

# A Study & Analysis on Effect of Ultrafine Grain Refinement on Mechanical Properties of Non-Heat Treatable Alloys

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## ABSTRACT

Among the procedure devised for grain refinement, severe plastic deformation is of particularly interest and the focus of this study. This project work deals with the concept of ECAP used to introduce severe plastic deformation in non-heat treatable alloy. In this work, the non-heat treatable alloy selected is aluminium low alloy 1100 for the study. Detailed FEM analysis has been done using commercial software DEFORM3D™ for single ECAP pass. Deep literature survey has done to understand correct method to do analysis of the billet using FEM. Results has calculated and collected for different die geometries i.e. die corner angle and die channel angle. Effect of die geometry on strain distribution and strain homogeneity has studied. Also the parameters are compared for in homogeneity index and corner gap introduced during ECAP process. Die has been manufactured using optimum die geometry combination to get better strain homogeneity as well as strain distribution through the billet material. Detail manufacturing process has discussed. Also the difficulties occurs during the experimentation has been explained.

**Keywords:** ECAP, Non-heat treatable alloy, FEM, Die geometry, Strain homogeneity.

## 1. INTRODUCTION

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 viable 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 study tells 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 P.W. Bridgman 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 [1, 2]. Bridgman 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”.

The most common process of SPD is the equal channel angular pressing (ECAP), which involves pressing a billet through a die consisting of two channels of equal cross sections, intersecting at an angle, typically 90°. The process of ECAP allows us to introduce very large plastic deformations to a work-piece without altering the overall geometry of the work-piece.

The aim of project is to study ECAP process, improvement in properties of non-heat treatable alloys, simulation, determination of optimum die geometry for ECAP, and validate the results experimentally.

Among the procedures devised for grain refinement, Severe Plastic Deformation (SPD) techniques are of particular interest and are the focus of the present study. These techniques enjoy great popularity owing to their ability to produce considerable grain refinement in fully dense, bulk-scale work-pieces, thus giving promise for structural applications. The achievable grain sizes lie within the submicrometer (100 – 1000 nm) and nano meter (<100 nm) ranges.

Equal channel angular pressing (ECAP) is a very interesting method for modifying microstructure in producing ultra-fine grained materials. It consists of pressing test samples through a die containing two channels, equal in cross section and intersecting at a certain angle. As a result of pressing, the sample theoretically deforms by simple shear and retains the same cross sectional area to repeat the pressing for several cycles. UFG materials exhibit both excellent strength at ambient temperature and, if the grains are reasonably stable, outstanding superplastic properties at elevated temperatures. These materials also have a high innovation potential for use in commercial applications. At low temperatures, the strength generally follows the Hall–Petch relationship [3] so that,

$$\sigma_y = \sigma_0 + K_y d^{-1/2} \quad \text{Eq. (1.1)}$$

Where,  $\sigma_y$  is the yield stress,  $\sigma_0$  is the lattice friction stress,  $K_y$  is a constant of yielding and  $d$  is the average grain size.

Every material has constant value for  $\sigma_0$  and  $K_y$ . Following table 1.1 shows values of constants  $\sigma_0$  and  $K_y$  for some polycrystalline material.

Element	Fe	Mo	Nb	Cu	Al	Zn
$\sigma_0$ (MPa)	48	110	126	26	16	33
Ky(MN/m <sup>3/2</sup> )	0.71	1.77	0.034	0.11	0.068	0.22

Table- 1: Values of constants

Thus, the strength of the material increases when the grain size is reduced.

For the materials in which strength cannot be improved by heat treatment or any other method, one way to improve its mechanical properties is to fine the grain size by severe plastic deformation, which can be done by ECAP. The mechanical properties of the ECAP processed material get improved by several amount, UTS get increased by 130-170%, Hardness by 110-150%, Elongation by 110-160% [4].

The die geometry is defined by the cross section area and the two angles  $\Phi$  and  $\Psi$ , the angle of intersection between the two channels, and the arc of curvature at the outer point of intersection respectively. It is possible to calculate, from the two angles, the shear strain or the effective von Misses strain resulting from pressing through the die. The advantage with the ECAP method is that it is possible to introduce severe plastic deformation (SPD) by repeated pressing of the billet without any significant change in the cross section. Altering the billet orientation after each press, thereby modifying the shear plane and shear direction, makes it possible to control the microstructure and texture of the material, thus, altering the mechanical properties.

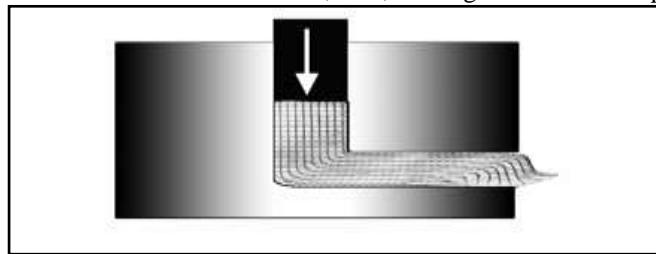


Fig-1: Sketch of ECA pressing [11]

### 1.1 ESTIMATION OF THE STRAIN IN ECA PRESSING

The shear strain  $\gamma$  for simple shear is defined as in Figure 3.4,  $\gamma = a/h$ . Simple shear involves a shape change produced by displacement along a single set of parallel planes. The shear strain introduced by the ECAP will first be derived for the case of simple shear, assuming a square cut die and neglecting the friction effects. Figure 3.3 shows a cubic element abcd along the centre line in the ECAP die. The die is defined by the angles  $\Phi$  and  $\psi$ . If we follow the element through the die, we will end up with the orthogonal element a'b'c'd', deformed by shear during the passage through the die. Following the notation in Figure 3.3, it follows that the shear strain  $\gamma$  is given by,

$$\gamma = \frac{a'u}{d'u} = \frac{rc' + as}{ad} = \frac{\Psi \cdot ad \cdot \operatorname{cosec}(\frac{\phi}{2} + \frac{\psi}{2}) + ad \cdot \cot(\frac{\phi}{2} + \frac{\psi}{2}) + ad \cdot \cot(\frac{\phi}{2} + \frac{\psi}{2})}{ad}$$

This reduced to,  $\gamma = \Psi \operatorname{cosec}(\frac{\phi}{2} + \frac{\psi}{2}) + 2\cot(\frac{\phi}{2} + \frac{\psi}{2})$  Eq. (3.2)

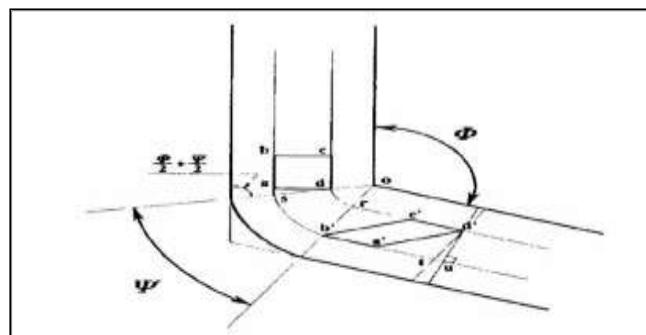
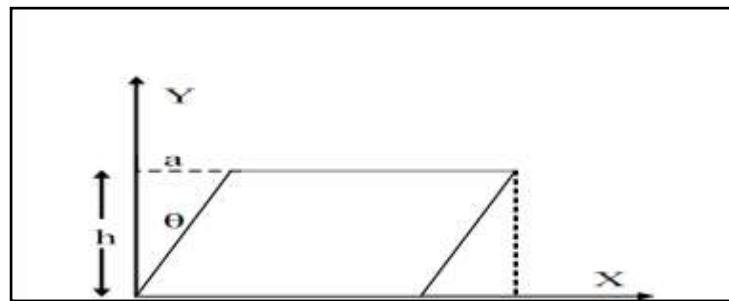


Fig-2: Schematic drawings of a deforming element moving through the ECAP die [11]



**Fig-3: Deformed rectangle element [11]**

Also, the magnitude of equivalent effective plastic strain ( $\epsilon_{eq}$ ) after N passes is given by the following relationship,

$$\epsilon_{eq} = \frac{N}{\sqrt{3}} \left[ 2 \cot\left(\frac{\Phi+\Psi}{2}\right) + \Psi \cosec\left(\frac{\Phi+\Psi}{2}\right) \right] \text{Eq.(3.3)}$$

#### A. Material selected for billet

##### 1100 (ISO)

1100 aluminium alloy is an aluminium-based alloy in the "commercially pure" wrought family (1000 or 1xxx series). With a minimum of 99.0% Aluminium, it is the most heavily alloyed of the 1000 series. It is also the mechanically strongest alloy in the series, and is the only 1000-series alloy commonly used in rivets. At the same time, it keeps the benefits of being relatively lightly alloyed (compared to other series), such as high electrical conductivity, corrosion resistance, and workability. It can be strengthened by cold working, but not by heat treatment.

#### B. Chemical composition

Table 2 gives maximum percentage of alloying element determined in the metallurgy lab using chemical analysis spectrometer.

Element	Si	Fe	Cu	Mg	Zn	Al
Percentage Content	0.2	0.6	0.1	0.05	0.03	99.00 min

**Table-2 Composition of 100 Al alloy**

#### C. Mechanical properties

1. UTS= 80 MPa.
2. Young's modulus (E) = 70 GPa
3. Elongation at break = 38%

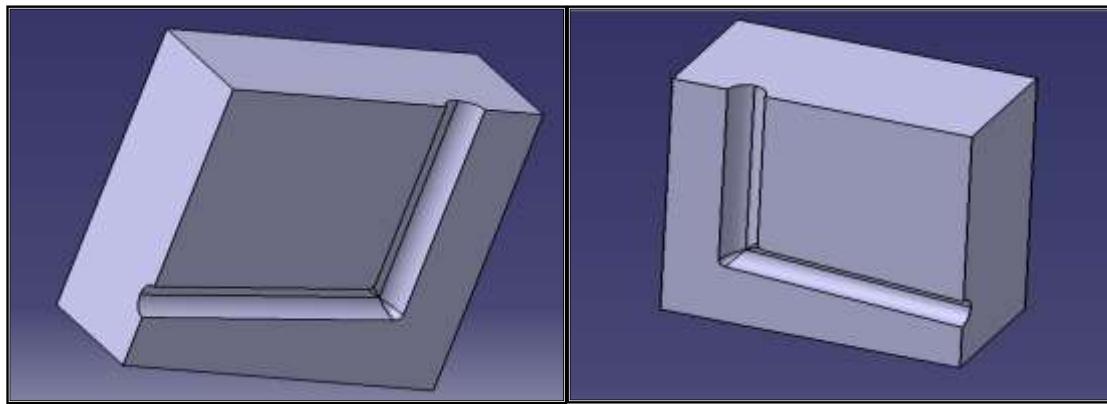
#### D. Applications

Aluminium 1100 alloy is widely used in fin stock, heat exchanger fins, spun hollowware, dials and name plates, decorative parts, giftware, cooking utensils, rivets and reflectors, and in sheet metal work.

## 2. GEOMETRIC MODELING AND SIMULATION

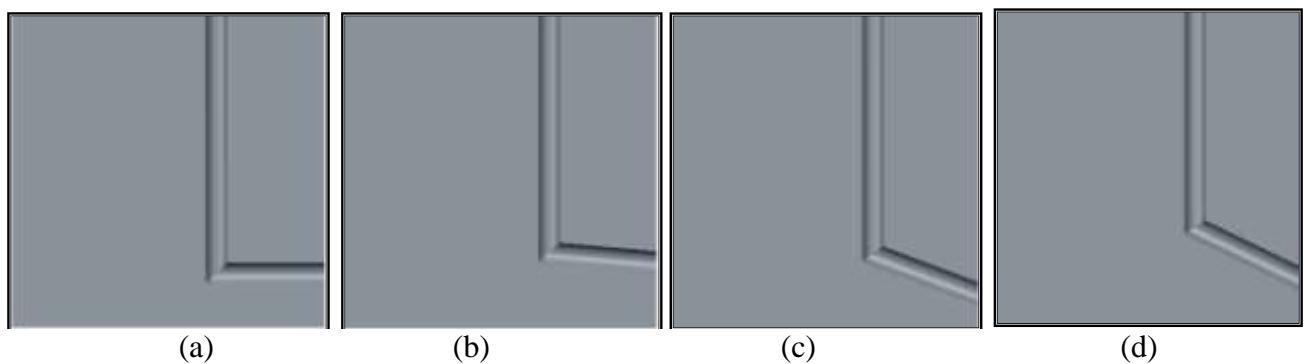
#### A. Geometric Modelling of Die

Die is modelled using part design in CATIA. The die is modelled into two symmetric parts as shown in Figure 4. At the time analysis the inner surfaces of die kept as bonded.



**Fig-4: Solid model of Die**

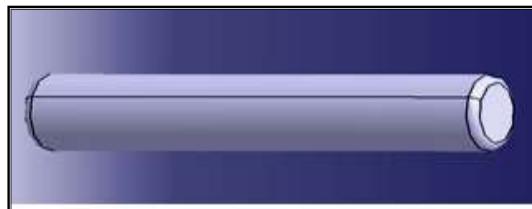
Modelling has done for constant angle  $\psi = 150$  and for  $\Phi$  with increment of 50 starting from 900 to 1200 as shown in Figure 5. To make the file suitable to import to the Finite element analysis software, model was saved with extension .part, .igs, .stp.



**Fig-5: Solid model of die with different channel angle (a)90° (b)95° (c)115° (d) 120°**

#### B. Billet

Billet was also modelled in module part design in CATIA with the specified dimensions, and saved with extension .part, .igs, .stp. The model is shown in Figure 5.3. Billet dimensions Diameter = 9.8 mm, Length = 100 mm.



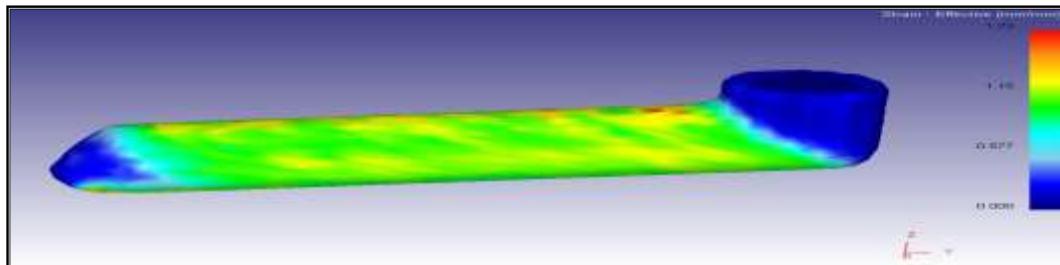
**Fig-6: Billet modelled in CATIA**

#### C. Simulation using DEFORM3DTM

For choosing the optimum die dimension as well to find out the stress developed in the billet during the process, the amount of force required to press the billet through the die channels for number of channel angles, simulation has done in commercial FEM software DEFORM3DTM-F23. Results were collected for die channel angle starting from 90° with the increment of 5°. We were more focused on billet, therefore only billet is kept as plastic. The die and punch were kept as rigid. The value of 2 mm/s was assigned to the ram speed. The optimum mesh element numbers were chosen as 20,000 and automatic re-meshing was used to accommodate large deformation in analyses. The value of 0.12 was selected as a friction coefficient and all analyses were performed at the ambient temperature.

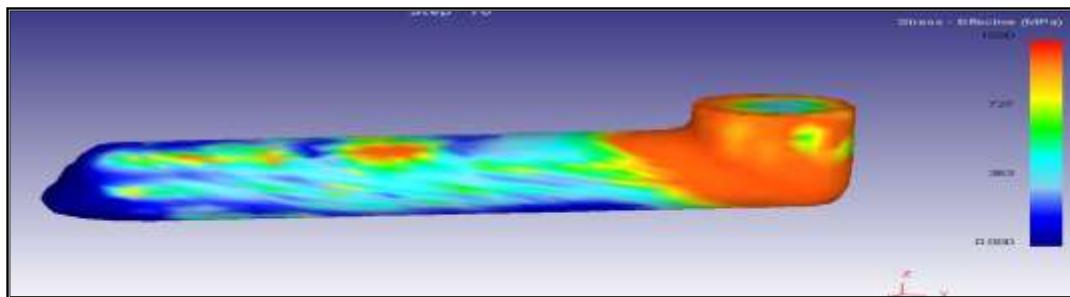
#### D. Post

Results obtained from simulation were displayed in post module. These results have been used for further calculations. Figure 7 shows deformed billet and the values of effective strain developed in 100° die channel angle.



**Fig-7 : Billet after simulation**

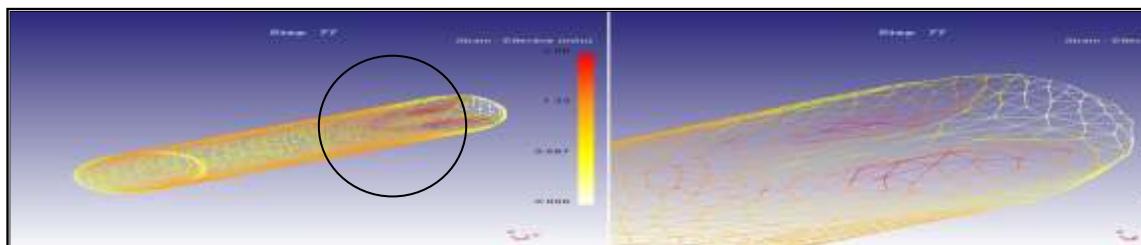
Figure 8 shows the effective stress distribution in the ECAPed billet for 100° die channel angle. It can be observed that the max stress area lies near to the intersecting line of channels and at the contact area of billet and plunger.



**Fig-8: Stress variation in billet**

One of the objectives of simulation is to find out the ramming force. We can directly plot graph of time v/s load in z direction which gives value of load at every point of displacement of plunger through die.

Figure 9 shows the maximum strain area lies at the end of billet material, and its same for all die geometries.



**Fig-9: Maximum strain area**

Though the Figure9 showing maximum strain, we consider the strain values at the areas except the end portions of the billet.

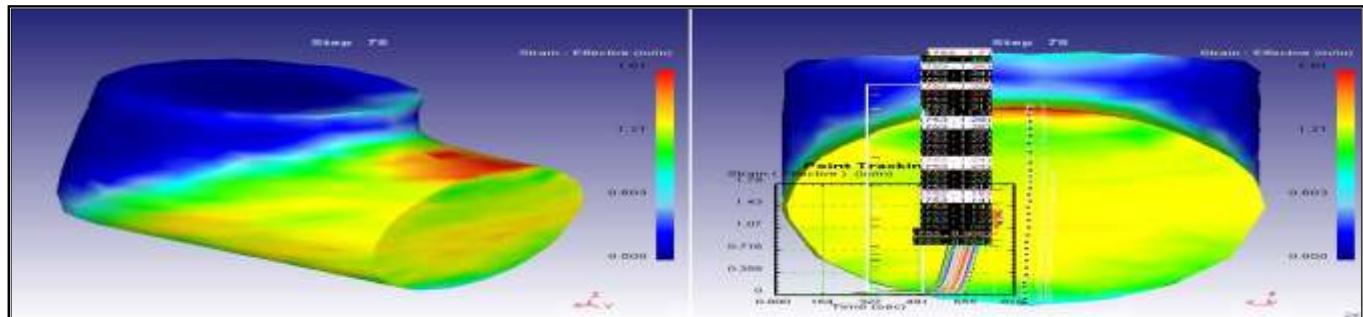
#### E. Results

$\phi$ (Die channel angle)	$\Psi$ (Die corner angle)	strain effective Max.	stress effective	
			MPa	
90°	15°	1.81	1113	
95°	15°	1.77	1100	
100°	15°	1.73	1090	
105°	15°	1.13	1020	
110°	15°	1.11	1020	

**Table- 3 Result data**

To calculate in homogeneity index,  $\epsilon_{\max}$  and  $\epsilon_{\min}$  on a single plane should be known. The values of strain at different nodes on a single plane can be taken by selecting points on that plane as shown in Figure10. To calculate  $\epsilon_{\text{avg}}$  30 points were plotted on single

section plane and the corresponding strain values were used for calculations. The graph in the Figure 10 gives the values of strain at a single plane for 30 points for  $\phi=90^\circ, 95^\circ, 100^\circ, 105^\circ, 110^\circ$  respectively.



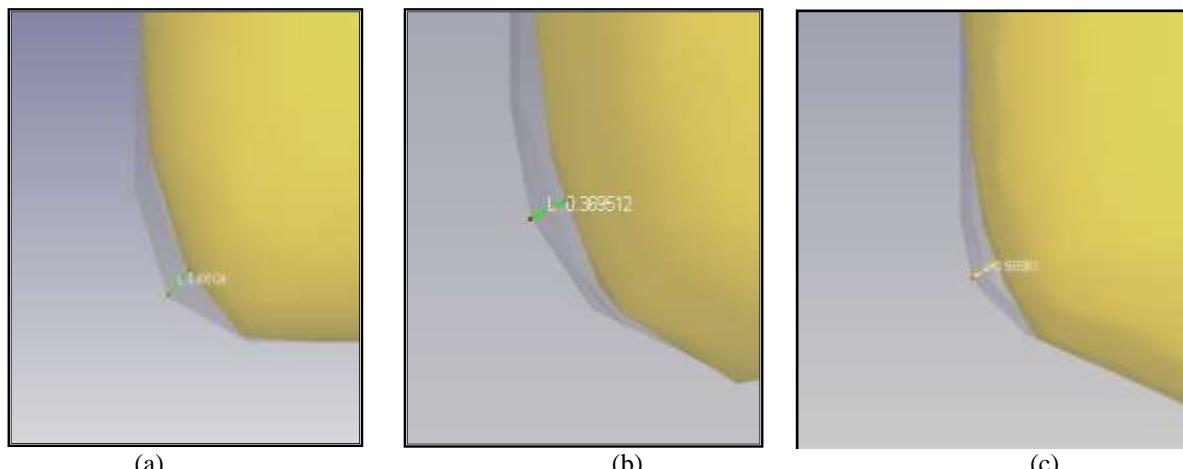
**Fig-10. Values of strain at 30 different points for  $\phi=90^\circ$**

Figure 5.14 shows different 30 points selected on a single plane inline to calculate average effective strain induced in billet passed through die channel angle of  $90^\circ$ . The in homogeneity index came out as 0.2. Colour layers shows the strain is not uniform in the cross section of the billet.

## F. Die channel angle

Deformation behaviour and strain homogeneity is essential to design an ECAP die. Complicated and smooth deformation behaviours are observed with  $\phi<110^\circ$  and  $\phi>110^\circ$ , respectively [12]. The influence of channel angle on the deformation behaviour and strain homogeneity in ECAP are obtained by conducting finite element simulations with DEFORM 3DTM for a range of channel angles. The deformation behaviour can be explained in the form of deformation steps and corner gap/dead zone formation for different channel angles.

From the simulation results the corner gap can be measured, Figure 6.4 (a), (b), (c), (d) and (e) shows the amount of corner gap formed in die channel equal to  $90^\circ, 95^\circ, 100^\circ, 105^\circ$  and  $110^\circ$  respectively. It is observed from the figure that at  $100^\circ$  die channel angle the value of corner gap is less compare to other die channel angles.



**Fig-11. Corner angle for different values of  $\phi$  (a)  $\phi=90^\circ$ , (b)  $\phi=95^\circ$  (e)  $\phi=110^\circ$**

Deformation is taking place in three stages with acute channel angles ( $\phi<110^\circ$ ), and in two stages with perpendicular and obtuse channel angles ( $\phi>110^\circ$ ). With  $\phi=50^\circ, 70^\circ, 90^\circ$  and  $100^\circ$ , the front left corner of the Sample sticks to the bottom of the die, fills the entire die, and only then the deformation starts. Strain homogeneity in the ECAP is mainly dependent on the simple shear deformation. Simple shear deformation can be obtain only when the channel volume get completely filled with billet material [4]. The corner gap results into deformation by bending more than by shear. The corner gap formed for different angles are shown in Figure 11. According to the result we obtained from the simulation  $\phi = 100^\circ$  gives good results. Maximum channel volume gets filled with billet material.

The graph plotted from the result of simulation shows, in homogeneity index calculated for  $\phi=100^\circ$  is less than in homogeneity index for other angles less than  $100^\circ$ , but for  $\phi>100^\circ$  though the Ci is less, the effective maximum strain values are

less than those for  $\phi=100^\circ$  as calculated and shown in Table 4. Considering all the parameters in mind  $\phi=100^\circ$  is chosen to get optimum results.

N (No. of passes)	$\Psi$ (Die corner angle)	$\Phi$ (Die channel angle)	$\epsilon$ (Plastic strain)
1	15	90	1.07688
1	15	95	0.99337
1	15	100	0.91515
1	15	105	0.84150
1	15	110	0.77180
1	15	115	0.70551
1	15	120	0.64218
1	15	125	0.58140

Table- 4 Theoretical value of strain for different Die angles

### 3.EQUIPMENT AND EXPERIMENTAL ANALYSIS

#### A. ECAP (Equal Channel Angular Pressing)

##### i. Preparation of billet

Billet is machined to required size, 100 mm in length, 9.5 mm diameter, and fillet of 1mm at the edges. It is then polished using abrasive paste of 400 microns. Annealing has been done on the machined billet to get homogeneous properties throughout the length of the material. Billets are held at 350°C temperature for 1hr in the electric furnace and then allowed to cool in furnace itself.

##### ii. Procedure for ECAP

UTM TUE-C-400 servo is used to perform ECAP. Experiment is performed at room temperature. Die channel is lubricated to avoid friction between surface of billet and channel. Well lubricated billet is inserted into the die channel. Mineral oil based grease is used as lubricant. Load has applied manually to the billet through plunger. Single pass is carried out and the passed billet is used for study. Actual setup is shown in Figure 7.2.



Figure 2 Test Setup

## 4. CONCLUSION AND FUTURE WORK

### Conclusion

The detail study of severe plastic deformation by ECAP has been studied.

1. It is concluded that the degree of severe plastic deformation depends upon the geometry of the die. It has been seen that the strain introduce in billet material is high at channel angle equal to 90° and reduces with the increase in channel angle.
2. Plastic deformation zone depends upon the die corner angle. For small die corner angle, plastic deformation zone accumulate near pure shear plane and spread away from pure shear plane as die corner angle increases. FEM analysis has successfully done for different die geometry.
3. With the combination of corner angle equal to 15° and die channel angle 100°, optimized result have observed in FEM analysis. ECAP die has been manufactured for die corner angle equal to 15° and die channel equal to 100°.

### Future work

1. The work can be done to eliminate the forming of burr at the billet material.
2. No. Of trials can be taken for passing the material through die, so as to study changes occurred into the material.

## 5. ACKNOWLEDGEMENT

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