td></td>
</tr>
<tr>
<td>-------</td>
<td>----------------------------------</td>
<td>--------------------------------------------------------------------------------------</td>
<td>-------------------------------------------------------------------------</td>
<td></td>
</tr>
<tr>
<td>1993</td>
<td>Salishchev et al. [36]</td>
<td>Torsion Pressing (HTP) is pressed under high hydrostatic pressure. The lower anvil rotates while upper anvil remains stationary producing excessive strain.</td>
<td>In each step the direction of force is kept different to the previous step.</td>
<td></td>
</tr>
<tr>
<td>1998</td>
<td>Saito, Tsuji, Utsunomiya, Sakai [45]</td>
<td>ARB Conventional rolling mills can be used for this process. Two or more thin foils are used to make a sample. Foils are pressed through rollers under high pressure which not only introduces shear deformation but also joins these foils with each other.</td>
<td>Cost of die is higher and tool is complicated.</td>
<td></td>
</tr>
<tr>
<td>2001</td>
<td>Huang et al. [36]</td>
<td>Repetitive corrugation and straightening (RCS) RCS technique is used to bend a straight sheet in a wavy form with corrugated tool and then restoring back to flat shape by a press.</td>
<td>Cost of die is higher and tool is complicated.</td>
<td></td>
</tr>
<tr>
<td>2004</td>
<td>Beygelzimer et al. [46]</td>
<td>Twist extrusion Billet is passed through a die in which it is twisted at a certain angle and then retwisted back at the same angle.</td>
<td>Multiple passes of the same billet are performed to achieve repeated plastic strain. This technique is well close to ECAP.</td>
<td></td>
</tr>
</tbody>
</table>
2.4 Physical characteristics influenced by ECAP

2.4.1 Grain refinement through ECAP

SPD techniques are used for grain refinement. The earlier research work performed on ECAP by different researchers explained the development of ultra-fine grains in Al-Mg [62, 120] and Al-Mg-Li-Zr alloys [75]. In 1997, Iwahashi and his co-workers conducted a comprehensive research on the microstructural development of pure Aluminum using ECAP [121]. Their work identified that the 1.0 mm grain size of pure Aluminum is converted into nearly 4 μm in single pass [9, 122, 123]. ECAP die was used to convert initial grains into bands of sub-grains. When these sub-grains structured material was again pressed through ECAP die, it transformed them into high angle orientations and finally produced equiaxed micro-structure [85, 124, 125]. These bands of sub grain appeared as regular band structure when a sample was examined after a single ECAP pass as shown in Figure 2.13.

![Figure 2.13: Bands of sub-grains structure after single pass](image.png)
2.4.1.1 Fatigue mechanism in SPD-ECAP

Almost all materials fail due to different loading and environmental conditions. Among all the failure types, fatigue failure is dominant. Fatigue failure consists of three stages known as stage-I, stage-II and stage-III. Stage-I is crack initiation, stage-II is crack propagation, and stage-III is final fracture. Crack initiation is at microscopic level and initiated by heterogeneous plastic flow. Normally cracks initiate from the points of high stress concentration in the material due to surface roughness, scratches, flaws, notches, blow holes etc. The heterogeneous plastic flow develops due to cyclic loading and consequently introduces surfaces resembling to a crack as shown in Figure 2.14.

As a result of continued cyclic loads these features progress and ultimately crack is initiated at the weakest point. The crack may also introduce in the material due to strain heterogeneity. For example, cracks may develop at grain boundaries where localized plastic flow is not adjusted. Another reason of crack initiation is due to the stress concentration at critical location such as sharp edges, and abrupt change in geometry.
By improving fatigue properties of materials, crack nucleation or propagation can be arrested. Resistance to fatigue failure can be improved by grain refinement. The change in fatigue strength with respect to grain refinement is governed by original fine-grained microstructure, fatigue mode low cycle fatigue and high cycle fatigue (LCF/HCF), the dominating slip mode and cyclic softening or hardening that may take place [127]. The objective of ECAP process is to modify grain structure to achieve higher fatigue resistant in addition to improvement in other mechanical properties of the materials [128].

2.4.1.2 Fatigue crack growth rate (FCGR)

FCGR is extension in the crack length per unit cycle, \((da/dN)\) and is determined by slope of the FCG curve in stage-II region. Collini et al. showed that specimens prepared from ECAP material had higher fatigue resistance compared to the base material [129]. In his work, Collini [129] compared FCG resistance of copper
processed through ECAP with base metals and found that the ECAP specimens showed higher FCG resistance as compared to base material. Zhemchuzhnikova [130] identified the effect of ECAP on fatigue behaviour of Al-Mg-Si alloy. The results described that UFG ECAP material provided 25% improvement in fatigue strength. Similar results were gained by Dumitru [131]. Hockauf et al. [48] observed the crack path deviation associated with different orientations specimens of Aluminum alloy. The results showed that higher crack growth rates are observed at higher orientation and near-threshold region.

Temperature has a great influence on the mechanical properties of the material. Researchers investigating tensile and fatigue properties of wrought and PM AA 6061 at different temperatures revealed that increase in temperature resulted in decrease in FCG resistance [107, 132-134]. It was also investigated that fatigue crack growth resistance in T6 is better than that in the T4 temper [135, 136]. The fatigue crack growth is outstanding between 25 to 200°C and 250 to 300°C for T4 and T6 respectively. When compared PM alloy with ingot metallurgy alloy, the PM alloy has poor crack growth resistance [107, 109].

2.4.1.3 Fracture toughness mechanism

Fracture toughness is the ability of the material to absorb energy and deform plastically without rupture and is determined by stress intensity factor. It has mainly three modes known as mode I (opening), mode II (tearing), and mode III (shearing). The fracture toughness can be determined by performing $K_{1c}$ and $J_{1c}$ tests. Researchers showed that aging and heat treatment processes improved fracture toughness of Aluminum alloy [137, 138] which is dependent on notch root radius [139]. Das et. al [140] reported in their research work that increase in
number of passes, improved fracture toughness due to grains refinement and grain fragment with high angle boundaries.

2.4.1.4 Shearing characteristics of material in ECAP

When the billet is passed through ECAP die, severe shear deformation in the material occurs [141]. Refinement of grains is attained through high shearing forces resulting from successive passes of billets after changing their rotation. Figure 2.15 describes the four routes developed after two passes in an ECAP die. Figure indicates that when route C is adopted, the shear deformation occurs along a single plane, but for route A and B C, there are two unique shear planes and for route B A, there are four different shear planes [66].

The effect of different shear planes on the deformation, in which a cubic billet is extruded from ECAP die having die angle $\phi = 90^0$ and die corner angle $\Psi = 0^0$, gives better grain refinement and hence material strength is improved [142]. The rotation for these four routes against first eight passes along X, Y, and Z planes can be observed in Figure 2.16. X-plane is perpendicular to the extrusion direction, Y-plane is perpendicular to the thickness and Z-plane is perpendicular to the width of the billet [6, 143]. All these planes are deformed in a single direction by route A or B A. Most of the researchers used a die having a die angle $\phi = 90^0$ and found that routes B C and C are more useful than routes A and B A. For a cubic element, route B C is re-established after each of four passes and in route C after each of two passes. By using this technique, Zhu et al. [66] found different results with a die angle $\phi = 120^0$. They explained that for developing a sub-microcrystalline structure during the SPD techniques a constant strain path must be maintained. It was shown that for grain refinement, route A was the most

40
useful, routes $B_A$ and $B_C$ had an intermediate effectiveness, and route $C$ was the least efficient [25]. The latest research work showed that the accumulated strain and the interaction of geometrical constraints had significant effect on grain refinement during ECAP process [66].

Figure 2.15: Shear deformation planes during the first four extrusion process in ECAP for four different routes under a die angle of (a) $\Phi = 90^\circ$ and (b) $\Phi = 120^\circ$[5]
2.4.1.5 Yield strength and hardness of ECAP

Research work of Zhao et al. established that ECAP process is very effective for strengthening of material and showed that the yield strength of ECAP specimens was increased by 23% compared to as-received specimens [101, 144].

Hardness of the materials increases when different forging operations are performed. The materials that have strong intermolecular forces or bonds also possess higher micro-hardness. Xu et al. [145] examined in their research work
that with increase in number of ECAP passes, micro-hardness of the material improved.

2.5 Geometrical analysis of the ECAP-billet

ECAP process is SPD technique in which a billet can be processed many times without changing its cross-section. The Figure 4.12 shows that the shape and size of a specimen remains same when processed through ECAP. Figure 2.17 (a) shows the starting position of the specimen which is ready for ECAP processing; (b) the position of the reference specimen before ECAP processing; and (c) the final position when processed through ECAP [146].

Depending on the deformation behaviour, the ECAP specimen was divided into three regions, one the leading specimen end CDE, the middle BCEF, and the tail specimen ABF as shown Figure 2.17.

When specimen is inserted into the ECAP die Figure 2.17 (a), it moved freely and touched the bottom of the die channel. As the leading end of the specimen is pressed through a shear plane CE making an angle of 45 degree is deformed. The plunger applied a huge pressure on the specimen and local stresses are developed along the shear plane CE and thus a stable deformation zone is developed along CE. As plunger pushed the specimen, a continuous extrusion produced a uniformly deformed zone BCEF in the middle region as shown in Figure 2.17 (c).
After the completion of the extrusion, the tail end of the specimen is not experienced any deformation that resembled to CDE and remained un-deformed [147]. During the ECAP process the specimen ends are rotated by an angle $2\theta$ and as a result of that CD becomes bottom and AF becomes top surfaces of the ECAP specimen. This rotation of angle enabled the specimen to maintain its constant cross-section during ECAP processing.

### 2.6 Influencing factors and parameters in ECAP process

There are different SPD techniques used to enhance the mechanical properties of the materials, among them ECAP is one such technique used for material strengthening [5]. There are many parameters that play a vital role on the workability and the mechanical properties of the material when processed through ECAP such as yield strength, tensile strength, fatigue strength, fracture
toughness, and hardness etc. [100, 144]. Following are the factors that affect the material mechanical properties:

### 2.6.1 Die channel angle

Valiev et al. [35] concluded that the die angle (\(\phi\)) is the key element that plays very important role in developing severe plastic strain in the billet when processed by ECAP. The effect of die angle and corner angle on strain can be observed in the Figure 2.18 after completing a single pass [5].

![Figure 2.18: Effect of die angles on equivalent strain](image)

The Figure 2.18 shows that die angle (\(\phi\)) and corner angle (\(\Psi\)) have a great effect on the material characteristics when pressed through ECAP die. Since geometric parameters of die have a critical influence on the grain refinement, researchers used different die angles ranging from 90° to 150° in their work [15, 121].
2.6.2 Die corner angle

The die corner angle ($\Psi$) develops an outer arc of curvature where the two channels of the die intersect each other. This angle plays an important role for developing ultrafine grained materials. This angle plays an important role in hardening the materials which was investigated by different researchers using finite element modelling [62, 148, 149].

2.6.3 Ram speed

The ECAP process is usually performed at very high ram speeds using heavy duty hydraulic presses [150]. For high ram speed, the recovery process is very slow but if ram speed is kept slower, then recovery of angle is fast. If ram speed is very high, there is an abrupt heating of the billets due to friction between die wall and billet, whereas no significant heating takes place at slow speeds [102, 151].

2.6.4 Temperature effects

In ECAP process, temperature is another key factor which has great effect on the grain refinement. With the increase in temperature the grain elongates in the direction of flow of material and mis-orientation of strain induced boundaries decreases [19]. The experimental results show that if temperature increases, the average grain size increases, also the fraction of low-angle grain boundaries increases with the increase in temperature because there is a faster recovery at the higher temperatures [5, 148, 152]. In other words, the specimens can be easily pressed at the temperatures higher than the critical temperature, but below the critical temperature, the ultrafine grained microstructures can be achieved without producing cracks in the billets. The highest fraction of high-angle
boundaries and the smallest average grain size can be achieved by maintaining lower temperature [153].

2.6.5 Internal heating effects
Additional potential thermal effect due to any temperature change is internal heating which has a greater effect on the flow of material when processed through ECAP die. If temperature is kept below critical temperature, there is high dislocation density that results in high orientation in the axis [132, 154].

2.6.6 Back pressure
The back-pressure can be traced from the early work done on the ECAP mechanics [155]. A lot of work is also done by different researchers in the recent years on back-pressure and it has become an area of special interest [24, 156]. The ECAP dies have been developed and designed in such a manner that this process can be performed with very accurate back-pressure using computer controlled apparatus. The main advantage is the improvement in the workability of the processed billets, as well as suppression of the cracks. Also it improves the metal fluidity during ECAP operation. Hence, the lower surface of the billet attains uniform microstructural-refinement. Xia’s work revealed that the grain size can be reduced up to 0.24 μm without back pressure however it can be further reduced to 0.18 μm with back pressure. The back pressure can be achieved by increasing the friction in the exit channel or by introducing a viscous ductile medium [102, 156].
Table 2.1: Summary for the effect of different SPDs on material properties.

<table>
<thead>
<tr>
<th>Type of SPD</th>
<th>Researcher</th>
<th>Material</th>
<th>Shape of Specimen</th>
<th>Topic Discussed</th>
</tr>
</thead>
<tbody>
<tr>
<td>ECAP</td>
<td>Evren Tan. et al. [18]</td>
<td>Aluminum Alloy 6061</td>
<td>Strips</td>
<td>Mechanical and micro-structural characterization</td>
</tr>
<tr>
<td>ECAP</td>
<td>Mckenzie et al. [155]</td>
<td>Aluminum Alloy 6061</td>
<td>Rectangular</td>
<td>Optimum Strength and Ductility</td>
</tr>
<tr>
<td>ECAP</td>
<td>Djavanroodi et al. [157]</td>
<td>Pure Al, &amp; 6061</td>
<td>Cylindrical</td>
<td>Mechanical Properties</td>
</tr>
<tr>
<td>ECAP</td>
<td>P. Cavaliere et al. [158]</td>
<td>6106Zr, 6106Sc</td>
<td>Rounded</td>
<td>Microstructure and Fatigue Properties</td>
</tr>
<tr>
<td>ECAP</td>
<td>Roberto Braga et al. [21]</td>
<td>Copper</td>
<td>Rod</td>
<td>Investigation of grain refinement</td>
</tr>
<tr>
<td>ECAP</td>
<td>A.P. Zhilyaev [159]</td>
<td>Pure Aluminum</td>
<td>Plate</td>
<td>Microtexture and microstructure</td>
</tr>
<tr>
<td>HPT</td>
<td>Genki Sakai et al. [160]</td>
<td>Aluminum Alloy</td>
<td>Cylindrical</td>
<td>Microhardness &amp; Microstructural examination</td>
</tr>
<tr>
<td>HPT</td>
<td>M. Intan et al. [34]</td>
<td>AL 6061 Alloy</td>
<td>Disk</td>
<td>Microstructural refinement, Hardness</td>
</tr>
<tr>
<td>ARB</td>
<td>Seong-Hee Lee et al. [30]</td>
<td>AA1050/AA6061</td>
<td>Sheets</td>
<td>Grain refinement, Hardness &amp; Tensile strength</td>
</tr>
<tr>
<td>ARB</td>
<td>S.H. Lee et al. [45]</td>
<td>Pure Aluminum</td>
<td>Sheets</td>
<td>Grain refinement and Mechanical Properties</td>
</tr>
<tr>
<td>ARB</td>
<td>Nobuhiro Tsuji [146]</td>
<td>Al alloyes and steels</td>
<td>Rods and sheets</td>
<td>Analysis of grain refinement and Tensile strength</td>
</tr>
<tr>
<td>MDE</td>
<td>Sungwon Lee et al. [103]</td>
<td>Aluminum Alloy</td>
<td>Cylindrical</td>
<td>Investigation of grain refinement and evaluation of shear strain</td>
</tr>
<tr>
<td>CEC</td>
<td>J. Richert [44]</td>
<td>Heat-treated 6082 alloy</td>
<td>Square cross-section</td>
<td>Analysis of shear stress, shear strain, and grain structure</td>
</tr>
<tr>
<td>RCS</td>
<td>H. S. Siddesha et al. [161]</td>
<td>Aluminum</td>
<td>Sheets</td>
<td>Characterization of Mechanical properties</td>
</tr>
<tr>
<td>Asymmetric rolling (ASR)</td>
<td>J. Jiang et al. [119]</td>
<td>Pure Aluminum</td>
<td>Sheets</td>
<td>Mechanical properties and microstructures of ultrafine-grained</td>
</tr>
</tbody>
</table>
Table 2.2: Comparison between different SPDs

<table>
<thead>
<tr>
<th>SPD type</th>
<th>Name of researcher</th>
<th>MAT.</th>
<th>Yield Stress</th>
<th>UTS</th>
<th>NO. PASSES or turns</th>
<th>GRAIN size</th>
<th>DIE AGLE (Degree)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ECAP</td>
<td>J.K.Kim , 2001 [80]</td>
<td>Al 6061</td>
<td>20 to 30%</td>
<td>40%</td>
<td>5</td>
<td>3.9 µm</td>
<td>90 to 120</td>
</tr>
<tr>
<td>DCAP</td>
<td>Evern Tan, 2011 [18]</td>
<td>AL 6061-T6</td>
<td>65% increased</td>
<td>7%</td>
<td>02</td>
<td>50 nm</td>
<td>120</td>
</tr>
<tr>
<td>HPT</td>
<td>Zhilyaev, 2008 [12]</td>
<td>Al 1050</td>
<td>44%</td>
<td>5 GPa</td>
<td>20 turns</td>
<td>170 nm</td>
<td>Rotation</td>
</tr>
<tr>
<td>MDF</td>
<td>Rahul Gupta, 2014 [15]</td>
<td>Cu alloy</td>
<td>1.47 times</td>
<td>2.16 times</td>
<td>05</td>
<td>37 nm</td>
<td>110</td>
</tr>
<tr>
<td>ARB</td>
<td>Seong-Hee Lee, 2003 [30]</td>
<td>Pure Al</td>
<td>Up 387 MPa</td>
<td>1.36 times</td>
<td>03 cycles</td>
<td>0.62 µm</td>
<td>3rd Cycle</td>
</tr>
<tr>
<td>Asymmetric rolling (AS)</td>
<td>ZUO Fang-qing, 2008 [118]</td>
<td>Pure Al</td>
<td>Increased upto 20%</td>
<td>66%</td>
<td>02</td>
<td>0.50 µm</td>
<td></td>
</tr>
<tr>
<td>Repetitive corrugation and straightening (RCS)</td>
<td>H. S. Siddesha, 2013 [161]</td>
<td>Pure Al</td>
<td>20%</td>
<td>05 passes</td>
<td>27 µm</td>
<td>Multidirection</td>
<td></td>
</tr>
</tbody>
</table>

2.7 Numerical simulation for ECAP

Different FEA software packages are available to evaluate the material characteristics under different circumstances. Dong-Kyu Kim et al. [162] made a comprehensive research on the texture measurement in the severe strain regimes using numerical simulation. As strain path does not remain same during
ECAP simulation therefore, it is rather tough task to solve the problem [54, 94, 147, 163]. The finite element method (FEM) is a reliable and authentic for analysing the deformation behaviour of materials after different forming processes such as ECAP, DCAP, etc. FEA based analysis provides detailed information regarding strain rate, strain distribution, induced stresses and material flow rate during ECAP process. However, the effectiveness of FE analysis depends on level of accuracy of provided material properties such as shear stress, shear strain, temperature, plastic strain rate, and path of strain etc. [147].

2.8 Research gap

A lot of research work has been performed by the researchers on different materials using ECAP technique [6, 20, 47, 68, 164, 165]. The published research work is limited to mechanical properties determined by only monotonic tensile test. According to published research to this point, no study is performed using 3-point bend fatigue test at low frequency of 10 Hz at different stress ratios. Also the behaviour of the material deformation during ECAP has not been studied so far by engraved circular technique using polymeric materials (Nylon & Teflon). Moreover no published data is available which used image analysis technique for analysis of deformation caused by ECAP. Present research focuses on filling the aforementioned research gaps.

2.9 Research Limitations

In this section research limitations are discussed in brief. These limitations are related to the research methodology, followed by design, edge cracking, SPD methods of metals forming, ECAP and its validation.
2.9.1 Limitation of research methodology

As research is a qualitative process, so there are the possibility of bias and problems with validity, reliability and replication of the results due to the human aspect of the qualitative research method. To counteract the probability of bias and associated problems, the data must be collected through experimentations, performing FEM based analysis and from literature.

2.9.2 Design limitations

To design the mechanical components is a complex one. The designing of mechanical system, components and parts has been a problem since their designing. To perform ECAP process, the designing of ECAP die is very complicated because of two channels intersecting at an angle of 90°. As material progresses through these channels a sever shear strain is developed that is very necessary for the grain refinement. If inner and outer radius is not provided then cracking will developed causing defects in the processed materials

2.9.3 Limitation due to edge cracking

When material is processed be different techniques, the edge cracks are developed. One of the reasons for edge cracking behavior is due to the continuous increase in hardness and strength of the material when processed either in hot or cold state. The second reason is the presence of precipitates that hinder the movement of dislocations, thus resulting in nucleation and propagation of edge cracks due to dislocation pile up. The large straining during ECAP of material is another cause of edge cracking.
2.9.4 Limitations of SPD methods of metals forming

When material is processed through SPD to obtain UFG structure, the hardness and strength increases 3 to 5 times as compared to as-received material but ductility decreases. A similar tendency is well known for metals subjected to heavy straining by other process such as rolling or drawing. Strength and ductility are the key mechanical properties of any material but these properties typically have opposing characteristics thus, materials processed by SPD methods may be strong but rarely ductile. Strengthening at the cost of ductility is not uncommon, and it is not surprising for a high-strength material such as UFG metals or alloys to be prone to instabilities upon large plastic deformation. The UFG materials processed via SPD has no capability to sustain a sufficient high rate of strain hardening and start necking soon after yielding, leading to a plunging tensile curve almost from the outset, which is a major shortcoming of UFG materials. These drawbacks could be an insurmountable hurdle in bringing bulk UFG materials from laboratory to commercialization. Despite this limitation, it is important to note that the SPD processing leads to a reduction in the ductility, which is generally less than in more conventional deformation processing technique such as rolling, drawing and extrusion.

The low ductility is caused by the low strain-hardening rate, which cause early-localized deformation in the form of necking. In general, two factors are responsible for the low or zero strain-hardening rate.

➢ A high density of dislocations already exists in nanostructured materials processed by SPD and the density quickly reaches saturation upon further
deformation. Once the saturation is reached, dislocations no longer accumulate inside the grains and strain-hardening rate becomes zero.

- In the grains with very small diameters e.g. <100 nm dislocations are emitted from a grain boundary segment and disappear into another grain boundary segment on the opposite side of the grain without accumulating inside the grain. The saturation density of dislocation is determined by a balance between the dislocation generation rate and the recovery rate, and this saturation density is expected to be higher at lower deformation temperature and higher strains.

2.10 Summary

In this chapter, the previous work done by different researchers using various SPD techniques has been discussed. The basic requirements and effects of SPD on material characteristics have been reviewed. Among the SPD techniques reviewed, ECAP was studied in detail for its wide application on bulk material processing. Research gaps were identified in this literature review and a topic of research for this study was selected.
3 Die Load Calculation and Mathematical Formulation

Introduction

ECAP die significantly used to increase the strength of material throughout the investigation is presented in detail for load calculation. In view of the ECAP die geometry, the required plunger load was decided through mathematical modelling. The geometric parameters used for load calculation are: desired plunger velocity, deformation energy, equivalent energy, friction and die angle, corner angle, channel inner and outer radii etc. The velocity of the plunger/billet was determined by segmenting the die channel into three different zones. After load calculations, a mathematical model was developed to evaluate the shear strain induced during ECAP process. The flow diagram of this chapter is given in Figure 3.1

3.1 Introduction to ECAP die

The ECAP die is designed to perform the ECAP process for mechanical characterisation. The design parameters consist of die regimes, die corner angle, die angle, maximum pressing load, pressing speed, shape of specimen, size of specimen, working temperature of specimen, and temperature of die itself. The ECAP die consists of two channels intersecting each other at an angle of 90 degree within the die and making a shear plane. These two channels, that have same cross-section at entrance and at exit, constitute die angles known as \( \phi \) and
The die and plunger are made from AISI H13 material known as tool steel material. The ECAP die is shown in Figure 3.2.

Figure 3.1: Work flow diagram of chapter 3

Figure 3.2: Isometric view of ECAP die
3.2 Velocity and load calculation for ECAP

Before manufacturing the ECAP die and plunger, pressing load is calculated for ASTM AISI H13 tool steel. The surface friction or tresca friction is considered as 0.3, because a very high contact pressure developed between die walls and work piece, when huge stock is severely deformed [166]. ECAP die is composed of two channels and these channels are divided into three regimes 1, 2, and 3 for calculating the speed and load as shown in Figure 3.3.

![Figure 3.3: Schematic diagram of die regimes](image)

3.2.1 Velocity calculation of 1-2 regime

The schematic diagram is shown in Figure 3.4, in which velocity is determined from the initial position up to the regime 2. The specimen, which is pressed through the ECAP die with the help of plunger at a constant speed, is inserted in the die inlet channel. The plunger pressed the specimen through ECAP die inlet channel until it reached its bottom extreme position and then entered into the second regime. The initial velocity was considered $V_0$. 
Figure 3.4: Schematic diagram of die (1-2) regimes

\[ V_1 = V_0 \]
\[ V_{1N} = V_{2N} = V_0 \]
\[ V_{T1} = 0 \]
\[ \Delta V_{T(1-2)} = |V_{T1}| + |V_{T2}| = V_0 \]

Where \( V_T \) is the tangential component when billet entered into regime 2 from regime 1 and \( V_N \) is the normal component of the resultant velocity.

\[ \cos 45^0 = \frac{V_0}{V_2} = \frac{1}{\sqrt{2}} \]
\[ V_2 = \sqrt{2} V_0 \quad (3.1) \]

3.2.2 : Velocity calculation of 2-3 regime

Flow direction of material through zone 2 to 3 in ECAP die is shown in Figure 3.5, the velocity \( V_3 \) is calculated as:

\[ V_2 = V_{2N} = V_{3N} \]

Where \( V_{2N} \) is the normal component of the resultant velocity at zone 2.

\[ \cos 45^0 = \frac{V_3}{V_2} = \frac{1}{\sqrt{2}} \]
\[ V_3 = \frac{V_2}{\sqrt{2}} \]

\[ V_3 = \frac{\sqrt{2}V_o}{\sqrt{2}} \]

\[ V_3 = V_0 \]

![Figure 3.5: Schematic diagram of die (2-3) regimes](image)

Let

\[ \Delta V = \text{Change in velocity} \]

\[ S_\Delta V = \text{Rate of change in velocity} \]

Surface, \( h = 20\text{mm} \), the derived formulas are shown in table below

Table 3.1: Derived formula of velocity and rate of change in velocity

<table>
<thead>
<tr>
<th>Location</th>
<th>Surface</th>
<th>( \Delta V )</th>
<th>( S_\Delta V )</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC</td>
<td>H</td>
<td>( \sqrt{2}V_o )</td>
<td>( \sqrt{2}hV_o )</td>
</tr>
<tr>
<td>AB</td>
<td>H</td>
<td>( V_0 )</td>
<td>( hV_0 )</td>
</tr>
</tbody>
</table>

Let

\[ \overline{W_i} = \text{Deformation energy} \]
\( \bar{W}_f \) = friction energy

\( \bar{W}_e \) = Equivalent energy

\( \bar{m} \) = surface friction

\[
\bar{W}_i = \frac{\sigma_o}{\sqrt{3}} (S\Delta V) = \frac{\sigma_o}{\sqrt{3}} (\sqrt{2}hV_0 + h\nu_0)
\]

\[
\bar{W}_i = \frac{\sigma_o}{\sqrt{3}} hV_0(1 + \sqrt{2}) \quad (3.1)
\]

The friction energy is calculated as:

\[
\bar{W}_f = \frac{\bar{m}}{\sqrt{3}} \sigma_o (\Delta V)
\]

\[
\bar{W}_f = \frac{\bar{m}}{\sqrt{3}} \sigma_o \left[ (V_0a) + 4\sqrt{2}V_0 (2h) + 4(V_0b) \right] \quad (3.2)
\]

\[
\bar{W}_e = \bar{W}_i + \bar{W}_f
\]

\( \bar{W}_e \) = is the sum of deformation energy and friction energy and it will be applied by the hydraulic press.

\[
\bar{W}_e = \frac{\sigma_o}{\sqrt{3}} hV_0(1 + \sqrt{2}) + \frac{\bar{m}}{\sqrt{3}} \sigma_o \left[ 4V_0a + 2\sqrt{2}V_0h + 4V_0b \right]
\]

\[
= \frac{\sigma_o}{\sqrt{3}} V_0 \left[ (1 + \sqrt{2})h + \bar{m}(4a + 2\sqrt{2}h + 4b) \right] \quad (3.3)
\]

And for volume with thickness ‘L’;

\[
\bar{W}_e = \frac{\sigma_o}{\sqrt{3}} V_0 L\left[ (1 + \sqrt{2})h + \bar{m}(4a + 2\sqrt{2}h + 4b) \right]
\]

Now for force applied by the press on the specimen is evaluated as:


\[ FV_0 = \bar{W}_c \]

\[
F = \frac{\sigma_0}{\sqrt{3}} L \left[ (1 + \sqrt{2}) h + \bar{m}(4a + 2\sqrt{2}h + 4b) \right]
\]

(3.4)

Following values mentioned below are used to evaluate the desired force:

\( \sigma_0 = 35 \text{ MPa} \)

\( L = 20 \text{ mm} \)

\( h = 20 \text{ mm} \)

\( a = 140 \text{ mm} \)

\( b = 70 \text{ mm} \)

\( \bar{m} = 0.3 \) is the Tresca friction

Substituting these values in equation 3.4

\[
F = \frac{35}{\sqrt{3}} 20 \left[ (1 + \sqrt{2}) h + 0.3(4 \times 140 + 2\sqrt{2} \times 20 + 4 \times 70) \right]
\]

\[
F = 404[48 + 0.3(896)]
\]

\[
F = 128056 \text{ N}
\]

\( F \approx 13 \text{ Tons} \)

### 3.3 Formulation of Shear strain mathematical model

The assembly of die, plunger, base of the die and specimen is shown in Figure 2.6. When specimen is pressed through the ECAP die channels, a severe deformation took place at the intersection of the channels known as shearing
zone. A mathematical model is presented here to formulate the shear strain developed during ECAP process.

The shear strain is defined as

$$\gamma = \tan \theta$$, where \(\theta\) is the angular distortion developed due to shear as shown in Figure 3.6, then total shear strain was calculated of ECAP specimens, using relation mentioned by Xia et al. [76]

$$\gamma = 2 \cot (\frac{\Phi}{2})$$

(3.5)

where ‘\(\Phi\)’ is the ECAP die angle between two channels.

![Figure 3.6: A square deformed by tan \(\theta\) in simple shear [76]](image)

**Determination of principal strain and direction in ECAP**

Flow of material and its behaviour through ECAP die is shown in Figure 3.7. When material is pressed through channel one, each and every line like Bb remains parallel to line B1a, as shown in Figure 3.7 (a). When material is continuously pressed it crossed regimes 2 and entered in regime 3, the line Bb again remained parallel to line Aa. The mathematical modelling is performed according to Xia et al. [76]. The material flow velocity \(v_1\) at entrance of the die channel remained equal to the velocity \(v_2\) at the exit channel, and the
parallelogram BbaA is deformed across Oo to become B'b'a'A'. The angle between two channel is $\Phi$.

(a) The ideal ECAP in which the velocity in the entrance channel, $V_1$, is equal to the velocity in the exit channel, $V_2$, and the parallelogram, BbaA, is deformed across Oo to become B'b'a'A'.

(b) The coordinate transformation from $X_1O_1Y_1$ to $X_2O_2Y_2$

Figure 3.7: Schematic diagram for flow of material through ECAP [76]

The condition shown in Figure 3.7 (a) is re-plotted with a different way in Figure 3.7 (b). Here, lines Aa and Bb in the entrance channel move to become A'a' and B'b' in the exit channel, respectively, remaining parallel to each other and with the same spacing between them. The coordinate system is set up in the entrance channel with the origin O to be at B and the $Y_1$ axis along Bb. This coordinate system becomes $X_2O_2Y_2$ with the origin O to be at B' and the $Y_2$ axis along B'b' (so that no rigid body translation is considered). Point P with coordinates $x_1$ and $y_1$ in $X_1O_1Y_1$ becomes point 'P' with coordinates $x_2$ and $y_2$ in $X_2O_2Y_2$. Now considering Figure 3.7 (b), it is obvious that

$X_1 = O_1Q = O_2R = X_2$, and
$Y_2 = O_2T'$

$Y_2 = O_2\dot{T} = O_2\dot{U} + \dot{W} + \dot{W}T'$  \hspace{1cm} (3.6)

$Y_2 = AU + UP + W'T'$

As $O_2U' = AU = O_1S = Y_1$  \hspace{1cm} (a)

For $U'W' = UP$ &

$Tan\frac{\Phi}{2} = \frac{SP}{UP}$, $UP = SP[Cot\frac{\Phi}{2}]$  \hspace{1cm} (b)

For $WT'$

$Tan\frac{\Phi}{2} = \frac{TP'}{WT'} \Rightarrow WT' = TP[Cot\frac{\Phi}{2}]$

As $TP' = X_2$, so $WT' = X_1Cot\frac{\Phi}{2}$

Also $X_1 = X_2$, so $WT' = X_1Cot\frac{\Phi}{2}$  \hspace{1cm} (c)

Now from equation 3.7

$Y_2 = Y_1 + SP \left[Cot\frac{\Phi}{2}\right] + X_1Cot\frac{\Phi}{2}$

$Y_2 = Y_1 + X_1Cot\frac{\Phi}{2} + X_1Cot\frac{\Phi}{2}$

$Y_2 = Y_1 + 2X_1Cot\frac{\Phi}{2}$

That is, the coordinate transformation is

$X_1 = X_2$ and

$Y_1 = Y_2 - 2X_1Cot\frac{\Phi}{2}$  \hspace{1cm} (3.7)
Using relation, \( X_1^2 + Y_1^2 = R_0^2 \)

Substituting the values & transformation

\[
X_2^2 + \left( Y_2 - 2X_2\cot \frac{\Phi}{2} \right)^2 = R_0^2
\]

\[
X_2^2 + Y_2^2 + 4X_2\cot^2 \frac{\Phi}{2} - 4X_2Y_2\cot \frac{\Phi}{2} = R_0^2
\]

\[
\left[ 1 + 4\cot^2 \frac{\Phi}{2} \right] X_2^2 + Y_2^2 - \left( 4\cot \frac{\Phi}{2} \right) X_2Y_2 = R_0^2
\]  

(3.8)  

Using relation

\[
X_2 = X\cos\alpha - Y\sin\alpha      \quad (d)
\]

\[
Y_2 = X\sin\alpha + Y\cos\alpha      \quad (e)
\]

Substituting these values in equation (3.8)

(a). Illustration of measuring angle \( \alpha \) and \( \zeta \).  
(b). Illustration of a circle deformed to become an ellipse with its long axis at an angle \( \zeta \) to the longitudinal direction of the material in the exit channel for \( \Phi = \pi/2 \).

Figure 3.8: Coordinate transformation and formation of ellipse
\[
\left(1 + 4\cot^2 \frac{\Phi}{2}\right)(XC\alpha - YS\alpha)^2 + (XS\alpha + YS\alpha)
- 4\cot \frac{\Phi}{2} (XC\alpha - YS\alpha)(XS\alpha + YC\alpha) = R_o^2
\]

After simplification;

\[
\left[-\left(1 + 4\cot^2 \frac{\Phi}{2}\right)\sin 2\alpha + \sin 2\alpha - 4 \cot \frac{\Phi}{2} (\cos^2 \alpha - \sin^2 \alpha)\right] = R_o^2
\]

This represents an ellipse in \(X_2O_2Y_2\). Following a further coordinate transformation by rotating the \(X_2O_2Y_2\) system by an angle \(\alpha\) in the counter clockwise direction with a the angle between the long axis of the ellipse and \(O_2Y_2\) shown in Figure 3.8 (a) and determined by setting the xy term of above equation in the new coordinate system after the rotation to zero.

So taking coefficient of xy term equal to zero, to determine the value of \(\alpha\)

\[
-\left(1 + 4\cot^2 \frac{\Phi}{2}\right)\sin 2\alpha + \sin 2\alpha - 4 \cot \frac{\Phi}{2} \cos 2\alpha = 0
\]

\[
\sin 2\alpha \left(-4 \cot^2 \frac{\Phi}{2}\right) = 4 \cot \frac{\Phi}{2} \cos 2\alpha
\]

\[
\frac{\sin 2\alpha}{\cos 2\alpha} = -\frac{4 \cot \frac{\Phi}{2}}{4 \cot^2 \frac{\Phi}{2}}
\]

\[
\tan \alpha = -\tan \frac{\Phi}{2} \ [\text{to eliminate negative sign}]
\]

\[
\tan 2\alpha = \tan \left(\pi - \frac{\Phi}{2}\right)
\]

\[
2\alpha = \pi - \frac{\Phi}{2}
\]

\[
\alpha = \frac{\pi}{2} - \frac{\Phi}{4}
\]

\[
\tan \alpha = \tan \left(\frac{\pi}{2} - \frac{\Phi}{4}\right)
\]
\[
\tan \alpha = \cot \frac{\Phi}{4} = \frac{\cos \frac{\Phi}{4}}{\sin \frac{\Phi}{4}} = \frac{2 \cos \frac{\Phi}{4} \cos \frac{\Phi}{4}}{2 \sin \frac{\Phi}{4} \cos \frac{\Phi}{4}}
\]

\[
\tan \alpha = \frac{2 \cos^2 \frac{\Phi}{4}}{\sin \frac{\Phi}{2}}
\]

\[
\tan \alpha = \frac{1 + \cos \frac{\Phi}{2}}{\sin \frac{\Phi}{2}}
\]

Evaluating major and minor axis of the ellipse formed during ECAP process

The ellipse equation is

\[
\frac{x^2}{a^2} = \frac{y^2}{b^2} = 1
\]

Now for evaluating 'a' or 'R1'

\[
\left( 1 + 4 \cot^2 \frac{\Phi}{2} \right) \cos^2 \alpha + \sin^2 \alpha - 4 \cot \frac{\Phi}{2} \sin \alpha \cos \alpha \right) x^2 = R_0^2
\]

\[
a = \frac{R_0}{\left( 1 + 4 \cot^2 \frac{\Phi}{2} \right) \cos^2 \alpha + \sin^2 \alpha - 4 \cot \frac{\Phi}{2} \sin \alpha \cos \alpha}
\]

\[
\tan \alpha = \frac{1 + \cos \frac{\Phi}{2}}{\sin \frac{\Phi}{2}}
\]

\[
x = \sqrt{\left( \sin \frac{\Phi}{2} \right)^2 + \left( 1 + \cos \frac{\Phi}{2} \right)^2}
\]

\[
x = \sqrt{\sin^2 \frac{\Phi}{2} + 1 + \cos^2 \frac{\Phi}{2} + 2 \cos \frac{\Phi}{2}}
\]

\[
x = \sqrt{1 + 1 + 2 \cos \frac{\Phi}{2}}
\]

\[
x = \sqrt{2(1 + \cos \frac{\Phi}{2})}
\]

\[
\sin^2 \frac{\Phi}{2} + \cos^2 \frac{\Phi}{2} = 1
\]
Now,

\[
\sin \alpha = \frac{(1 + \cos \frac{\Phi}{2})}{\sqrt{2(1 + \cos \frac{\Phi}{2})}}
\]

\[
\cos \alpha = \frac{\sin \frac{\Phi}{2}}{\sqrt{2(1 + \cos \frac{\Phi}{2})}}
\]

Substituting the values in equation (3.9)

\[
a^2 = \frac{R_o^2}{(1 + \frac{4\cos^2 \frac{\Phi}{2}}{\sin^2 \frac{\Phi}{2}}) \frac{\sin^2 \frac{\Phi}{2}}{2(1 + \cos \frac{\Phi}{2})} + \frac{(1 + \cos \frac{\Phi}{2})^2}{2(1 + \cos \frac{\Phi}{2})} - 4 \frac{\cos \frac{\Phi}{2}}{\sin \frac{\Phi}{2}}X \frac{1 + \cos \frac{\Phi}{2}}{\sqrt{2(1 + \cos \frac{\Phi}{2})}} \frac{\sin \frac{\Phi}{2}}{\sqrt{2(1 + \cos \frac{\Phi}{2})}}}
\]

\[
a^2 = \frac{R_o^2}{\frac{\sin^2 \frac{\Phi}{2} + 4\cos^2 \frac{\Phi}{2}}{2(1 + \cos \frac{\Phi}{2})} X \frac{\sin^2 \frac{\Phi}{2}}{2(1 + \cos \frac{\Phi}{2})} + \frac{1 + \cos \frac{\Phi}{2}}{2} - 2 \cos \frac{\Phi}{2}}
\]

\[
a^2 = \frac{R_o^2}{\frac{\sin^2 \frac{\Phi}{2} + 4\cos^2 \frac{\Phi}{2}}{2(1 + \cos \frac{\Phi}{2})} + \frac{1 + \cos \frac{\Phi}{2} - 4 \cos \frac{\Phi}{2}}{2}}
\]

\[
a^2 = \frac{R_o^2}{\frac{\sin^2 \frac{\Phi}{2} + 4\cos^2 \frac{\Phi}{2}}{2(1 + \cos \frac{\Phi}{2})} + \frac{1 - 3 \cos \frac{\Phi}{2}}{2}}
\]

\[
a^2 = \frac{R_o^2}{\frac{\sin^2 \frac{\Phi}{2} + 4\cos^2 \frac{\Phi}{2}}{2(1 + \cos \frac{\Phi}{2})} + \frac{1 + \cos \frac{\Phi}{2} - 3 \cos \frac{\Phi}{2} - 3 \cos^2 \frac{\Phi}{2}}{2(1 + \cos \frac{\Phi}{2})}}
\]
\[ a^2 = \frac{R_o^2}{\sin^2 \frac{\phi}{2} + \cos^2 \frac{\phi}{2} + 1 - 2 \cos \frac{\phi}{2}} \]
\[ a^2 = \frac{R_o^2}{1 + 1 - 2 \cos \frac{\phi}{2}} \]
\[ a^2 = \frac{R_o^2}{2(1 - \cos \frac{\phi}{2})} \]
\[ a^2 = \frac{R_o^2}{2(1 + \cos \frac{\phi}{2})} \]

Multiplying and dividing by \((1 - \cos \phi/2)\)

\[ a^2 = \frac{R_o^2}{(1 - \cos \frac{\phi}{2})(1 - \cos \frac{\phi}{2})} \]
\[ a^2 = \frac{R_o^2}{(1 - \cos^2 \frac{\phi}{2})} \]
\[ 1 - \cos^2 \frac{\phi}{2} = \sin^2 \frac{\phi}{2} \]
\[ a^2 = \frac{R_o^2}{\sin^2 \frac{\phi}{2}} \]

Taking square root on both sides

\[ a = \sqrt{\frac{R_o^2}{(1 - \cos \frac{\phi}{2})^2 \sin^2 \frac{\phi}{2}}} \]
\[ R_1 = a = \frac{R_o \sin \frac{\Phi}{2}}{1 - \cos \frac{\Phi}{2}} \]  

(3.10)

Now 2\textsuperscript{nd} part of equation to find minor radius

\[ b^2 = \frac{R_o^2}{(1 + 4 \cot^2 \frac{\Phi}{2}) \sin^2 \alpha + \cos^2 \alpha + 4 \cot \frac{\Phi}{2} \sin \alpha \cos \alpha} \]  

(3.11)

\[ b^2 = (1 + \frac{4 \cos^2 \Phi/2}{\sin^2 \frac{\Phi}{2}}) \cdot \left( \frac{1 + \cos \frac{\Phi}{2}}{2 \left(1 + \cos \frac{\Phi}{2} \right)} \right)^2 + \left( \frac{\sin \frac{\Phi}{2}}{2 \left(1 + \cos \frac{\Phi}{2} \right)} \right)^2 \]  

\[ + \frac{4 \cos \frac{\Phi}{2}}{\sin \frac{\Phi}{2}} \left( \frac{1 + \cos \frac{\Phi}{2}}{2 \left(1 + \cos \frac{\Phi}{2} \right)} \right) \sin \frac{\Phi}{2} \]
\[
= \frac{1 + 3\cos^2 \frac{\Phi}{2} + 1 + \cos \frac{\Phi}{2} - \cos \frac{\Phi}{2} - 3\cos^2 \frac{\Phi}{2}}{2((1 - \cos \frac{\Phi}{2}))}
\]
\[
= \frac{2 + 2\cos \frac{\Phi}{2}}{2(1 - \cos \frac{\Phi}{2})}
\]
\[
= \frac{2(1 + \cos \frac{\Phi}{2})}{2(1 - \cos \frac{\Phi}{2})}
\]
\[
= \frac{1 + \cos \frac{\Phi}{2}}{\frac{\Phi}{2}} \cdot \frac{1 + \cos \frac{\Phi}{2}}{1 - \cos \frac{\Phi}{2}}
\]
\[
= \frac{(1 + \cos \frac{\Phi}{2})^2}{1 - \cos^2 \frac{\Phi}{2}}
\]
\[
= \frac{(1 + \cos \frac{\Phi}{2})^2}{\sin^2 \frac{\Phi}{2}}
\]

Now;

\[
R_2^2 = b^2 = \frac{R_0^2}{(1 + \cos \frac{\Phi}{2})^2} \cdot \frac{\sin^2 \frac{\Phi}{2}}{1 - \cos^2 \frac{\Phi}{2}}
\]
\[
R_2 = b = \sqrt{\frac{R_0^2}{(1 + \cos \frac{\Phi}{2})^2} \cdot \frac{\sin^2 \frac{\Phi}{2}}{1 - \cos^2 \frac{\Phi}{2}}}
\]
\[
R_2 = b = \frac{R_0 \sin \frac{\Phi}{2}}{1 + \cos \frac{\Phi}{2}}
\]  \hspace{1cm} (3.12)
Now if \( \Phi = \frac{\pi}{2} \)

\[
\tan \alpha = \frac{1 + \cos \frac{\pi}{4}}{\sin \frac{\pi}{4}}
\]

\[
\tan \alpha = \frac{1 + \cos(45)}{\sin(45)}
\]

\[
\alpha = 67.5^\circ
\]

It can be observed that \( \varepsilon_1 = \varepsilon_2 \) from Figure 3.8 (a) and in Figure 3.8 (b) that the angle between the maximum principal strain \( \varepsilon_1 \), and the longitudinal direction of the material in the exit channel is

\[
\xi = \alpha - \left( \frac{\pi}{2} - \frac{\Phi}{2} \right)
\]

\[
\xi = \frac{\pi}{2} - \alpha
\]

\[
\xi = 90 - 67.5
\]

\[
\xi = 22.5^\circ
\]

This is the theoretical elastic recovery angle and it is compared with experimental and numerical results in chapter 6 under article 6.3.3 on page 118 and error is only 10%.

\[
\varepsilon_1 = \ln \frac{R_1}{R_0}
\]

\[
\varepsilon_1 = \ln \frac{\sin \frac{\Phi}{2}}{1 - \cos \frac{\Phi}{2}}
\]

\[
\varepsilon_1 = \ln \frac{0.7071}{0.2929}
\]

\[
\varepsilon_1 = 0.88 \text{ or } 88\%
\]
\[ \varepsilon_2 = \ln \frac{R_2}{R_0} = \ln \frac{\sin \frac{\Phi}{2}}{1 + \cos \frac{\Phi}{2}} = \ln \left( \frac{0.7071}{1.7071} \right) \]

\[ \varepsilon_2 = \ln \left( \frac{0.7071}{1.7071} \right) \]

\[ \varepsilon_2 = \ln(0.4142) = (-0.88) \text{ or } -88\% \]

### 3.4 Summary

Die load is calculated, for the fabrication of hydraulic press, based on different geometric parameters such as plunger velocity, deformation energy, equivalent energy, friction between die walls and material, die angles, and channel inner and outer radii etc. The velocity of the plunger/billet is determined by segmenting the die channel into three different zones. The load required to push the material through ECAP die is calculated equal to 13 tons. Then mathematical model is formulated to determine shear strains and orientation in axis, processed through ECAP die.

### CHAPTER 4

#### 4 Experimental Set Up

#### 4.1 Introduction

In the previous chapter, the die load calculation and mathematical modelling is presented. In current chapter, the experimental setup is discussed in detail.
Initially, design and fabrication of the ECAP die and its plunger is discussed. Then, fabrication of hydraulic press to carry out ECAP experimentations is detailed. An electric furnace is fabricated on the hydraulic press bed to heat die and specimens during process. It is described that how the experiments are performed on Nylon, Teflon and Al-6061. The properties and characteristics of these materials are analysed and presented. The design and manufacturing of specimens made from Nylon, Teflon and Al-6061 used for experimentation are discussed in detail.

At the initial stage, the ECAP process is performed on Teflon and Nylon materials because plastic deformation in shear can be easily visualised in these materials and also to verify the mathematical model. Due to high elastic recovery after plastic deformation in polymers, the behaviour of Teflon and Nylon is investigated at different temperatures as they are famous for their low coefficient of friction and wall friction effects.

Different material characterizing tests are performed on as-received and ECAP specimens such as tensile test, hardness test and 3-point bend fatigue test. Metallography and fractography are performed on as-received and ECAP specimens for material characterization. The work flow diagram is shown in Figure 4.1.
4.2 ECAP die design

The ECAP die plays a vital role for grain refinement when billets are pressed through the die channels. The ECAP die consists of two channels, namely inlet and outlet channel. These channels generate two angles at their intersection and the zone so formed is known as main deformation zone as shown in Figure 2.7. The inside angle is known as die angle (\(\phi\)); whereas, the outside angle as corner angle (\(\Psi\)). The equal channel angular pressing (ECAP) die used in all the experiments is fabricated and manufactured. Die-sinking electrical discharge machine (EDM) shown in Figure 4.2 was used to create a channel having 20x20 mm\(^2\) cross-section. The die is made from AISI H13 tool steel material. Different
parts of ECAP die is explained in Figure 4.3. The detail drawing of Die with dimensions in millimetres is shown in Figure 4.4, the orthographic views of die base plate are shown in Figure 4.2

Figure 4.2: Die sinking EDM machine used to manufacture ECAP die

Figure 4.3: Isometric sketch of ECAP die
Figure 4.4: A detail drawing of ECAP die with dimensions
There are many ECAP die designing parameters that were taken into consideration while designing the die. These parameters include: (i) maximum pressing load, (ii) shape of specimen, (iii) size of specimen, (iv) working temperature of specimen, and (v) die itself.

Since resources are not available locally, therefore, the die angle is kept initially at 90° without providing die corner radius for a square cross section specimen of 20 x 20 mm² and length of 140 mm. For these parameters, the pressing load is calculated 55 tons approximately to process the specimen through ECAP die. After performing multiple experiments on Teflon, Nylon and Al-6061, the behaviour of die angle and die corner angle become obvious. At initial experimentations stage, surface cracks were developed on the processed
specimen due to very sharp corners. The distortion and cracks produced in the
first specimen pressed through ECAP die is shown in Figure 4.6. Later on, it is
decided to apply proper fillet radius at inner and outer corners of the channels. A
fillet radius of 3 mm and corner radius of 8.45 mm was provided by maintaining
corner angle ($\Psi$) of 22 degree. In order to adjust the specimen properly in die
channel, the length of specimen is reduced to 120 mm. It is also decided to heat
the specimen and die in a temperature controlled furnace for smooth flow of the
material. The die along with specimen is heated at a temperature of 450°C for
two hours for a homogeneous temperature.

![Figure 4.6: Behaviour of die angle on specimen for single pass](image)

In the light of problems identified in first series of experiments, second series of
experiments are performed on same materials i.e. Teflon, Nylon and Al-6061;
whereas, the die and specimens were heated separately in a temperature
controlled furnace at 450°C. Since the shifting of hot die and specimen from
furnace to hydraulic press performed in open environment, the sudden
temperature drop is observed at that stage which causes difficulty of billet flow in
die channel. Consequently, excessive cracks were produced in the specimen. To
minimize the problems faced in second series of experiments, the collective
heating of billet and die is decided. For this purpose, the specimen is first inserted
in die channel, and then both were heated. Although, the problem of temperature
variation between die and specimen was resolved; however, the temperature drop occurred during the transfer of die from furnace to hydraulic press. This temperature drop resulted in non-homogeneous flow of material and cracks were still present at certain locations on the ECAP specimen. To overcome this problem, the furnace was built on hydraulic press table around the die. For safety measurements, a high temperature insulation wool (HTIW) is used to minimize heat transfer to the surroundings. The die temperature is controlled by automatic control unit fitted with 4 x 950W heating elements. The detail of furnace is provided in Section 4.3.

4.2.1 Plunger design

The plunger is prepared from AISI H13 tool steel to push the material through ECAP die channels. A hole for mounting bolt is provided at the head of plunger, which is used to connect the plunger with the ram cup of hydraulic press as shown in Figure 4.7. The orthographic views of the plunger are shown in Figure 4.8.
Figure 4.7: Isometric sketch of ECAP plunger

Figure 4.8: Detail drawing of ECAP plunger
4.2.2 ECAP Die Material
Die is made from tool steel AISI H13. This material has high hardenability, excellent wear resistance, good abrasion resistance, resistance to heat checking and hot toughness, and good thermal shock resistance. Certain heat treatment processes such as nitriding, tempering and quenching are performed to improve material characteristics. Quenching is initially performed to increase the hardness and toughness. For further increase in hardness resistance, nitriding is performed. Finally double tempering is applied to remove the thermal stresses.

4.3 Electric conventional chamber furnace
To achieve smooth flow of the billet through ECAP die, the specimens are heated along with ECAP die up to 450°C for a period of two hours to attain a uniform temperature throughout. Since the lower critical temperature of Aluminum Al-6061 is around 450°C, therefore, the furnace is designed to maintain a constant temperature of 450°C fitted with automatic controlled unit. For this design consideration, an electric controlled furnace having element of 4 KW electric power, is developed on the bed of hydraulic press. Thermocouple is provided to measure and control the temperature during heating. The bed and surrounding of the furnace is isolated from hydraulic press with high temperature insulation wool (HTIW). The specific purpose wool had the capacity to withstand against high temperature greater than 1000°C as shown in Figure 4.9 (a) and (b).
4.4 Hydraulic press equipped with control unit

The frame of hydraulic press is made from steel having four columns and two MS plates (base and top) fixed with columns. The two limits switches are also fitted with one of the vertical column to control the span of piston movement. The pressure is applied through hydraulic pump. The slots are provided in the base plate of the press to support the fixture. The fixture is specially designed to prevent the movement of the die along with the plunger during reverse stroke. A solid stand is also provided to place the hydraulic press on it. To connect the plunger with piston, a threaded cup is attached at the bottom of the piston. The overall capacity of hydraulic press is 100 tons, and is shown in Figure 4.10.
Hydraulic press control Unit

To carry out ECAP experiments, a hydraulic control unit is used as shown in Figure 4.11. The pressure can be increased with the help of four wet type solenoid directional control valves. The heat exchanger having rating of 300 psi with $350^\circ$ F is also fitted with the control unit to maintain the temperature of hydraulic oil. The ram displacement is controlled electronically by placing two limits switches on the vertical column of the hydraulic press shown in Figure 4.10.
4.5 Testing specimens materials

Aluminum 6061 is selected for the proposed research work but to observe deformation and orientation in axis high elastic extruded plastic materials are selected at initial stage. Teflon and nylon are selected for the verification of the mathematical model by evaluating the shear strains and orientation in axis as visualization of deformation in Al-6061 at high temperature is very difficult. The physical properties of the above explained materials have been provided in section below.

**Nylon**

The condensation reaction between amino acids, dibasic acids and diamines produces Nylon. It is first developed in 1928 by Wallace Carothers and is considered to be the first engineering thermoplastic. It is made of many
heterochain thermoplastics which has atoms other than carbon (C) in the chain [167].

Following are the properties of Nylon:

- The maximum temperature of nylon is 210°F or 99°C; whereas, minimum temperature is -94°F or -70°C.
- Its melting point is 420°F or 216°C.
- Tensile strength of Nylon is 5,800 psi (39.99 MPa.)
- Nylon is excellent material for machining.
- It is tough, strong, and impact resistant material.
- Very low coefficient of friction

**Teflon**

Teflon (Poly-tetra-fluoro-ethylene) is a fluorocarbon-based polymer and is commonly abbreviated as PTFE.

Following are the properties of Teflon:

- Low and high temperature capability.
- Low friction resistance.
- It has excellent thermal and electrical insulation properties.
- It has tensile strength of 1310 - 4350psi (9 - 30 MPa).
- Its compressive strength is 1450 - 2180 psi (10 - 15 MPa).
- The melting temperature of Teflon is 335°C.
Al-6061 alloy

The material used for research work is Al-6061 alloy, commercially used in aerospace and automotive industries. Typical properties of Aluminum alloy 6061 include:

- It has medium to high strength.
- It contains good toughness.
- It offers excellent machinability and surface finish.
- Aluminum 6061 contains excellent corrosion resistance at atmospheric conditions.
- It has good corrosion resistance to sea water.
- It can be anodized.
- It contains good weld ability and brazability.

Some of the physical properties and mechanical properties of Aluminum 6061 is provided in Table 4.1.

Table 4.1: Mechanical properties of Al-6061

<table>
<thead>
<tr>
<th>E (GPa)</th>
<th>Tensile Strength (MPa)</th>
<th>Yield Strength (MPa)</th>
<th>Poisson’s Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>70</td>
<td>310</td>
<td>270</td>
<td>0.33</td>
</tr>
</tbody>
</table>

Before conducting experiments, the purchased material is chemically analysed through XRF. The observed chemical composition is shown in Table 4.2.

Table 4.2: Chemical composition (wt. %) of the Al-6061 alloy

<table>
<thead>
<tr>
<th>Element</th>
<th>Mg</th>
<th>Fe</th>
<th>Si</th>
<th>Cu</th>
<th>Mn</th>
<th>V</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight%</td>
<td>1.08</td>
<td>0.17</td>
<td>0.63</td>
<td>0.32</td>
<td>0.52</td>
<td>0.01</td>
<td>0.02</td>
<td>Remainder</td>
</tr>
</tbody>
</table>
4.6 Preparation of specimens and testing

4.6.1 ECAP specimens

The specimens for ECAP are prepared from as purchased material. The purchased material of Aluminum was in the shape of a plate with thickness of 22 mm. From that plate specimens of dimension 20x20x120 mm$^3$ are prepared as shown in Figure 4.12.

![Figure 4.12: Isometric view of ECAP specimen of Aluminum 6061](image)

As explained in previous section, Teflon and Nylon specimens with same dimensions as of Al-6061, are used for the determination of deformation after ECAP process and for the verification of mathematical model by evaluating shear strain and orientation in axis, shown in Figure 4.13.

![Figure 4.13: ECAP specimen of Teflon material](image)

To measure material deformation, the specimens with cross-section of 20 x 20 mm$^2$ are further cut into two pieces with cross-section of 10 x 20 mm$^2$. Five numbers of circular grooves with 16 mm diameters are engraved in the
specimens. These grooves are filled with lead shots as shown in Figure 4.14 and Figure 4.15 for Teflon and Nylon materials respectively.

Figure 4.14: Un-deformed specimen of Teflon

Figure 4.15: Un-deformed specimen of Nylon

After filling lead shots in the grooves, both parts are re-joined and inserted into the ECAP die inlet channel to process. The main objective of these lead shots is to create a permanent impression in the specimens after ECAP and also to reflect a very clear deformation and orientation.

4.6.2 ASTM-E8 standard tensile specimen

For Teflon, Nylon and Al-6061 materials, dog-bone shaped specimens are machined by the milling operation. The dimensions of the specimens are set according to ASTM- E8 standard [168]. The thickness of all specimens is kept equal to 6 mm. The geometry of the specimens is shown in Figure 4.16.
4.6.3 ASTM-E647 standard three-point bend fatigue specimen
Fatigue test are performed on as received and post-ECAP specimens of Al-6061. Single edge notched specimens are prepared by EDM. The specimens are prepared according to ASTM standard E647 [169]. The single edge notch is developed to hold the clip-on-gage for measuring crack mouth opening displacement (CMOD). With the help of CMOD stress intensity factor is determined. The 3-point bend fatigue test specimen is shown in Figure 4.17.

4.6.4 Metallographic Specimen
For metallography, the specimens are grinded using emery papers of different grades ranging from 350 to 2500 µm, after grinding, polishing is performed using diamond past of 1 µm and buffing paper. When mirror like surface finish is
achieved, the specimens are etched in a solution of 1 gm NaOH, 4 gm KMNO₄, and 95 gm distilled water for metalography.

4.6.5 Fractographic specimen
Microscopy is performed using optical and scanning electron microscopes on the fractured specimens obtained from tensile test and 3-point bend test.

4.6.6 Hardness specimens
Vickers and microhardness tests are performed on as-received and ECAP specimens to check the hardness. For microhardness testing, the specimens are polished mirror-like using diamond paste as per ASTM standard E384 [170].

4.7 ECAP specimen testing
The ECAP experiments performed on materials have been discussed in detail in this section. The ECAP experiments are performed on the following materials:

- Nylon
- Teflon and
- Aluminum 6061

At initial stage, the experiments are performed on Nylon and Teflon due to their high elastic-plastic behaviour to check the die performance and deformation behaviour due to shear strain. After performing ECAP on above mentioned material, ECAP of the focused material Al-6061 is carried out.

The ECAP testing is performed at different temperatures. A set of specimens are prepared for each of the materials (Nylon, Teflon and AA-6061) and pressed through ECAP die. When these specimens are pressed through ECAP die the engraved circular slots (Nylon and Teflon) are converted into an elliptic shape
with inclined orientation as shown in Figure 4.18, 4.27, and 4.28, Nylon, Teflon, and Aluminum respectively.

Figure 4.18: Formation of Ellipse of Nylon processed by ECAP

Figure 4.19: Formation of Ellipse of ECAP processed Teflon specimen

Figure 4.20: ECAP processed specimen of Al-6061 processed
Analysis of ECAP specimens

In order to find out the rotation angle in the ECAP specimens of Teflon and Nylon, the image of deformed specimens are imported into graphical designing software AutoCAD™ by using “imageattach” command.

In above mentioned software, arcs are drawn along the contour of the specimens at their both sides (above and below). Polygons are drawn coinciding with these arcs; the diagonals of these polygons are drawn with the help of lines. The minor and major axis are also drawn along the deformed shape of the circle, converted into ellipses, as shown in Figure 4.21 and Figure 4.22, and then the rotation angle is determined shown in Table 6.12.

After completing preliminary experiments on Teflon and Nylon for verification of experimental setup, actual study is performed using Al-6061.

Figure 4.21: Image Analysis of ECAP processed of Nylon specimen
4.8 Testing of specimens

To evaluate the effect of ECAP process on the characteristics of Al-6061, a variety of tests are performed. These tests include:

1. Tensile test
2. 3-point bend fatigue test
3. Metallographic test
4. Fractographic test
5. Vickers hardness test
6. Microhardness Test

These tests are performed on as-received and ECAP specimens for comparison purposes.

4.8.1 Tensile Test

Tensile tests are conducted on dog-bone samples, made from Nylon, Teflon and Al-6061, to determine yield strength, shear stress, shear strain and ultimate tensile strength. All experiments are performed in a servo-hydraulic, closed-loop mechanical test machine (MTS) with 100 kN capacity, interfaced to a computer.
for machine control and data acquisition as shown in Figure 4.23. All tests are conducted in air and at room temperature. The machine has the capability to perform experiments at different frequencies ranges from 1 Hz to 60 Hz. The machine possesses the capability to perform a variety of tests including tensile test, compression test, bend test, fracture test and fatigue test. It is equipped with a variety of accessories such as grippers, fixtures and CMOD clipper.

![Material testing system MTS-810](image)

**Figure 4.23: Material testing system MTS-810**

### 4.8.2 Manipulation for MTS Useful Data

When tensile testing is performed on MTS, thousands of data points are generated for each load cycle. These data points are reduced by data filtration using MS Excel database. The data points having abnormal values are filtered
out. These filtered out data points are used to determine stress intensity factor and FCGR, which are presented in Chapter 6.

4.8.3 Hardness Test

Hardness of the as-received and post-ECAP material is determined by Vickers and micro-hardness tests, explained below.

**Vickers hardness test**

To check the hardness of as-received and ECAP specimens of Al-6061 alloy, universal hardness testing machine is used. This machine has the capability to perform Brinnel, Vickers and Rockwell hardness tests as shown in Figure 4.24. The machine has the capacity to apply load ranging from 98 N to 1831 N.

![Universal hardness tester](image_url)

*Figure 4.24: Universal hardness tester*
Micro-hardness test

Micro-hardness tests are performed on as-received and ECAP specimens, across the length of specimens. These tests are performed on Leco 400T micro-hardness tester at 100 gram load. Micro-hardness tests are carried out at different locations throughout the length of specimen. The results of Micro-hardness tests are discussed in the chapter 6.

4.8.4 Fatigue crack growth measurement

Crack growth in 3-point bend fatigue test is measured visually on MTS shown in Figure 4.23. The crack length is measured using a travelling microscope attached with digital vernier caliper. Collection of data is initiated after initiating the crack at the notch tip. The tests are performed in load control mode. Thus crack length is measured visually after given a number of load cycles. To follow the COD (crack opening displacement) standard procedure, a notch is created in the specimens. The maximum fluctuating fatigue load applied to test the specimens is kept below 20% of the elastic limit of the Al-6061. Due to small applied stresses, the crack propagation remained elastic-plastic according to LEFM theory.

The obtained results are evaluated and crack ratio (a/w), for every crack lengths, is determined. The plots of crack opening displacement and fatigue crack growth have been shown and explained in chapter 6.

4.8.5 Three-point bend fatigue test

3-point bending tests at different stress ratios (R= 0.1 and 0.7) are performed on as-received and ECAP specimens of Al-6061. The specimens having 16.3 mm width, 6 mm thickness, and 84 mm length are machined and prepared as shown
in Figure 4.25. The experimental set up of 3-Point bend fatigue test is shown in Figure 4.26.

![Image of specimen for 3-point bend fatigue test](image1)

**Figure 4.25: Specimen for 3-point bend fatigue test**

![Image of experimental setup](image2)

**Figure 4.26: Experimental setup of 3-point bend test**

The specimen span length of 75 mm is kept between supports. The crack length is measured with the help travelling microscope fitted with digital vernier caliper and maximum and minimum values of strains are recorded with the help of clip-on gauge, mounted in the knife edge of the specimen. High Cycle Fatigue (HCF) tests are performed on MTS at different stress ratios (R = 0.1 and 0.7) with
maximum load of 1.25 kN, and at frequency of 10 Hz. The crack measurement arrangement for three Point bend fatigue test is shown Figure 4.26, in which an optical microscope is mounted with vernier caliper for magnification purpose so that fatigue crack visualized. With the help of this travelling microscope installed on the MTS machine, crack lengths are measured visually after certain number of cycles.

4.8.6 Microscopy

The microstructure of the material before and after ECAP is obtained and fractographic investigations of the broken specimens after tensile and fatigue tests are performed.

Metallography

The chemical etching is performed according to AISIE 407 [171] standard to obtain clear grain boundaries, grain sizes and precipitate particles. For metallography, the specimens are grinded using emery papers of different grids ranging from 350 to 2500 µm. and then polishing is performed using diamond past of 1 µm and buffing paper to obtained mirror like surface finish.
After achieving a mirror like surface finish, the specimens is etched in 1 gm. NaOH, 4 gm. KMNO$_4$, and 95 gm. distilled water. The surfaces of the specimens so prepared are examined through Optical microscope, metallurgical microscope BX-RLA2 (Figure 4.27) as well as by SEM, TSCAN VEGA3.

**Fractography**

Fractography of the broken specimens by tensile and fatigue test are carried out using SEM at different magnifications. The behaviour of crack initiation, propagation and rupture is studied and discussed in chapter 6.

**4.9 Summary**

In this chapter the fabrication of the hydraulic press, die and plunger is completely described. An electric furnace is installed on the bed of hydraulic
press for heating die and billet for the smooth flow of material through ECAP die. Preparation of the specimens for ECAP process, made of Nylon, Teflon and Aluminum alloy 6061 are detailed. From the ECAP material, specimens are prepared for material characterization such as tensile testing, hardness measurements and 3-point bend fatigue tests. The metallographic procedure for the determination of ECAP effects on the grain structure is described in detail. Also the fractured surfaces of the as-received and ECAP materials are investigated for crack growth behaviour under different types of loading conditions. The procedure for the determination of shear strain in terms of change in geometry is also discussed.
5 Numerical Simulation

5.1 Introduction

With the progress and advancement in the field of information technology and computer sciences, numerical simulation has made it possible to deal with complicated real-time problems. Simulation means to examine the performance of the components or systems working under different loading conditions over a period of time by applying suitable boundary conditions. This is used to reproduce the performance and behaviour’s criteria of the elements or the process by using application software [172]. Simulation has been used in variety of fields i.e. engineering, medical, entrainment, and military, to check the behaviour of the system or components under different proposed conditions [173]. In this chapter numerical simulation is discussed in detail using ABAQUS® 6.10.1 on ECAP process. The numerical simulation was performed to validate the mathematical model by evaluating shear strain and orientation in axis when processed through ECAP [174, 175]. The work flow diagram of this chapter is shown in Figure 5.1.
5.2 Numerical simulation procedure

Numerical simulation of ECAP was performed on three dimensional specimens made from Nylon, Teflon and Al-6061 alloy. Teflon and Nylon were selected due to their high elastic behaviour for the verification of mathematical model. After verification of the model it was applied on Al-6061 and shear strain was
determined at different nodes along major and minor axis of the deformed specimen.

The ECAP process was modelled and simulated using ABAQUS/Explicit™. Initially the geometric model with the dimensions of 20x20x120 mm³ was modelled. Flow behaviour of material and orientation in the axis was observed by numerical simulation. For tracing and visualizing the behaviour of material, a circular impression was developed on the specimen surface as shown in Figure 5.2. Numerical simulation was performed on Nylon, Teflon and Al-6061 alloy. Properties of these materials are given in Table 5.1.

![ECAP specimen for numerical simulation with circular rings](image)

**Figure 5.2: ECAP specimen for numerical simulation with circular rings**

<table>
<thead>
<tr>
<th>Name of material</th>
<th>Modulus of Elasticity (GPa)</th>
<th>Density kg/m³</th>
<th>Poisson’s ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-6061</td>
<td>70</td>
<td>2700</td>
<td>0.33</td>
</tr>
<tr>
<td>Teflon</td>
<td>1.2</td>
<td>2200</td>
<td>0.46</td>
</tr>
</tbody>
</table>
Experimental values of plastic shear strain for both of the materials were used for numerical simulation obtained from tensile tests.

5.2.1 Boundary conditions

Detailed finite element analysis was performed on a 3D rigid model shown in Figure 5.2. All degrees of freedom of the die were constrained whereas the plunger had been given a velocity \( V_2 = 0.5 \text{ m/sec} \) in negative \( y \)-direction with the constraints along \( X \) and \( Z \) axis.

5.2.2 Meshing

The billet was meshed with linear hexahedral element type C3D8R having 15977 number of elements, total nodes of 18522, an 8-node linear brick, reduced integration, hourglass control element with approximate size of 0.1 mm. The geometry of the brick element with eight nodes is presented in Figure 5.3. The meshed specimen is shown in Figure 5.4, containing circular rings.

Figure 5.3: Geometry of brick element with 8 nodes
Die was considered as rigid body, meshed with discrete rigid element R3D3 with 0.5 element size, a 3-node 3-D rigid triangular facet. The boundary conditions for die were “Encaster”. The plunger was meshed with discrete rigid element R3D4, A 4-node 3-D bilinear rigid quadrilateral just to increase the computation speed. The plunger was given a boundary condition with velocity of 0.5 m/sec in negative y-direction. The change in shape of the specimen and formation of ellipse was due to the rotation of axis, when pressed through ECAP die. The specimen with die and plunger is shown in Figure 5.5.
5.2.3 Single ECAP pass deformation simulation

The flow behaviour of the materials is shown in Figure 5.6 when pressed through the ECAP die as a rigid perfectly plastic. The predicted flow pattern reflected by the figure is in agreement with the geometrical deformation behaviour discussed in article 2.3.3, chapter 2. The leading and trailing ends of the billet CDE and ABF alternatively maintained their triangular shapes and were only slightly deformed as shown in Figure 2.17. However, finite element meshes in the middle section were uniformly sheared, as predicted, after passing through the deformation zone. Sharp and straight strain contours localized at channel intersection revealed that the deformation zone was very narrow and almost confined to the shear plane.

![Figure 5.6: Nylon deformed specimen by ECAP](image)

First, the leading billet-end tended to be separated from the die surface. Second, the billet completely fills the lower left die corner and there was no gap appeared between the billet and the die corner. The round die corner had an effect on the deformation. The inner radius of the die has dominant effect as compared to outer radius. The RPP specimens developed uniformly distributed strain along the cross section for both channels.
The frictional forces experienced by the billet surface were expected to resist the extrusion and produce a larger deformation near the bottom side where the friction force was large enough. For the RPP billet, when coefficient of friction was kept moderate, the effective strain rose up to 2.33 at the bottom surface and decreased to a stable value of 1.12 at the 25% of the billet width[24].

5.2.4 Effect of ECAP on the specimen ends

The leading edge of billet was uplifted when exited from the die processed through ECAP and upper end of the specimen moved backward slightly. The shape of circular rings, engraved in the specimens, was converted into elliptical shapes as shown in Figure 5.6. It means that the specimen was forced to move towards the opposite end of the billet along the bottom and upper surfaces of die respectively.

Segal et.al [1] worked experimentally and studied deformation behaviour of the ECAP specimens and noted that macro flow pattern has four different regions that depend on the direction of the flow. Segal explained graphically the flow direction of ECAP specimen processed through multi pass in ECAP channel. The formation and development of a uniform deformed region in the middle of the specimen is shown in Figure 5.7. At the start, the region appeared as a six sided polygon and with progress of the process this was converted into four sides. The width of the specimen remained constant up to 3rd pass, but after that, width of specimen starts to decrease.
5.3 Summary

In this chapter, numerical simulation of two polymeric materials (Nylon and Teflon) and Al-6061 is performed to understand the behaviour of material when processed through ECAP. For this purpose, the material behaviour after ECAP was studied by introducing circular rings in the simulation model as it was done experimentally. Simulation was also performed on Al-6061 to determine shear strains at different nodes along minor and major axis and orientation developed in the ECAP model. A die containing two channels intersection at 90° is taken rigid and plunger is given downward motion at the velocity of 0.5 m/s. Angle rotation and ellipse axes completely described the shearing behaviour of the selected material after ECAP process.
6 Results and Discussion

6.1 Introduction

Results obtained from the series of experiments performed during this research work, are presented and discussed in this chapter. As highlighted earlier, material behaviour processed by ECAP under severe deformation conditions is the focus of this work. Tensile tests performed on Teflon, Nylon and Aluminum alloy (as-received and ECAP) are presented and discussed. Teflon and Nylon were used to develop a mathematical model of ECAP strain as tracking the deformation of Aluminum at 450°C is very difficult. These results are consequently used in numerical simulations. Material characterization was done and analysed to investigate the metallurgical changes on as-received and ECAP processed material. Tensile strength, yield strength, Vickers hardness, micro hardness and fatigue crack propagation properties were evaluated. The results thus obtained were compared with literature and conclusions drawn.

The fractured surfaces and crack paths were observed to determine the failure mechanism under varying loading conditions. Micro-structure analysis was carried out on as received and ECAP specimens. The detail work flow of this chapter is illustrated by Figure 6.1.
6.2 Mechanical properties of tested specimens

The results of different test performed on different types of specimens are discussed and noted in the following sections.
6.2.1 Nylon and Teflon tensile test for ECAP model verification
Tracking the deformation behaviour of Aluminum at 450°C is a difficult task. Such tracking helps to identify strain and orientation in axis, which are necessary input for numerical simulation. To overcome this difficulty, tracking was traced in Teflon and Nylon through engraved circular markings as explained in Chapter 4. The plastic strain observed through tensile tests of Nylon and Aluminum was used in ABAQUS™ for ECAP simulation. The experimental yield strength and ultimate tensile strength of Teflon and Nylon are shown in Table 6.1.

Table 6.1: Tensile test results of Teflon and Nylon

<table>
<thead>
<tr>
<th>Specimen Type</th>
<th>Yield Strength (MPa)</th>
<th>Ultimate Tensile Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Teflon</td>
<td>8</td>
<td>18</td>
</tr>
<tr>
<td>Nylon</td>
<td>14</td>
<td>30</td>
</tr>
</tbody>
</table>

6.2.2 Aluminum tensile test
Al-6061 alloy was used for mechanical characterization. Engineering stress-strain curves of as-received and ECAP specimens of Al-6061 material are shown in Figure 6.2. The curve reveals that there is considerable increase in yield strength and ultimate tensile strength after performing ECAP.
A combined stress strain curve is also presented in Figure 6.3 for the purpose of comparisons. The tensile test results are shown in Table 6.2. The experimental results show that:

- Yield strength increased by 15% approximately
- Vickers hardness by 25% and
- Tensile strength by 35% of ECAP specimens.


Table 6.2: Comparison b/w tensile and hardness results of Al-6061 after single pass found in literature

<table>
<thead>
<tr>
<th>Researcher</th>
<th>Yield Strength ($\sigma_y$) (MPa) (Al-6061)</th>
<th>%age increase in $\sigma_y$</th>
<th>Ultimate Tensile Strength ($\sigma_u$) MPa (Al-6061)</th>
<th>%age increase ($\sigma_u$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>As-is</td>
<td>ECAP</td>
<td></td>
<td>As-is</td>
</tr>
<tr>
<td>Kaka Ma et al. [40]</td>
<td>270</td>
<td>270</td>
<td>---</td>
<td>310</td>
</tr>
<tr>
<td>Valiev et al. [35]</td>
<td>270</td>
<td>300</td>
<td>11</td>
<td>310</td>
</tr>
<tr>
<td>N. A. Anjum</td>
<td>270</td>
<td>310</td>
<td>15</td>
<td>310</td>
</tr>
</tbody>
</table>

6.2.3 Numerical analysis of ECAP process

The deformation behaviour of specimens when processed through ECAP using ABAQUS™ was investigated. In order to check the deformation behaviour circular slots were engraved in the Teflon and Nylon specimens. After passing
the specimens through ECAP die the well circular slots were converted into elliptical forms as shown in Figure 6.4. The lengths of the major and minor axis of so formed ellipse were measured and recorded. The rotation, produced in the ellipse after passing through the ECAP die, was measured and listed.

The coordinate system was determined according to which the rotation angles were measured using digital image processing. Distance formula was applied to determine the lengths of major and minor axis. The comparison between graphical and experimental ellipse is shown in Figure 6.4

![Figure 6.4: (a) Analytically calculated ellipse   (b) Experimentally observed ellipse](image)

**6.2.4 Area comparison**

The areas of graphically and experimentally observed ellipse were evaluated and presented in Table 6.3.

**Table 6.3: Areas comparison of deformed and un-deformed specimens**

<table>
<thead>
<tr>
<th>Area calculation method</th>
<th>Major radius (a) mm</th>
<th>Minor radius (b) mm</th>
<th>Area mm²</th>
<th>Percentage error in area</th>
</tr>
</thead>
<tbody>
<tr>
<td>Analytically</td>
<td>15.22</td>
<td>3.20</td>
<td>153</td>
<td>4%</td>
</tr>
<tr>
<td>Experimentally</td>
<td>11.00</td>
<td>4.25</td>
<td>147</td>
<td></td>
</tr>
</tbody>
</table>
6.2.5 Determination of minor and major axis of ECAP specimens

To evaluate shear strains in the ECAP specimens, the major and minor axes were determined using AutoCAD. Dimensions of major and minor axis were determined by counting number of pixels for deformed specimens of Nylon and Teflon. The procedure for the determination of ellipse dimensions is given in the following sections.

6.2.5.1 Nylon Un-deformed specimen

Equation 6.1 was used to evaluate the distance ‘D’ between the coordinate points.

\[ D = \sqrt{(X_2 - X_1)^2 + (Y_2 - Y_1)^2} \]  \hspace{1cm} (6.1)

Figure 6.5: Un-deformed Nylon specimen circular rings filled with lead shot

Referring Figure 6.5, pixels were measured using an external computer application, the results were determined according to the procedure mentioned below for un-deformed and deformed specimens:

\[ X_1 = 1822 \text{ px} \quad Y_1 = 1145 \text{ px} \]
\[ X_2 = 1822 \text{ px} \quad Y_2 = 1593 \text{ px} \]

Total pixels = 448 px

Scale Conversion

No. of pixels per mm = \( \frac{(1625-1109)}{20} = 25.8 \text{ px/mm} \)
Where 20 is the width of the specimen.

Original Length = 448/25.8 = 17.36 mm

6.2.5.2 Nylon deformed specimen

The specimens of Nylon and Teflon were processed through ECAP die at temperature of 10, 50, and 100°C. Image analysis was performed on deformed specimen shown in Figure 6.6 to determine major and minor axis. The coordinates of major and minor axis were determined. Equation 6.1 was used to find out the lengths of major and minor axis as calculated below:

For Minor Axis of ellipse
\[X_1 = 946 \text{ px}, \quad Y_1 = 991 \text{ px}\]
\[X_2 = 1236 \text{ px}, \quad Y_2 = 1551 \text{ px}\]
The total pixels = 630 px

For Major Axis of the ellipse
\[X_1 = 1254 \text{ px}, \quad Y_1 = 1183 \text{ px}\]
\[X_2 = 928 \text{ px}, \quad Y_2 = 1347 \text{ px}\]
The total pixels: = 365 px
Following this procedure, lengths of major and minor axis of every specimen made from Nylon and Teflon were calculated and results are summarized in Table 6.4.

Table 6.4: Evaluation of major and minor axis of Nylon specimens

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Major axis (X₁,Y₁)</th>
<th>Major axis (X₂,Y₂)</th>
<th>Minor axis (X₁,Y₁)</th>
<th>Minor axis (X₂,Y₂)</th>
<th>Distance (major axis)</th>
<th>Distance (minor axis)</th>
<th>Temperature °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>NY-1</td>
<td>1997,1101</td>
<td>1526,1572</td>
<td>1609,1163</td>
<td>1912,1436</td>
<td>666</td>
<td>428.5</td>
<td>10</td>
</tr>
<tr>
<td>NY-2</td>
<td>1452,1051</td>
<td>2015,1614</td>
<td>1834,1231</td>
<td>1596,1469</td>
<td>797</td>
<td>336.0</td>
<td>50</td>
</tr>
<tr>
<td>NY-3</td>
<td>946,991</td>
<td>1236,1551</td>
<td>1254,1183</td>
<td>928,1347</td>
<td>630</td>
<td>365.0</td>
<td>100</td>
</tr>
</tbody>
</table>

Figure 6.7: Teflon deformed specimen through ECAP

6.2.5.3 Deformed specimens of Teflon
Following the same procedure as mentioned in above article, Teflon specimens shown in Figure 6.7 were analysed for major axis and minor axis, and results are presented in Table 6.5.

Table 6.5: Results of major and minor axis of Teflon specimens

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Major axis (X₁,Y₁)</th>
<th>Major axis (X₂,Y₂)</th>
<th>Minor Axis (X₁,Y₁)</th>
<th>Minor axis (X₂,Y₂)</th>
<th>Distance (major axis)</th>
<th>Distance (minor axis)</th>
<th>Temp. °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>TF-1</td>
<td>867,1051</td>
<td>1155,1501</td>
<td>1173,1175</td>
<td>835,1393</td>
<td>534</td>
<td>530</td>
<td>10</td>
</tr>
<tr>
<td>TF-2</td>
<td>1683,1163</td>
<td>2061,1541</td>
<td>2042,1193</td>
<td>1737,1498</td>
<td>500</td>
<td>495</td>
<td>50</td>
</tr>
<tr>
<td>TF-3</td>
<td>1606,1164</td>
<td>1836,1624</td>
<td>1896,1311</td>
<td>1541,1488</td>
<td>528</td>
<td>510</td>
<td>100</td>
</tr>
</tbody>
</table>
6.3 Determination of shear strain in ECAP specimens

Form the values shown in Table 6.4 and Table 6.5 were used to evaluate shear strain produced in ECAP specimens and is discussed and listed below.

6.3.1 Nylon

Using the information given in Table 6.4, the strain produced during ECAP was evaluated and listed. Procedure for calculation of strain is as:

**Scale conversion**

No. of pixels per mm = (1641-1080)/20 = 28.05 px / mm

Total pixels (major axis) = 630 px

\[ L_f = \text{Change in length} = 630/28.05 = 22.45 \text{ mm} \]

Total pixels (minor Axis) = 365 px

\[ L_f , \text{Change in length} = 365/28.05 = 13 \text{ mm} \]

\[ \text{Strain} = \frac{\Delta L}{L_0} \quad (6.2) \]

Using equation 6.2, the strain along major and minor axis is calculated as:

\[ \varepsilon_1 (\text{major axis}) = (22.45-17.36)/17.36 = 0.29 \]

\[ \varepsilon_2 (\text{minor axis}) = (13-17.36)/17.36 = 0.25 \text{ (compressive)} \]

**Table 6.6: Shear strain produced in Nylon specimens**

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Distance (major axis) (px)</th>
<th>Distance (minor axis) (px)</th>
<th>( \Delta L ) (major axis) mm</th>
<th>( \Delta L ) (minor axis) mm</th>
<th>Strain (major axis)</th>
<th>Strain (minor axis)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NY-1</td>
<td>666</td>
<td>364.8</td>
<td>23.90</td>
<td>15.41</td>
<td>0.37</td>
<td>0.11</td>
</tr>
<tr>
<td>NY-2</td>
<td>797</td>
<td>336</td>
<td>25.90</td>
<td>10.90</td>
<td>0.49</td>
<td>0.37</td>
</tr>
<tr>
<td>NY-3</td>
<td>630</td>
<td>365</td>
<td>22.45</td>
<td>13.00</td>
<td>0.29</td>
<td>0.25</td>
</tr>
</tbody>
</table>

6.3.2 Teflon

The strain produced in Teflon during ECAP process is evaluated from the information given in Table 6.5 and noted in Table 6.7.
Table 6.7: Shear strain produced in Teflon specimens

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Distance (major axis) (px)</th>
<th>Distance (minor axis) (px)</th>
<th>ΔL (major axis) Mm</th>
<th>ΔL (minor axis) mm</th>
<th>Strain (major axis) ε₁</th>
<th>Strain (minor axis) ε₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>TF-1</td>
<td>534</td>
<td>530</td>
<td>25.735</td>
<td>25.542</td>
<td>0.4855</td>
<td>0.4494</td>
</tr>
<tr>
<td>TF-2</td>
<td>500</td>
<td>495</td>
<td>24.038</td>
<td>23.798</td>
<td>0.3995</td>
<td>0.3567</td>
</tr>
<tr>
<td>TF-3</td>
<td>528</td>
<td>510</td>
<td>20.887</td>
<td>20.565</td>
<td>0.2268</td>
<td>0.2008</td>
</tr>
</tbody>
</table>

6.3.3 Al-6061 alloy

Numerical simulation of Al-6061 was performed on same model shown in Figure 5.2 by assigning mechanical properties to the model. The values of shear strain were taken along major axis and minor axis of the deformed shape as shown in Figure 6.8

Figure 6.8: Al-6061 deformed specimen through ECAP

Shear strain was determined on different nodes along major and minor axis after performing numerical simulation using ABAQUS™ and results are tabulated in Table 6.8 and Table 6.9. The results shown are in good agreement with the mathematical model. The recovery angle ξ determined by three different methods (Mathematical, Numerical, & Experimental) are also listed in Table 6.8
Table 6.8: Shear strain produced in Al-6061 specimen along major axis

<table>
<thead>
<tr>
<th>Node No.</th>
<th>Coordinates x-axis</th>
<th>Coordinates y-axis</th>
<th>Shear strain</th>
<th>ξ (Numerical)</th>
<th>ξ (Theoretical)</th>
<th>ξ (Experimental)</th>
<th>%age Error (col 5 &amp; 6)</th>
<th>%age Error (col 6 &amp; 7)</th>
</tr>
</thead>
<tbody>
<tr>
<td>82</td>
<td>-0.0421365</td>
<td>0.0465192</td>
<td>0.771453</td>
<td>25</td>
<td>22.5</td>
<td>18.6</td>
<td>10</td>
<td>17.3</td>
</tr>
<tr>
<td>109</td>
<td>-0.0452028</td>
<td>0.0409009</td>
<td>0.803721</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>586</td>
<td>-0.0436232</td>
<td>0.0437286</td>
<td>0.780914</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5513</td>
<td>-0.0463493</td>
<td>0.0383861</td>
<td>0.834892</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5544</td>
<td>-0.0471661</td>
<td>0.0364929</td>
<td>0.84559</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 6.9: Shear strain produced in Al-6061 specimen along minor axis

<table>
<thead>
<tr>
<th>Node No.</th>
<th>Coordinates x-axis</th>
<th>Coordinates y-axis</th>
<th>Shear strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>-0.047811</td>
<td>0.0270846</td>
<td>0.874375</td>
</tr>
<tr>
<td>617</td>
<td>-0.048166</td>
<td>0.0272961</td>
<td>0.865277</td>
</tr>
<tr>
<td>604</td>
<td>-0.049203</td>
<td>0.0261913</td>
<td>0.867084</td>
</tr>
<tr>
<td>115</td>
<td>-0.049288</td>
<td>0.0286884</td>
<td>0.850151</td>
</tr>
<tr>
<td>610</td>
<td>-0.0481043</td>
<td>0.0261125</td>
<td>0.852313</td>
</tr>
</tbody>
</table>

6.3.4 Comparison between results

The tests carried out on different specimens show close correlation and little scatter in the strain measurements. The experimental results and forms of ellipse was compared with the simulation results for Al-6061 and Teflon. A close proximity in the simulation versus the experimental results can be observed as shown in the Figure 6.9.

![Figure 6.9: Comparison between numerical and experimental ellipse](image-url)
The strains were calculated by three different methods described below,

- Mathematical model.
- Image analysis using designing software and
- Numerical simulation using ABAQUS™.

The results calculated from above mentioned techniques are tabulated in Table 6.10 for the purpose of comparison.

Table 6.10: Shear strain obtained by different techniques

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Analytical shear strain (Standard)</th>
<th>Experimental shear strain (image analysis)</th>
<th>Numerical shear strain (FEA)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\varepsilon_1$</td>
<td>$\varepsilon_2$</td>
<td>$\varepsilon_1$</td>
</tr>
<tr>
<td>TF-1</td>
<td>0.4995315</td>
<td>-0.4995315</td>
<td>0.485507816</td>
</tr>
<tr>
<td>TF-2</td>
<td>0.3843754</td>
<td>-0.3843754</td>
<td>0.399494941</td>
</tr>
<tr>
<td>TF-3</td>
<td>0.2020742</td>
<td>-0.2020742</td>
<td>0.22681328</td>
</tr>
</tbody>
</table>

The results of the shear strain by image analysis and FEA analysis were compared with analytical analysis (standard values), the percentage error is given in Table 6.11.

Table 6.11: Percentage strain error

<table>
<thead>
<tr>
<th>Material</th>
<th>%age error Image analysis</th>
<th>%age error FEA analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\varepsilon_1$</td>
<td>$\varepsilon_2$</td>
</tr>
<tr>
<td>TF-1</td>
<td>2.808</td>
<td>10.04</td>
</tr>
<tr>
<td>TF-2</td>
<td>3.933</td>
<td>7.20</td>
</tr>
<tr>
<td>TF-3</td>
<td>12.24</td>
<td>0.06</td>
</tr>
<tr>
<td>Average</td>
<td>6.327</td>
<td>5.77</td>
</tr>
</tbody>
</table>

6.3.5 Rotation produced in ellipse during ECAP

ECAP processed specimens experienced shearing as well as rotation during the process. In order to find the rotation angle, the image files of deformed specimens of Nylon and Teflon were imported into AutoCAD. Drawing the arcs along the contour of the ECAP specimens and then minor and major axis were
drawn as shown in Figure 6.6, the rotation angles were determined and results are shown in Table 6.12.

Table 6.12: Rotation produced in specimens

<table>
<thead>
<tr>
<th>Specimen No</th>
<th>Temperature ºC</th>
<th>Rotation ºC</th>
</tr>
</thead>
<tbody>
<tr>
<td>NY-1</td>
<td>10</td>
<td>85</td>
</tr>
<tr>
<td>NY-2</td>
<td>50</td>
<td>85.71</td>
</tr>
<tr>
<td>NY-3</td>
<td>100</td>
<td>89.9</td>
</tr>
<tr>
<td>TF-1</td>
<td>10</td>
<td>86</td>
</tr>
<tr>
<td>TF-2</td>
<td>50</td>
<td>85</td>
</tr>
<tr>
<td>TF-3</td>
<td>100</td>
<td>101</td>
</tr>
</tbody>
</table>

6.4 Al-6061 specimen test results

6.4.1 Hardness test Al-6061

Hardness tests were performed on as-received and ECAP specimens to measure Vickers and micro-hardness as mentioned above article that hardness of Aluminum 6061 increased by 26% due to ECAP. The results of Vickers hardness are presented in Table 6.13

Table 6.13: Vickers hardness for single pass of Al-6061

<table>
<thead>
<tr>
<th>Specimen Type</th>
<th>Al-6061 as-received</th>
<th>Al-6061 ECAP</th>
<th>percentage increase</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardness (HRC)</td>
<td>27</td>
<td>34</td>
<td>26</td>
</tr>
</tbody>
</table>

Micro-hardness test was performed on as-received and ECAP specimens, across the length of specimens and results are shown Figure 6.10. The variation in micro-hardness of ECAP specimen is due to the effect of environment on heat treatment. The leading end of the specimen had a comparatively low micro-hardness as compared to trailing end, because the trailing end of the specimen remained in the ECAP die at a constant temperature up to the cooling of die. The die took approximately two hours to cool down to room temperature.
6.4.2 Three point bend fatigue test results of Al-6061

Members with crack-like flaws or defects, the fatigue life may be predicted using a crack growth rate analysis. Theoretically, based on an elastic material behaviour, a tension stress acting on a cracked specimen will open a crack but when the tension stress is removed, crack opening displacements return to zero.

Reliable fatigue crack growth prediction is critically important for safe design and maintenance of engineering structures subjected to cyclic loading. Usually, in such analyses the range of the stress intensity factor (SIF), $\Delta K$, is utilized as a crack driving force.

Experiments were performed in a servo-hydraulic, closed-loop mechanical test machine (MTS) with 100 kN capacity, interfaced to a computer for machine control and data acquisition. All tests were conducted in air and room temperature, at a frequency of 10 Hz. The specimens were supported on rollers
for 3-point bend fatigue tests. The crack length was measured using a travelling microscope attached with digital vernier caliper. Collection of data was initiated after initiating the crack at the notch tip. The tests were performed in load control mode.

For this purpose, 3-point bend test specimens of 6061 Aluminum alloy were used for fatigue testing. Single edge notched specimens were prepared by EDM from as-received and ECAP material. The specimens were prepared according to ASTM E647 standard. The single edge notch was developed to hold the clip-on-gage for measuring crack mouth opening displacement (CMOD). The data obtained from CMOD was used to determine stress intensity factor.

### 6.4.3 Measurement of crack length

The fatigue crack length was determined by mounting a microscope on MTS data acquisition system. The crack ratio \( a/W \) was determined according to ASTM E399-90 standard, as represented in equation 6.3 [176].

\[
\frac{a}{W} = 0.9997 - 3.95U + 2.982U^2 - 3.214U^3 + 51.52U^4 - 113.0U^5 \tag{6.3}
\]

Where

\[
U = \frac{1}{1 + \sqrt{\left(\frac{\dot{E} BVm}{P}\right) \left(\frac{4W}{S}\right)}} \tag{6.4}
\]

Where

\( E' = \) Effective young’s modulus in Pa \( (E' = E \) for plane stress & 

\( E' = E(1- \nu^2) \) for plane strain.

\( \nu = \) Poisson’s Ration and
S, B, W, and a are already defined.

The curve between crack ratio and stress intensity factor $\Delta K$ at $R=0.1$ is shown in Figure 6.11.

Crack ratio is the ratio between crack length (a) and width of the specimen (W).

Stress intensity factor $\Delta K$ is calculated by using equation 6.5. The crack initiated at lower values of $\Delta K$ in specimens made from as-received but having greater values to initiate in ECAP specimens. The reason is that ECAP specimen is having higher fracture toughness than as-received. The behaviour of crack length with stress intensity factor for stress ratio $R=0.7$ is shown in Figure 6.12.
6.4.4 Paris curve for 3-point bend test

The stress intensity factor of 3-point bend test of 6061 Aluminum alloy was determined using the following formula according to ASTM E399-90 standard [177]

\[
K = \left( \frac{PS}{3BW^2} \right) \times f\left(\frac{a}{W}\right) \tag{6.5}
\]

For 3-Point bend test specimen, the geometric correction factor \( f(a/W) \) is given in equation 6.6 [177].

\[
f(a/W) = 3 \sqrt{\frac{a}{W}} \left[ 1.99 - (a/W)(1 - a/W) \left( 2.15 - 3.93\left(\frac{a}{W}\right) + 2.7a^2/W^2 \right) \right] \]
\[
= \frac{2(1 + 2a/W)(1 - a/W)^{3/2}}{2(1 + 2a/W)(1 - a/W)^{3/2}} \tag{6.6}
\]

Figure 6.12: Variation in \( \Delta K \) with change in crack ratio (a/W) at R=0.7

Relation b/w crack ratio and \( \Delta K \)

ECAP and as-received specimens at R=0.7

AA-6061, 34HRC,
3-point bend, H/W=2,
t=2.0mm, θ=25°C, R0.7
The Paris curves for both as-received & ECAP materials were developed. During fatigue crack propagation, crack length was evaluated from the CMOD values acquired from MTS. The data points so obtained was used to plot Paris curves for these two types of specimens on a single sheet for comparison as shown in Figure 6.13.

From Figure 6.13 crack growth is slower in ECAP specimen as compared to as-received material. This slow rate of FCG is mainly due to the increase of strength of the material as processed through ECAP.

The threshold value ($\Delta K$) in the region of 19 MPa√m to 28 MPa√m, crack growth ($da/dN$) values are relatively stable. Stress intensity factor ($\Delta K$) values greater than 28 MPa√m, the crack growth rate reflects an abrupt acceleration in as-received specimens whereas ECAP specimen shows a different behaviour.
Stress intensity factor up to 31 MPa√m of $\Delta K$, acceleration in crack growth can be observed but for higher values of $\Delta K$ it slows down.

The relationship between as-received and ECAP specimens at stress ratio $R=0.7$ is shown in Figure 6.14.

![Log-log plot b/w $\Delta K$ Vs $da/dN$ at $R=0.7$ for as-received and ECAP of Al 6061](image)

Crack growth rate in ECAP specimens is slower at stress ratio $R=0.7$ compared to as-received. This slow rate of FCG is mainly due to the increase in mechanical properties of the material as processed through ECAP.

The threshold value ($\Delta K$) in the region of 20 MPa√m to 28 MPa√m, crack growth ($da/dN$) comparatively stable and $\Delta K$ values greater than 28 MPa√m, crack growth rate reflects an abrupt acceleration in as-received specimens whereas ECAP specimens, up to 28 MPa√m of $\Delta K$, an acceleration in crack growth can be observed but for higher values of $\Delta K$ it slows down.
It is concluded from the results that the increasing $\Delta K$ gives less scatter than decreasing $\Delta K$. This difference is due to interaction between cyclic plastic zones and their effect on fatigue damage.

### 6.4.5 Crack ratio as a function of crack mouth opening displacement

The growth of crack was very fast in as-received material as compared to ECAP. The comparison between crack ratio and crack length is shown in Figure 6.15. The difference between as-received and ECAP specimens processed at $R=0.1$, shows that crack closure is very slow in ECAP specimen. This difference may be due to the grains refinement and enhancement of mechanical strength due to severe plastic deformation.

The ASTM E399-90 standard cannot be applied to evaluate crack length using CMOD if large crack closure exists as this only permits up to a limited crack lengths [176].
Figure 6.15: Comparison b/w CMOD and crack ratio (a/W) at R=0.1

Figure 6.16: Comparison b/w CMOD and crack ratio (a/W) at R = 0.7
In Figure 6.16, the relationship between crack ratio \((a/W)\) and CMOD \(\delta\) is presented. Crack ratio \((a/W)\) of as-received material is lower than that of ECAP material, however, the slope of the trend line of as-received, representing crack ratio with respect to CMOD, is more which also seems to intersect the trend line corresponding ECAP material at stress ratio \(R0.7\).

### 6.4.6 Relationship of \(K_{\text{max}}\) with crack growth rate

The behaviour of the crack growth rate with respect to \(K_{\text{max}}\) for as-received and ECAP material is shown in Figure 6.17. The curves are showing that the crack is initiating at the approximate same value of \(K_{\text{max}}\), but FCGR of the ECAP material is relatively slower than that of as-received material, which shows that the toughness of the material is improved by ECAP process.

![Log-log Plot b/w \(K_{\text{max}}\) and crack growth \((da/dN)\), at stress ratio \(R=0.1\), Al 6061](image)

Figure 6.17: Fatigue crack growth rate vs \(K_{\text{max}}\) plot on log-log scale at \(R=0.1\)
The behaviour of the material can be observed in Figure 6.18 at stress ratio 0.7. The crack is initiated at the low value of stress intensity factor in as-received as compared to ECAP specimen. Crack grows very rapidly in as-received material, showing lower toughness as compared to ECAP material. The improvement in the fracture toughness by ECAP pressing is due to grain refinement.

6.4.7 Relationship b/w crack length and number of cycles

The graphs between crack length and number of cycles (N) for as-received and ECAP material at stress ratios R=0.1 and R=0.7 are shown in Figure 6.19 and Figure 6.20 respectively. It was observed that the crack initiation took place earlier in ECAP specimens at R= 0.1 and R=0.7 as compared to as-received but crack growth rate is faster in as-received specimens. It represents that toughness of ECAP specimen has increased.
Figure 6.19: Crack length Vs number of cycles (a-N) curve at stress ratio R0.1

Figure 6.20: Crack length Vs number of cycles (a-N) curve at stress ratio R0.7
6.4.8 Effect of crack driving force \((K^*)\) on fatigue crack growth

Fatigue crack growth rate can be estimated using crack driving force parameters for different R ratios discussed by Kujawski et al.[178, 179]. The crack driving force can be estimated by using equation 6.7 [180].

\[
K^* = K_{\text{max}}^\alpha (\Delta K^*)^{(1-\alpha)} \tag{6.7}
\]

Using equation 6.7 for \(\alpha = 0.0174\) for ECAP specimens and \(\alpha = 0.95\) for as-received specimens, the behaviour of fatigue crack growth closure is shown in Figure 6.21 and Figure 6.22. The mentioned figures show that all the FCGR curves collapse onto each other as the value of alpha \(\alpha\) is approaches to 0.0174 and 0.95 for ECAP and as-received specimens respectively.

![Log-log plot b/w K* and da/dN at different stress ratios, Al 6061](image)

Figure 6.21: Consolidation of fatigue crack growth data for R=0.1 and R=0.7, using the \(K^*\) parameter
These curves also reflect that by applying alpha (\(\alpha\)) as crack growth driving parameter eliminates the effects of crack closure and even in the presence of high crack closure rates it provides an integrated testing parameter. The equation 6.7 can be used for the prediction of crack growth rate for different stress ratios.

It is concluded that stress and strain inside plastic zone, causing fatigue damage, can be controlled by the elastic field around it. Existence of tensile stresses in the process zone is a necessary condition for fatigue crack propagation. The crack growth rate is slower in ECAP specimens as compared to as-received. The crack propagation is faster in as-received as compared to ECAP. This is due to increase in toughness, hardness, and yield strength because of grain refinement.

Figure 6.22: Consolidation of fatigue crack growth data for R=0.1 and R=0.7, using the \(K^*\) parameter
6.5 Microstructural analysis

This section is about metallographic and fractographic analysis of fractured surfaces tested for 3-point bend and tensile testing for both as-received and ECAP Aluminum 6061. The metallographic and fractographic analysis for as-received and tensile specimens was performed using optical microscope and scanning electron microscop (SEM). In metallographic analysis the effect of ECAP on microstructure was analysed and results obtained were compared with as-received material. Fractographic analysis was performed to check different parameters such as ductile failure, brittle failure, crack growth direction, slip bands, inclusions, etc.

6.5.1 Metallography

For optical analysis the specimens were ground and then polished with diamond paste and finally etched by Weck’s double reagent (KMnO₄-NaOH). Results presented here are obtained by optical microscopy of the as-received and ECAP specimens. The micrograph of as-received Aluminum alloy is shown in Figure 6.23, where grain and grain boundaries can be observed clearly.
The change in grain geometry and grain boundaries of ECAP specimen can be observed in Figure 6.24. It is noted that the grains are elongated by ECAP
pressing. The grain elongation is due to the shear straining of the material. Recrystallization has not been found by heat treatment. All mechanical characterisation is done on this microstructures.

### 6.5.2 Fractographic analysis

The fractography of the as-received and ECAP materials after tensile and fatigue testing is performed. The flow diagram of this analysis is presented in Figure 6.25.

![Fractography Flow Diagram](image)

Figure 6.25: Flow diagram of fractography.

Specimen preparation for metallography is discussed in Chapter 4. In Figure 6.26, the structure and behaviour of as-received (a) and ECAP (b) specimens under tensile loading is presented. As-received material had larger voids as compared to the ECAP material that is due to grain refinements. Also deep secondary cracks were observed in the bottom of voids in ECAP material shown by region A. The edges of the micro-voids in case of as-received specimens are
sharp, however the edges of the ECAP specimen were truncated and fine voids were formed due to plastic final failure.

Figure 6.26: Behaviour of as-received and ECAP specimen under tensile loading

Figure 6.27: Behaviour of river patterns of as-received and ECAP specimen under fatigue loading (3-point Bend)
Figure 6.28: Crack initiation for as-received and ECAP specimens under fatigue loading (3-point Bend)

Figure 6.29: Crack propagation for as-received and ECAP specimens under fatigue loading (3-point Bend)
In Figure 6.27, the river pattern representing the direction of crack propagation for as-received and ECAP material under fatigue loading are shown. It is observed that the flow of the river pattern is normal to the edge of the specimen; however it is extending from surface in the form of ripples.

In Figure 6.28, the crack nucleation due to fatigue loading of the as-received specimen is seen near the surface. Multiple crack initiation sites can be noticed from the surface for ECAP specimens.

In Figure 6.29, fractured surface at mid crack propagation region of as-received and ECAP specimens are presented. It is perceived inter-granular fracture is occurred in the as-received specimens, whereas trans-granular fracture took place in ECAP specimen. This response seems due to the breakage of grains by ECAP pressing.
In Figure 6.30, the formation of the cracks near the ECAP surface are detected which can be normal or parallel to the surface. These pre-existing cracks in the ECAP material can cause early crack initiation when subjected to fatigue loading. The effect of early crack initiation in ECAP specimen as compared to as-received specimen can also be observed in Figure 6.28.

6.6 Self-criticism

To accomplish this research work there were different types of limitations such as mathematical modelling, to trace recovery angle in Aluminum, speed of ram was also a problem. Teflon and Nylon were used to develop a mathematical model of ECAP strain as tracking the deformation of Aluminum at 450°C is very difficult. The mathematical modelling to determine orientation in angle, recovery angle after ECAP and determination of shear strains was another problem. Development of mathematical modelling remains a problem in the history of the engineers and always searched for better mathematician to develop mathematical model. The processing speed was another issue; in this research, the ram speed remains constant throughout ECAP process that was 9 mm/sec. The research work should be performed by variable speeds of ram to investigate its effects on grains. For this purpose, the hydraulic pump must be equipped with electronic speed controller so that speed of ram can be increased or decreased and should be investigated its effect on grain structure.

Limitations of ECAP

As mentioned in chapter one and two that conventional ECAP involves pushing a billet through two channels that meet at an angle of 90° Figure 2.6. For an ideal rigid plastic material, once the applied force reaches a critical value, the billet
material undergoes plastic deformation, and the process continues without further increase in load. When a metal billet is used, due to elastic deformation and the Poisson effect, there is a lateral expansion of the billet in the entrance channel, and very high frictional forces develop between the work-piece and the channel walls. The force required to press a billet through the die increases very rapidly with the length of the billet. Correspondingly, there is also a large increase in stresses experienced by the die. These two factors limit the length of the billet that can be practically processed by ECAP. At the start of this project, the largest billet that could be processed by ECAP was 20 mm square by 150 mm length at the Fracture Mechanics laboratory of Mechanical Engineering Department and most scientific studies had been conducted on samples 20 mm square made from Nylon, Teflon and Aluminum.

6.7 Summary

For material characterization of as-received and ECAP Aluminum alloy 6061, a comprehensive experimental layout was established explained in Figure 1.4. Fatigue and tensile tests were performed on the research material. The tensile and yield strengths of the material increased along with hardness after performing ECAP process. The 3-point fatigue bend tests were also performed at different stress ratios on as-received and ECAP specimens. The behaviour of the material was examined under fatigue crack growth rate. From the experimental results different curves were drawn to analyse their behaviour against different parameters. The metallography and fractography was also performed on as-received and ECAP specimens. The grain structure, grain boundaries were investigated and analysed.
7 Discussion of the Research Results

7.1 Introduction
This research work is carried out to study the effects of ECAP on cyclic and monotonic mechanical properties of Al-6061 and compare it with Al-6061-T6. The thesis starts with an introduction to different severe plastic deformation techniques followed by a literature review, mathematical modelling of ECAP, experimentation, mechanical characterisation and finally microscopy.

This research deals with the processing of Al alloy through severe plastic deformation to increase the monotonic (tensile) and fatigue strength of material. The process chosen is the ECAP process. Other process like, High pressure torsion, accumulative roll bonding, dissimilar angular pressing, multidirectional forging, etc. also exist.

7.2 Contribution to knowledge
When materials processed through SPD techniques, material grain size converted into ultrafine grains due to severe plastic deformation. This severe plastic deformation causes shear strain which cuts the grains along crystallographic planes which later recrystallize into very refined grains. The grain refinement as a function of plastic strain depends upon the following factors.

- Die geometry consisting die angle, die corner angle and channel sizes.
- Materials properties such as hardening behaviour and strength.
- ECAP process variables such as lubrication, speed of ram, temperature etc.
The research is based primarily on two parts. Initially, a mathematical model for the determination of strains in ECAP was developed which takes into account elastic recovery of materials after angular extrusion. Due to the difficulty in tracking strains and orientations in Al alloy, the model was verified using experimentation on Teflon and Nylon specimens engraved with circular markings. These specimens were passed through an ECAP die and the shear strains were measured using digital image processing. These shear strains were then compared with those in the ECAP model and conclusions drawn. Numerical simulation was also performed on Al-6061 and results obtained were compared with mathematical model and a close agreement was found.

In view of the ECAP die geometry, the required plunger load was decided through mathematical modelling.

The geometric parameters used for load calculation were plunger velocity ($v=0.5$ m/sec), friction energy ($W_f=6.062\Delta V$), deformation energy ($W_i=975.69V_o$), equivalent energy ($W_e=128217V_o$), friction between die wall and material (Tresca friction, $\bar{m}=0.3$), die angles ($\phi=90^\circ$, $\Psi=22^\circ$), flow stress ($\sigma_o=35$ MPa) of material, channel inner radius (2 mm) and outer radius (8.45 mm). The velocity of the plunger/billet was determined by segmenting the die channel into three different zones. After load calculations, a mathematical model was developed to evaluate the shear strain induced during ECAP process discussed in chapter 3.

Characterization of the processed alloy was performed on as-received and ECAP specimens. Tensile test, hardness test, micro-hardness test and 3-point bend fatigue test followed by microscopy and fractography is carried out.
Reliable fatigue crack growth prediction is critically important for safe design and maintenance of engineering structures subjected to cyclic loading. Usually, in such analyses the range of the stress intensity factor (SIF), $\Delta K$, is utilized as a crack driving force. The stress intensity factor $\Delta K$ is effected by the following factors:

- The load ratio $R$ (minimum load/maximum load)
- Crack closure
- Overload
- Crack size
- Environment
- Microstructure
- Geometry
- Temperature, etc.

Most load bearing components and structures experience both an alternating load and a mean load during their service application. The mean load effect on fatigue crack growth rate is commonly introduced through the load ratio $R$. Therefore, the ability to correlate and to predict the fatigue crack propagation rate, $\frac{da}{dN}$, for different $R$-ratios is of significant importance to design engineers.

### 7.3 Conclusions

1. Aluminum 6061 specimens, with square cross-section of 20x20 mm$^2$, were severely deformed by Equal Channel Angular Pressing (ECAP) technique with die angle 90° at 450°C.

2. Mechanical properties such as hardness, yield strength, & tensile strength of the material were observed and recorded.
3. It was noted that mechanical properties such as hardness, micro-hardness, yield strength and ultimate tensile strength of ECAP specimens were increased due to ECAP process because of grain refinement.

4. The Vickers hardness increased by 26%, Yield strength by 14% and ultimate tensile strength by 34.37%.

5. Microstructural analysis was also carried out for grain structures, grain boundaries along with fractography of as-received and ECAP specimens.

6. Fatigue strength increased after ECAP process.

7. Crack growth rate is faster in as-received material compare to ECAP.

8. Recovery angle is validated with mathematical model.

9. After performing experimentations, the metallography and fractography was also performed on as-received and ECAP specimens to observe grain structure and grain boundaries.

10. After testing it was observed clearly that as-received material had larger voids as compared to the ECAP specimens that were due to grain refinements.

11. Deep secondary cracks were also observed in the ECAP specimens.

12. The edges of the micro-voids in case of as-received specimens were found very sharp, whereas the edges of the ECAP specimens were truncated and fine voids were formed due to plastic final failure.

13. In as-received specimen cup and cone morphology was present showing a ductile failure whereas in ECAP specimens craters and flat areas were observed showing a mix mode failure (ductile and brittle).

14. Large pockets were also observed in ECAP specimens, in which the cleaved facets of intermetallic inclusions were recorded.
15. The river type patterns in as-received were recorded showing the direction of crack propagation whereas ripple type patterns in ECAP under fatigue loading were observed. It was also observed that the flow of the river pattern is normal to the edge of the specimen; however it is extending from surface in the form of ripples in ECAP specimens.

16. Multiple crack initiation sites were also noticed from the surface in ECAP specimens. Inter-granular fracture is occurred in the as-received specimens, whereas trans-granular fracture took place in ECAP specimen. This response seems due to the breakage of grains by ECAP pressing.

7.4 Future research direction
Current ECAP setup can be improved and modified for multi-passes for better understanding of mechanical characterization and investigation of the change in mechanical and physical properties of the processed materials. The effect of following factors during ECAP process can be included for detail analysis as:

1. The effect of friction between die walls and billets should be included in the research while processing through the ECAP die and use of different lubricant to minimize the friction effect.

2. The effect of processing speed should be investigated and analysed and its effect on grain size, grain boundaries, shape of grain should be included in the research work.

3. The effect of ECAP on different grade of Aluminum alloys must be experimentally analysed and comparisons should be drawn.

4. The effect of back pressure when processing through ECAP should also be investigated.
5. The effect of temperature on Aluminum alloy should also be investigated for grain size, grain shape, grains boundaries, hardness, strength etc.

6. The variation in hardness of the material should be taken by nano-indentation to avoid the influence of imperfection.

7. As discussed in the thesis that SPD techniques are used for the refinement of coarse grained materials into ultrafine grained materials. The mechanical and physical properties of the material changed completely. Among these properties temperatures, thermal coefficients, diffusion along with elastic properties are the most important properties of the materials. This change in material properties has a great effect on thermal, magnetic, electrical, optical and mechanical properties of the materials. These change in material properties exposed new research areas for future researchers.

8. A detailed FEM based study can be done for comprehensive information about the material modes of deformation, material flow properties, processing parameters and growth of texture.
REFERENCES


[16] H. Alihosseini, M. A. Zaeem, K. Dehghani, and H. Shivaee, "Producing ultrafine-grained aluminum rods by cyclic forward-backward extrusion:"


