Producing Ultra Fine Grain Pure Aluminum Tubes Using Tubular Channel Angular Pressing (TCAP)

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This master thesis is dedicated to my parents.

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ABSTRAK

poli-kristal halus bahan-bahan halus dan ultra nano logam berstruktur mempunyai sifat mekanikal yang lebih baik berbanding konvensional logam poli-kristal. Oleh itu, banyak percubaan dalam 20 tahun yang lalu telah dibuat untuk menghasilkan bahan-bahan ultra halus dan bernanostruktur denda oleh ramai penyelidik di seluruh dunia. Jumlah yang agak besar kaedah untuk menghasilkan bahan-bahan logam besar pernah ditawarkan, bagaimanapun, walaupun keperluan yang meluas untuk paip dengan kekuatan yang tinggi kepada badan, kerja kurang lakukan untuk menghasilkan paip halus ultra halus dan nano berstruktur.

Sehubungan dengan pembuatan paip UFG dua kaedah ASB dan HPTT telah pun disediakan. Prosedur ini mempunyai kelemahan, seperti; mikrostruktur dan inhomogeneity tekanan, memerlukan peralatan yang kompleks dan mahal, kuasa besar, lekatan antara lapisan dan keupayaan Perindustrian rendah. Oleh itu, menyediakan yang berkesan, murah, dengan keupayaan industri dan produktiviti yang tinggi diperlukan untuk paip. Baru-baru ini kaedah novel bertajuk sudut Channel tiub Menekan (TCAP) telah dibangunkan oleh Faraji pada tahun 2011 di Iran yang merupakan perunding projek semasa. Mereka telah mengemukakan satu kaedah yang dapat mengatasi kebanyakan kelemahan kaedah sebelumnya. Faraji et al telah memohon TCAP pada AZ91 aloi tetapi kaedah ini tidak digunakan pada aloi aluminium. Dalam projek ini, kaedah baru ini dengan kelebihan kos rendah, tidak mempunyai had dimensi untuk tiub, mengenakan tekanan yang teruk plastik ricih, tekanan hidrostatik yang tinggi dan keupayaan untuk menghasilkan tiub ultra-halus dan paip logam bernanostruktur dengan kekuatan tinggi digunakan untuk aluminium paip untuk kali pertama. Halus (UFG) tiub silinder dihasilkan melalui dibangunkan baru-baru tiub saluran sudut menekan (TCAP) proses melalui pas berbeza daripada Aluminium tulen. Mikrostruktur dan sifat mekanik tiub diproses melalui 1-3 pas proses TCAP telah disiasat. Penyiasatan mikrostruktur menunjukkan terutamanya mengurangkan saiz bijirin kepada kira-kira 350 nm daripada nilai utama ~ 56 µm. Microhardness tiub diproses telah meningkat kepada 49.4 Hv selepas satu hantaran dari nilai awal 32.9 Hv. Peningkatan dalam bilangan kelulusan daripada 1 ke nombor yang lebih tinggi pas tidak mempunyai kesan yang lebih kepada microhardness itu. Hasil kekuatan dan muktamad telah meningkat 2.5 dan 2.28 kali berbanding sebagai membuang keadaan. Terutamanya meningkatkan kekuatan telah dicapai selepas satu pas TCAP manakala bilangan yang lebih tinggi pas mempunyai kesan tidak lebih.

ABSTRACT

Poly-crystalline ultra fine grained and nano structured metallic materials have superior mechanical properties compared to conventional poly-crystalline metals. Therefore, many attempts in the past 20 years have been made to produce ultra fine grained and nano structured materials by many researchers around the world. Relatively large number of methods for producing bulk metallic materials ever offered, however, despite the widespread need for tubes with high strength to weight, less work is doing to produce ultra fine grained and nano structured tubes.

In connection with the manufacturing of UFG tubes two methods of ASB and HPTT have already been provided. These procedures have disadvantages, such as; microstructure and strain inhomogeneity, requires complex and expensive equipment, large forces, adhesion between the layers and low Industrial capabilities. Therefore, providing an effective, inexpensive, with industrial capability and high productivity is required for tubes. Recently a novel method entitled Tubular Channel Angular Pressing (TCAP) has been developed by Faraji in 2011 in Iran who is consultant of current project. They have presented a method that is able to overcome most of the disadvantages of previous methods. Faraji et al have applied TCAP on AZ91 alloy but this method has not been applied on aluminum alloys. In this project this new method with the advantages of low cost, having no dimensional limitation for the tube, imposing severe plastic shear strain, high hydrostatic pressure and the ability to produce ultra-fine tubes and nanostructured metal tubes with high strength are applied to aluminum tubes for the first time. Ultrafine grained (UFG) cylindrical tubes were produced via recently developed tubular channel angular pressing (TCAP) through different passes from pure Aluminum. The microstructure and mechanical properties of processed tube through one to three passes of TCAP were investigated. Microstructural investigation shows notably decrease in the grain size to around 350 nm from the primary value of ~56 µm. Microhardness of the processed tube was increased to 49.4 Hv after one pass from an initial value of 32.9 Hv. An increase in the number of passes from 1 to higher number of passes has not more effect on the microhardness. Yield and ultimate strengths were increased 2.5 and 2.28 times compared to as cast condition. Notably increase in the strength was achieved after one pass TCAP while higher number of passes has not more effect.

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CHAPTER ONE

INTRODUCTION

1.1 Introduction

From the viewpoint of crystalline, metallic materials can be divided into mono crystalline and polycrystalline materials.

We can determine physical and mechanical properties of all crystalline materials with several items. Usually, the materials average grain size has the notable and often preponderant role. Thus, the strength of all polycrystalline materials is related to the grain size, d. Eq. (1), Hall-Petch , states that the yield stress σy , is achieved as below:

$$\sigma_{yield} = \sigma_o + k_y d^{-1/2} \qquad (1\text{-}1)$$

Where σ_0 is symbol of the friction stress and k_y is refer to the constant of yielding. Based on Eq. (1-1), by decreasing the size of grains, the strength will increase. In turn, this has led to a rise in the interest of constructing materials with extremely tiny grain sizes.

Eq. (1) denotes that the yield stress magnifies with reduction of the square root of the grain size. In other words via increasing one, the other one will decrease. A higher tensile strength is achieved with the decrease of grain size [1].

Firstly, is essential to present a precise definition of ultrafine-grained materials (UFG).

1.2 Ultra-Fine Grained (UFG) materials

1.2.1 Definition of (UFG) materials

BY referral to the specifications of polycrystalline materials, UFG materials are described as:

polycrystals in which the grain sizes of them are average, less than 1 micrometer. For bulk shape materials that their microstructure is UFG, there are the further specifications of reasonably equiaxed and moderately homogeneous microstructures and with a majority of grain boundaries having high angles of misorientation. The existence of a high angle grain boundaries is influential because of produce advanced and novel attributes [2, 3].

Table1.1 definition of nano structured, Ultra fine grain and coarse grain materials

0-100 nm	100nm-1 µm	1mm-1 µm
Nano structured	Ultrafine grain	Coarse grain

Table 1.1 shows a brief definition of nano structured, Ultra fine grain and coarse grain materials.

Ultra-fine grained and nano structured materials have a strength of about 2-4 times higher than conventional ones. So they can withstand much higher pressures than conventional materials.

By using this method, it`s the possible to produce high strength tubes which they have the ability to be used in different industries such as automotives, aircrafts, medical and so on. Using high strength/weight materials cause to decrease the weight of structures and reduce consumption of energy.

1.2.2 Advantages of Ultra-fine grained materials

Ultra-fine grained materials have a higher strength rather than materials that made with traditional shaping methods.

Hence it gains the attention of many researchers, industrial and research centers in various countries [4].

1.3 Grain Size

Metals are crystalline in nature, except on few occasions, and apart from single crystals, they are composed of internal boundaries recognized as grain boundaries.

When a new grain is nucleated during processing, the atoms within each growing grain are lined up in a specific pattern which depends on the crystal structure of the alloy or metal. With growth, each grain will in the long run contravene on others and form an interface where the atomic orientations are different.

From year 1900, this fact is clear that most of mechanical properties of materials were improved while the size of the grains decreased. In order to achieve the desired grain size, alloy composition and processing procedure, must be controlled.

1.3.1 Grain Boundary Formation

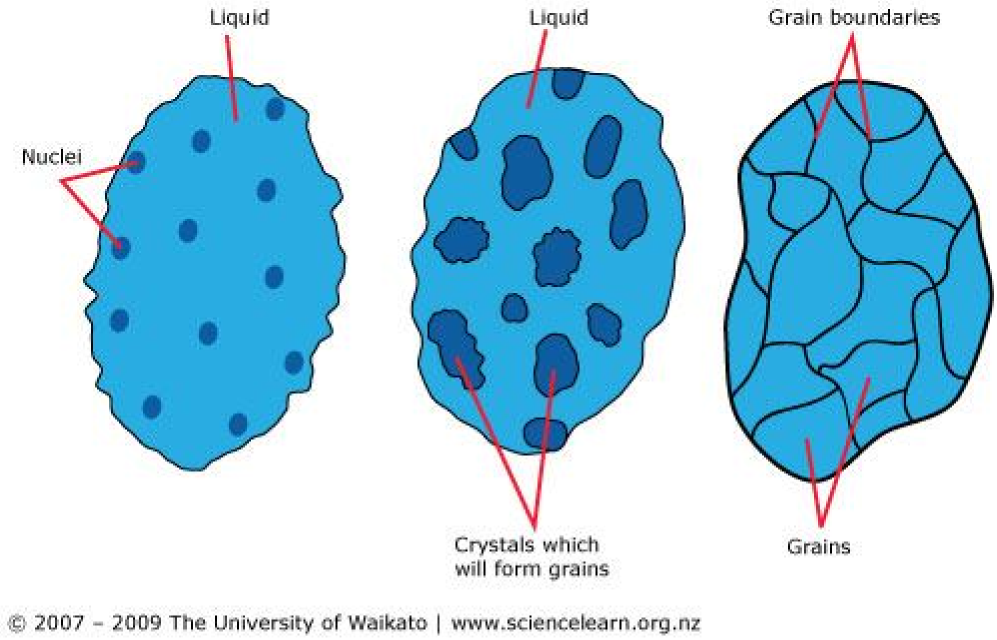


Figure 1.1 Grain Boundary Formation

Figure (1.1) is a good example of grain boundary formation. As you can see when the liquid metal is cooled, the metal atoms shaped into a crystal lattice. Considering sufficient time and the ideal conditions, the crystal lattice can grow up to be very great, with a full-scale inner crystal structure.

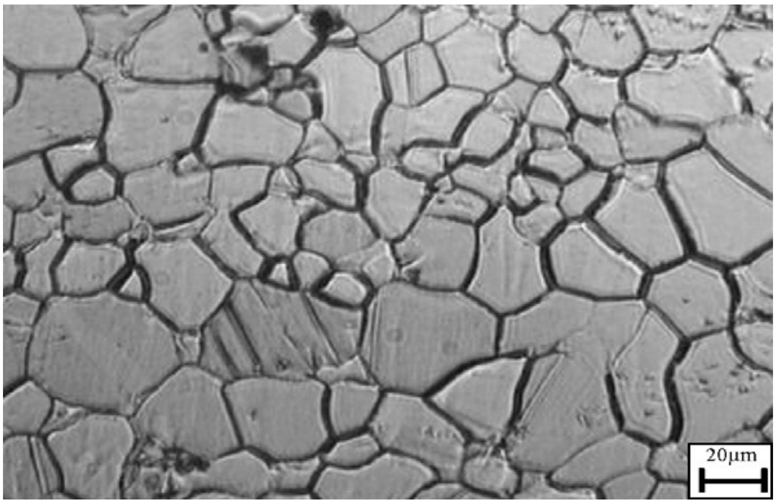


Figure 1.2 The grain size of magnesium metal alloy

Figure (1.2) shows the grain size of magnesium alloy.

Serrano et al [5], have shown great examples of units for measuring the length as you can see in Fig.(1.3)

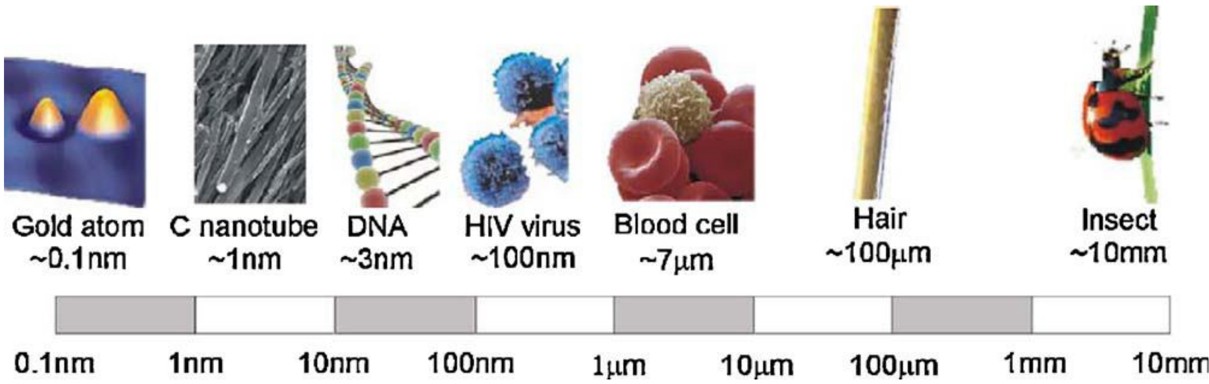


Figure 1.3 examples of length scale.

1.3.2 Role of Grain Boundaries

Grain boundaries have very important role in plastic deformation of materials that they have polycrystalline structure.

We outline below important aspects of grain boundaries.

1. At low temperature ($T<0.5T_m$, where T_m is the melting point in K), grain

boundaries operate as strong obstacles into dislocation motion. Mobile dislocations can pile up against the grain boundaries and thus give rise to stress concentrations that can be relaxed by initiating locally multiple slip.

2. Within the deformation of polycrystals; there exists a harmony condition among the neighboring grains, that is, if the expansion of voids or cracks is not permitted, the deformation in each grain must be accommodated by its neighbors.

Figure (1.4) shows typical equiaxed grain configurations for polycrystalline titanium.

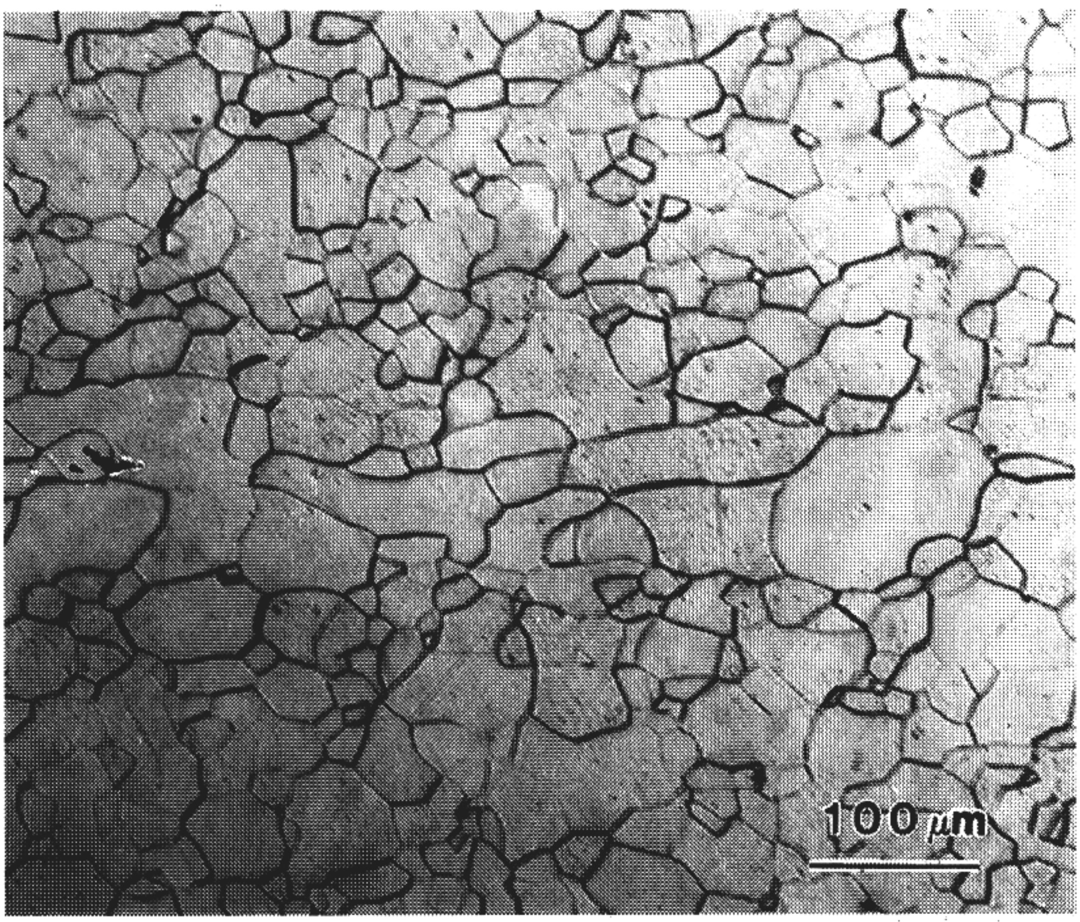


Figure 1.4 Micrographs showing polycrystalline Titanium

One example of a material property that is dependent on grain size is the strength of a material; as grain size is increased the material becomes weaker . strength is expressed in units of stress (MN/m^2).Grain size of a material can be increased by annealing. Hardness measurement can provide a measure of material`s strength.

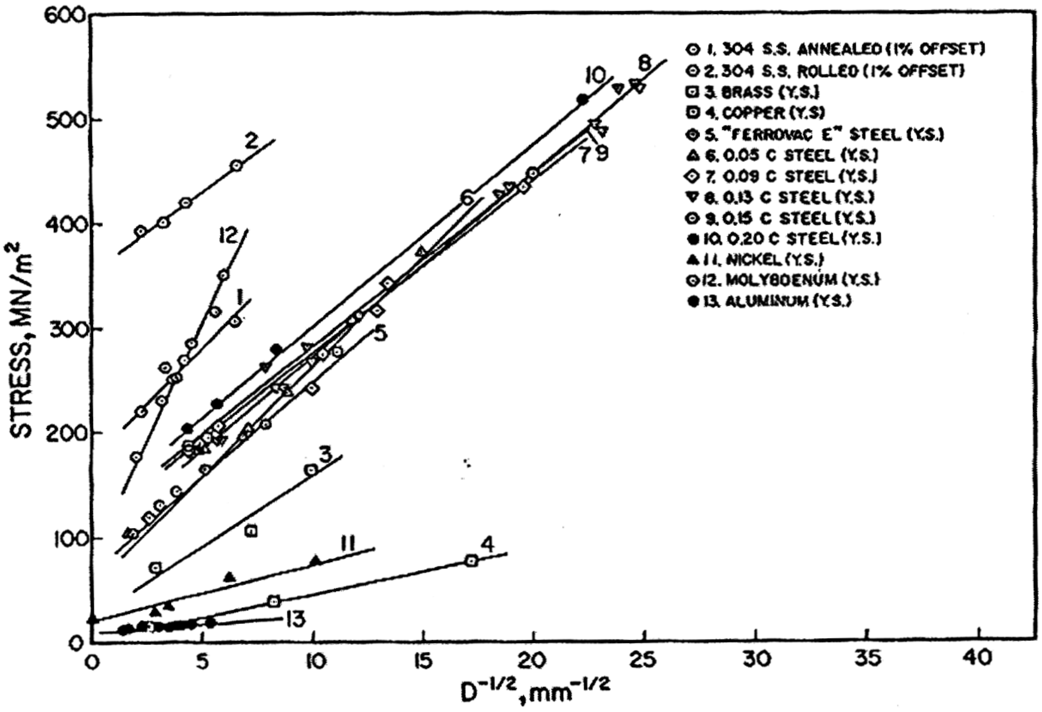


Figure1.5 strength dependence of grain size for a number of metals and alloys.

Fig (1.5) shows the strength dependence of grain size for a number of metals and alloys.

1.4 Producing Ultra-Fine Grain Materials

Nanocrystalline materials can be synthesized either by consolidating small clusters or breaking down the polycrystalline bulk material into crystalline units with dimensions of nanometers. These approaches have been classified into bottom-up and top-down .in the other word, two fundamental and supplementary tactics have build up for the combination of UFG materials .They are describe as ‘‘bottom-up’’ and the ‘‘top-down’’ [6].

1.4.1 ‘‘Bottom-Up’’ approaches

At ‘‘bottom-up’’ approach, we have to arrange the nanostructure atom-by-atom, layer-by-layer. Some instance of these tactics are as mentioned in following; inert gas condensation, electrodeposition [7], ball milling with subsequent consolidation [8],and cryomilling with hot isostatic pressing [9, 10],where cryomilling is selfsame mechanical milling in liquid nitrogen (at a temperature of - 180 ° C) .Actually, these methods are mostly restricted to manufacture of very small samples that they have applications in some fields like microelectronics components, and generally they are not suitable for big-scale structural usages.

Besides, the products made from these methods have some residual porosity and contamination that is presented within manufacturing procedure[11].

1.4.2 ‘‘Top-Down’’ approach

In ‘‘top-down’’ approach we start with bulk material by a comparatively coarse grain size and break down the microstructure into a nanostructure. This method does not have dimensional restriction for the sample, in addition the contamination and residual porosity which are inseparable part of the ‘‘bottom-up’’ approach, do not exist in them. Another advantage is that, these methods can be used for a wide range of alloys and metals.

Preliminary studies on the production of materials with ultra-fine grain microstructure were done by Russian scientists in 1990 with the publication of several articles on pure metals[12].

First publications has been produced about describing the methods of applying severe plastic strain in production of the bulk material with the grain size of less than a micron with a high-angle grain boundaries.

It should be noted these initial propagation give a direct representation of the ability to apply heavy plastic straining in the manufacturing of bulk materials which have justly homogeneous and equiaxed microstructures with grain sizes in the range of submicrometer with a high-angle grain boundaries[2, 3].

In order to convert a coarse-grained metals into a material with ultrafine grains, it is essential to inflict an extraordinary high strain in order to present a high density of dislocations and then these dislocations setup to form new grain boundaries[3].Actually, traditional deformation methods like extrusion or rolling has some limitations to production of UFG structures due to two significant reasons. First, at imposing strain by traditional methods, the cross-section of the sample will change.

Second, the strains imposed in traditional method for producing UFG structures because of the generally low workability of metallic alloys at ambient temperatures are insufficient.

Because of these limitations, efforts to develop new methods based on the application of severe plastic deformation that large strain at low temperatures without any change in initial cross-section of samples has been earmarked.

1.5 Severe Plastic Deformation

Processes of severe plastic deformation (SPD) are defined as metal forming processes in which a very large plastic strain is imposed on a bulk process in order to make an ultra-fine grained metal. The objective of the SPD processes for creating ultra-fine grained metal is to produce lightweight parts by using high strength metal for the safety and reliability of micro-parts and for environmental harmony [1].

In short, forms of severe plastic deformation methods for producing ultra-fine and nano-structured materials must have the following properties:

1 - The ability to manufacture microstructures with high angle grain boundaries.

2 - Ability to produce homogeneous micro-architecture parameters to produce sustained properties in the entire sample.

3 – Produced sample be free of mechanical defects or cracks.

Traditional methods can not satisfy the above conditions and therefore, other methods must be developed under severe plastic deformation methods.Perhaps the traditional methods such as extrusion, rolling and stretching are useful for increasing strength, but it significantly decreases smoothness. Severe plastic deformation methods allow simultaneously increase in strength and softness.

1.6 Objectives

The overall purpose of this research was;

- To produce ultra fine grained pure aluminum tubes.
- To investigate mechanical properties of produced tubes such as yield strength, ultimate strength and micro hardness.
- To investigate effects of number of passes on the microstructure and mechanical properties of product at room temperature.

CHAPTER TWO

REVIEW OF RELATED LITERATURE

2.1 Methods of severe plastic deformation

Ultra-fine grained and nano-structured materials have a higher strength rather than materials that made with traditional shaping methods. Hence it gains the attention of many researchers, industrial and research centers in various countries by using ultra-fine materials, it is possible to build cars, airplanes and machines with lighter weight, and therefore there is less energy consumption and contamination.

Methods of severe plastic deformation should meet a number of requirements which are to be taken into account while developing them for formation of nanostructures in bulk samples and billets.

Production of ultra-fine microstructures or nanostructures is possible with severe plastic deformation methods. an obtained grain size and specification of a nanostructure forming is depend on many parameters such as the type of process, process parameters, The total cumulative strain, form and substance of the elements, initial microstructure of a material, phase composition temperature, pressure, etc. Depending on these factors, it is possible to obtain grain sizes below 100 nm. application of ECAP pressing can provide mean grain sizes around 200-400nm[2].

In order to transform course grain materials into ultra fine grain materials, it`s necessary to applied very large plastic strain on them. In recent years, various methods of severe plastic deformation are presented and have been developed.

Over the last years many works have been done to improve material specification by grain purgation, with using of severe plastic deformation techniques. These methods includes process of equal channel angular pressing(ECAP)[13], high pressure torsion (HPT)[14], accumulative roll bonding (ARB)[15], Repetitive Corrugation and Straightening(RCS) [16],Dissimilar-channel angular pressing(DCAP)[17], accumulative back extrusion (ABE) [4],cyclic extrusion compression method(CEC)[18], friction stir processing (FSP)[19],twist extrusion(TE), multi-directional forging[20, 21], constrained groove pressing (CGP)[22],cylinder covered compression (CCC)[23], and submerged friction stir processing(SFSP)[24] .All of these procedures are capable of introducing large plastic straining and significant microstructural refinement in bulk crystalline solids.

The process of equal-channel angular pressing (ECAP), known also as equal-channel angular extrusion (ECAE), was first introduced by Segal and his co-workers in the 1977 in order to produce ultra-fine material[12].

This process is commonly applied on bulk forming metal. It is possible to used for consolidation of metallic powder[12]. High pressure torsion process was first introduced by Bridgman in 1935. At that time there was no significant attention in microstructure change. Saito and his co-workers developed ARB process for the first time[15]. This method is based on the traditional rolling. It's suitable for the production of ultra-fine and nano- structure sheets.

Huang and his co-workers offered other method in 2001 under the name of repetitive corrugation and straightening[16] ,in purpose of production sheets and plates. In that study, the mentioned method was tested on a copper plate and microstructure of the samples was studied using electron microscopy. It has been reported that Copper nanostructured are visible with low and high angle grain boundaries.

Lee and his co-workers provided the method of Dissimilar-channel angular pressing based on ECAP process in year2002 as a proper method for mass production of nano-structured sheet metal [25]. They used combination of ECAP process and rolling to produce mass production of sheet. Also the feasibility of this method was evaluated by using experimental tests and finite element methods (FEM).

Korbel and Richert have presented cyclic extrusion-compression process(CEC) based on the extrusion process. They have checked formation of shear bands in the microstructure of aluminum ,during the process at high strain amplitude .They showed that the strain hardening and microstructure of sample at strains on the order of 6.0 enters the saturation stage[26].Faraji and his co-workers[4], imposed accumulative back extrusion (ABE) process that was presented by Fatemi-varzaneh and Zarei in 2009 on AZ91 magnesium alloy and studied the microstructure and Micro-hardness of it. They had shown that the microstructure of the samples in different area was different and there is relatively high inhomogeneity.

Akbar Mousavi and his co-workers [27],have studied another process under the name of twist extrusion with using numerical and experimental tests.

They have investigated the influence of this process on strains distribution and microstructure of pure aluminum and showed that applying direct extrusion with a 50% reduction in cross sectional area, after twist extrusion process can Improve strain homogeneity and consequently homogeneity in mechanical properties of sample.

Process of Constrained groove pressing are provided by Shin and colleagues in year 2002 [22],as a SPD method for production of ultra-fine grain steel sheets and plates. They have reported grain size of less than one micron in the aluminum sheet after applying this process.

Among the above mentioned approaches, process of Equal Channel Angular Pressing (ECAP), High Pressure Torsion (HPT) and Accumulative Roll Bonding (ARB) are more interested since they provide large plastic deformations and cause formation of ultrafine and nano grained structures. In continue brief introductions of these important techniques are discussed.

2.1.1 Equal Channel Angular Pressing (ECAP)

The schematic representation of the ECAP process is shown in Fig (2-1)

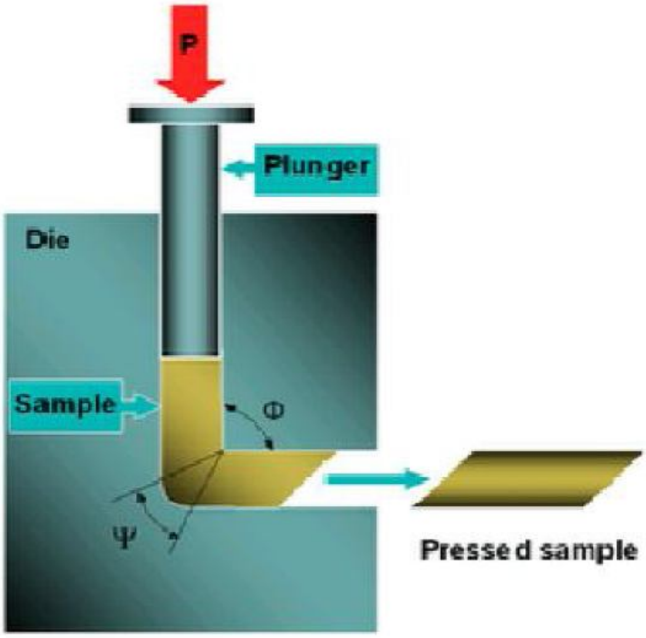


Figure 2.1 Equal-Channel Angular Pressing (ECAP)

A sample is pressed through a die contained within a channel bent through an angle close to 90°.[28]

The specimen is side extruded through the shear deformation zone with the dead zone in the outer corner of the channel. When the work piece is side extruded through the channel, the total strain is given by Eq.(2-1)[29]:

$$\varepsilon = \frac{1}{\sqrt{3}}\left\{2\cot\left(\frac{\phi}{2}+\frac{\varphi}{2}\right)+\varphi \operatorname{cossec}\left(\frac{\phi}{2}+\frac{\varphi}{2}\right)\right\} \qquad (2\text{-}1)$$

Where φ is the angle of intersection of the two channels, and φ is the angle subtended by the arc of curvature at the point of intersection. When φ = 90˚ and φ = 0˚, the total strain from the above equation is ε = 1.15. After *n* passes, the total strain becomes $n \times \varepsilon$.

Among the various methods of SPD equal-channel angular pressing is an especially attractive processing technique for several reasons:

First, this method can be used for producing parts with large dimensions such that there is the potential for producing materials that may be used in a wide range of structural applications.

Second, the method is using relatively simple equipment that it can be found in almost all laboratories.

Third, this method can be used for a variety of materials with different crystalline structures, Single-phase and multi-phase alloys and also metal-based composites.

Fourth, Parts made with this method have a relatively good homogeneity.

Nature of the ECAP process is that the Intensive shear strain is applied in the corner and where the two channels intersect. It can be seen that despite the introduction of very large strains on the Substance, the cross section area remains constant after passing the channel, and it is the most important feature of severe deformation processes. After one time the sample was pressed in ECAP die it may needed to re-press it, to achieve higher strain.

Four different processing routes have been introduced in ECAP. Including: route A, where the sample is pressed repetitively without any rotation. Route BA, where the sample is rotated by 90˚ in alternate directions between consecutive passes. route BC, where the sample is rotated in the same sense by 90˚ between each pass and route C, where the sample is rotated by 180˚between passes[29].

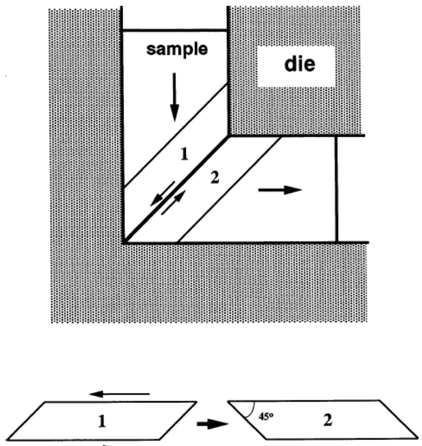


Figure 2.2 The principle of ECAP showing the shearing plane within the die: the elements numbered 1and 2 are transposed by shear as indicated in the lower part of the illustration[29]

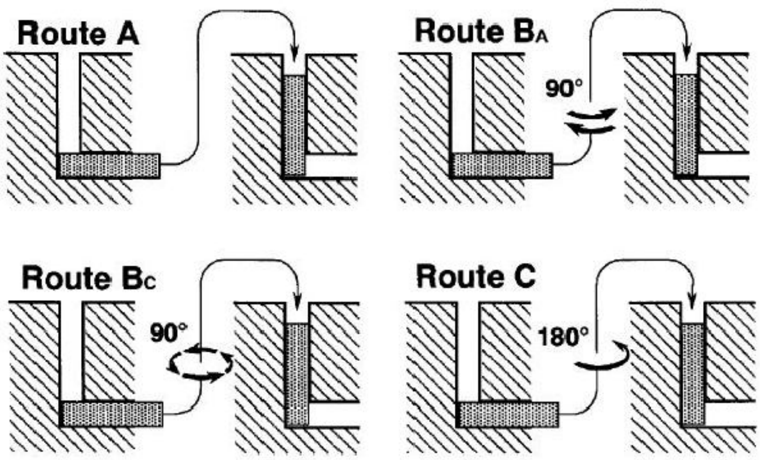


Figure 2.3 The four fundamental processing routes in ECAP [29]

There is an important limitation in arbitrary ECAP, when sample is under the multi-pass ECAP process, between the passes, the sample should be removed from the die and reinserted into the die after rotating .The problem is more time is needed.

To facilitate the process, especially when more passes are required, several plans, such as special multi-pass dies[30], rotary dies[31], that it does not need to be removed from the die and replacing and also using of side-extrusion process[32], are provided. ECAP process usually used for Circular and quadrilateral samples ,nevertheless there is reports on the application of this method on plate samples [33],that have been published. Efforts have been made to develop continuous processes that include ECAP–Conform [34] and ECAR [35]methods .

2.1.2 High-Pressure Torsion process (HPT)

In this method according to the figure a very thin disk is compressed in a closed die by a very high pressure .torque is transmitted via friction from mandrel to the disk. tensional strain imposed can be compute by Eq.(2-2)[36].

$$\gamma(r) = \frac{2\pi nr}{l} \qquad (2\text{-}2)$$

Where r is the radius of the disk, l is the disk thickness, and n, is the number of revolutions.

Effective strain according to von Mises yield criteria can be compute by Eq. (2-3)

$$\varepsilon(r) = \frac{\gamma(r)}{\sqrt{3}} \qquad (2\text{-}3)$$

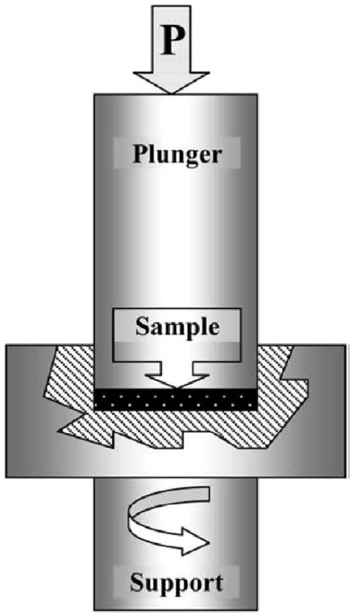


Figure 2.4 Schematic illustration of HPT processing[36]

Disadvantage of this method is that, it utilizes specimens in the form of relatively small discs and is not available for the production of large bulk materials. Another disadvantage of this method is that the microstructure produced depend on the applied pressure and the location within the disc[36].

2.1.3 Accumulative Roll-Bonding (ARB) Process

The process is simple. The roll surface is carefully cleaned and a strip of the metal is rolled to 50 % reduction, usually without a lubricant.

After rolling, it is cut into two parts, cleaned very carefully and stacked, one on top of the other part, resulting in a strip whose dimensions are practically identical to the starting work piece. The strain after n cycles of the ARB process can be expressed as,

$$\varepsilon = \frac{\sqrt{3}}{2}\ln(r),\ r = 1 - \frac{t}{t_0} = 1 - \frac{1}{2^n} \qquad (2\text{-}4)$$

Where t_0 is the initial thickness of the stacked sheets, t, the thickness after roll-bonding and r, the reduction in thickness per cycle.

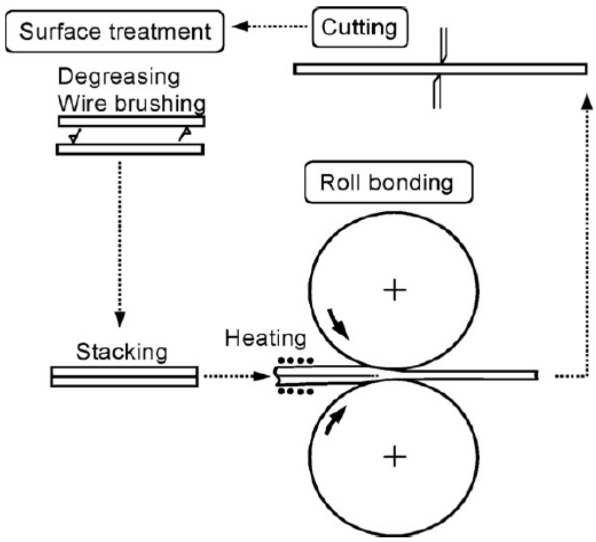


Figure 2.5 Diagrammatic representation of the accumulative roll-bonding (ARB)[36]

2.2 Methods of producing ultra fine-grained and nanostructured tubes

There are a lot of inventions and different methods for making bulk nano structured materials, but so far in the history of industry, a few works has been done for producing a nano structured tubes.

2.2.1 High Pressure Tube Twisting (HPTT)

The first method of Severe plastic deformation which is suitable for production of ultra-fine and nano- structure tubes was introduced in 2009 by Toth and colleagues[37]. Process name is High pressure tube twisting (HPTT).

The basic of process is shown in Fig 2.6. In this process, the metal tube is placed inside a rigid cylinder. A mandrel is placed into the tube, which is compressed with a compression machine in its elastic regime.

Tube Length is slightly greater than the length of the rigid cylinder and with two discs which are connected together with screwed is compressed in plastic zone. This compression is happen before compression of mandrel. Due to this compression, there is a material flow from the tube all around the upper and lower ends of the tube.

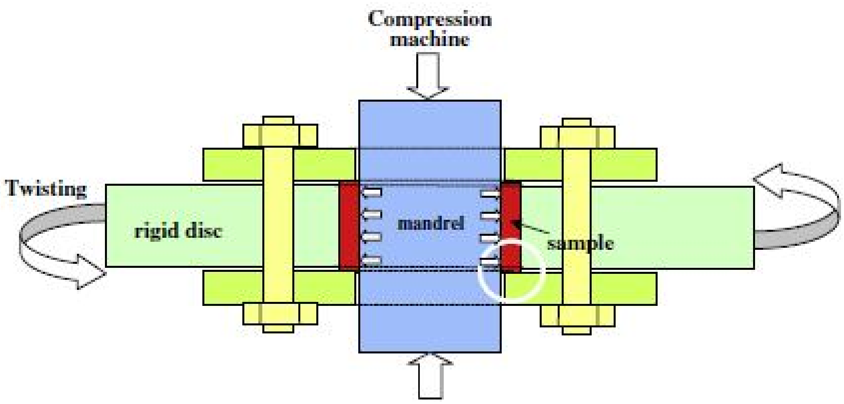


Figure 2.6 The schematic representation of the HPTT process[37]

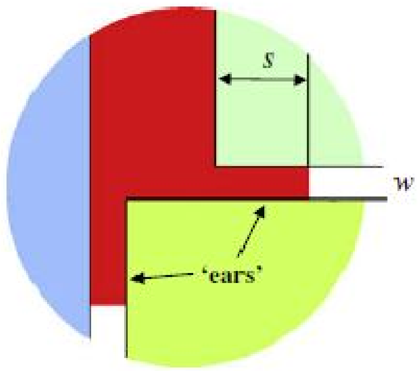


Figure 2.7 -material outflow from the sample

Disadvantages of this method are listed as below:

-Require complex and expensive equipment,

-non-uniform microstructure along the thickness of the tube,

-being time consuming process,

-Low life of equipment, due to existing very high strain,

-Build the pipe with greater length, diameter and thickness is very difficult -and perhaps impossible.

2.2.2 Accumulative Spin-Bonding (ASB)

The next process is Accumulative spin-bonding (ASB) process. The process is presented by Mohebbi ,Akbarzadeh in 2010 from Sharif University of Technology [38]. This method is based on ARB process that was presented for production of ultra fine sheets.

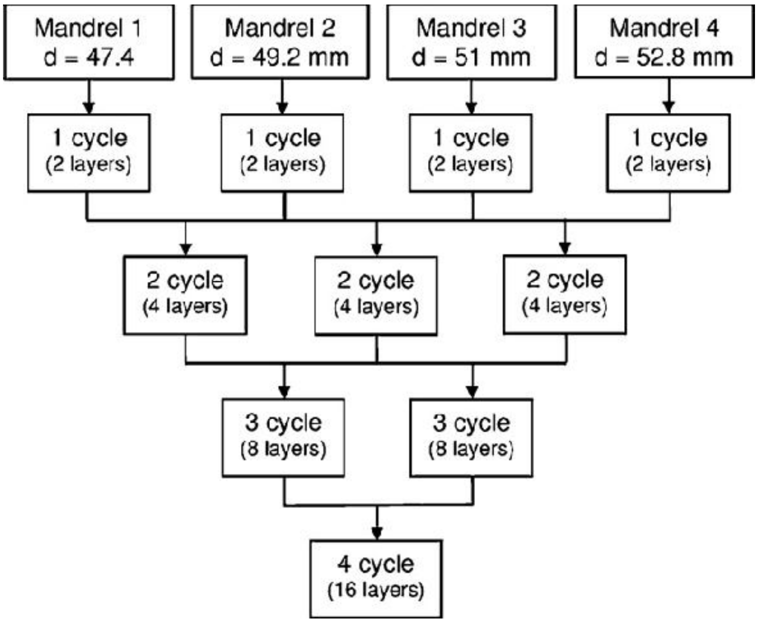


Figure 2.8 Design of four cycles of ASB utilizing four mandrels with different diameters[38]

Table 2.1 A brief presentation on the history of plastic deformation processes in the research background

Method explanation	Sever plastic deformation method	Reference number
This method is very suitable method for bulk materials	ECAP	[3]
This method is based on ECAP and rolling, suitable for the production of nanostructured plates and sheets	DCAP	[25]
This method is based on rolling, special for production of nanostructured sheets	ARB	[15]
This method has availability of applying high Hydrostatic pressure and shear strain simultaneously. It`s for small disk shape parts.	HPT	[36]

Method for production of nanostructured plates and sheets but since Hydrostatic pressure is low; There is high probability of defects and cracks.	CGP	[22, 26]
It`s similar to CGP method, But it is a continuous process for mass production.	RCS	[16]
Although based on back extrusion but resulting microstructure is usually non homogeneous. The homogeneity is improved by increasing the number of passes.	ABE	[39]
The process is based on friction and confusion, since usually the temperature is high, usually it does not have the capability to produce nanostructured metals. And often is used to modify the surface structure.	FSP	[19]
It does the shear strain, with turning the cross sections of sample.	TE	[27]
Suitable method for bulk materials with Circular cross section .At the parts with square and rectangular cross sections strain homogeneous is not good.	CEC	[26]
This method is used for producing tubes, based on HPT. Disadvantages: complicated equipment, non-uniform microstructure, high costs, caused severe flash. Low life of equipment.	HPTT	[37]

This method is used for producing tubes, based on ARB. Disadvantages: Adhesion layer, a very time consuming process, requiring expensive equipment.	ASB	[38]

Table (2.1) shows a brief presentation on the history of plastic deformation processes by other researchers over the past 15 years. As can be seen most methods is for producing ultra fine grain and nanostructure bulk material with circular and square cross sections shapes as well as sheets and plates.

Both methods (ASB, HPTT) have been proposed for the productions of nanostructured tubes have disadvantages as we mentioned them earlier. Therefore, providing an effective, inexpensive, with industrial capability and high productivity is required for pipes.

Recently a novel method entitled TCAP has been developed by Faraji in 2011 [40],in Iran who is consultant of current project. They have presented a method that is able to overcome most of the disadvantages of previous methods. Such as microstructure and strain inhomogeneity, requires complex and expensive equipment, large forces, adhesion between the layers, and so far, have been largely eliminated. Faraji et al have applied TCAP on AZ91 alloy but this method has not been applied on aluminum alloys.

Aluminum and it`s alloys has many applications in various industries, specially at the medical, automotive, defense and so on in the world, therefore I choose it to work on ,in this thesis.

CHAPTER THREE

METHODOLOGY

3.1 Introduction

The main processes that we will discuss about it, in this thesis, is Tubular channel angular pressing (TCAP) to produce ultra-fine grain and nano-structure tubes.

Further details of the research methods used in this thesis are presented in this chapter. Empirical tests were performed to investigate metallurgical and mechanical properties of produced tubes.

3.2 Principles of TCAP

Fig.3.1 (a) shows the principle of TCAP process. The tube material is pressed by a hollow cylindrical punch into a gap between die and mandrel which form a tubular angular channel with three shear zones I, II and III. The cross-section of the T CAP processed tube remains unchanged at the end of process. In multi pass TCAP, the process is repeated as many times as necessary to achieve distinct strains without any reduction in the cross section of work piece.

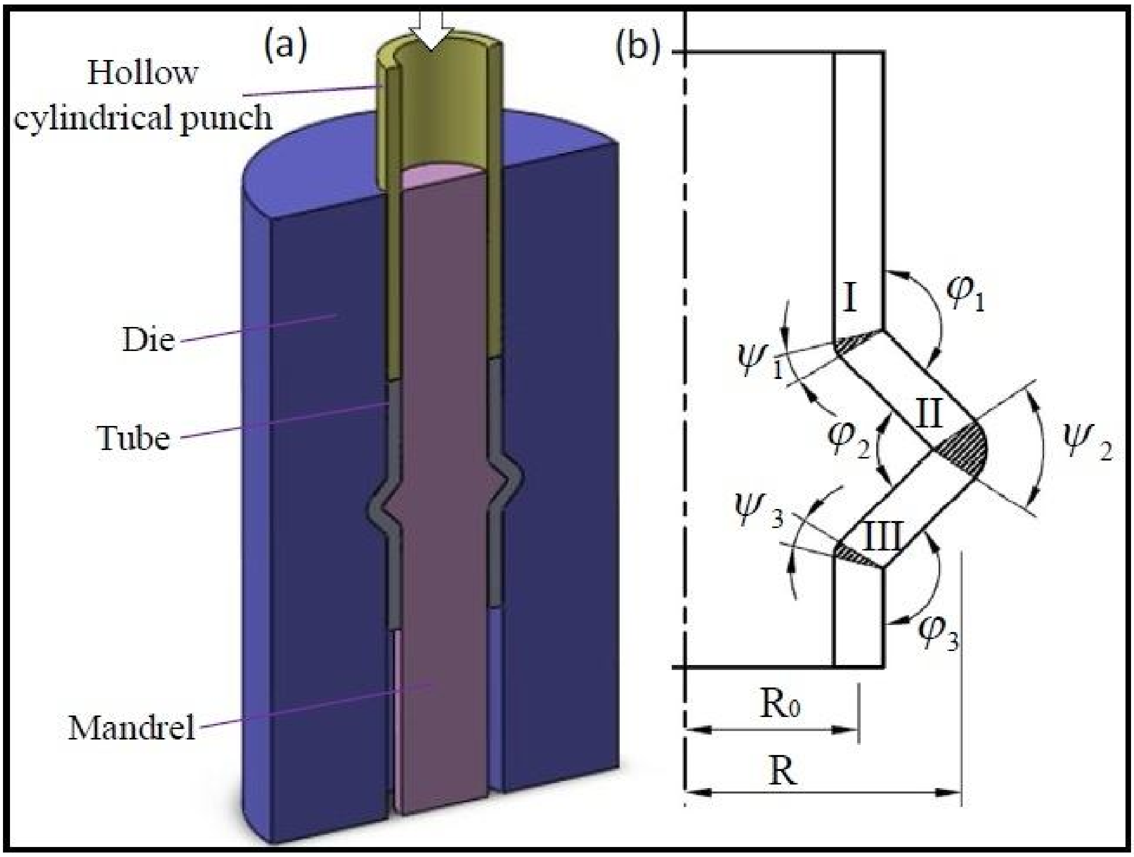


Figure 3.1(a) A schematic of TCAP (b) processing parameters

As mentioned in Ref. [41], the strain–stress state during TCAP is different from that of the ECAP. In the ECAP process, the strain state could be considered as simple shear, while in TCAP there are some additional radial and circumferential tensile and compressive strains. The exact value of total accumulated strain (ε_T) after N passes of TCAP processing can be calculated by the following relationship, which results from common engineering plasticity formula and the geometry of fig3.1(b)[40].

$$\bar{\varepsilon}_{TN} = N\left\{\sum_{i=1}^{3}\left[\frac{2\cot(\varphi_i/2+\psi_i/2)\ +\ \psi_i\ \operatorname{cosec}(\varphi_i/2+\psi_i/2)}{\sqrt{3}}\right]+\frac{4}{\sqrt{3}}\ln\frac{R}{R_0}\right\}\varepsilon=\frac{\sqrt{3}}{2} \tag{3-1}$$

From Eq.(2-4), the total equivalent plastic strain in TCAP with the parameters used in this work after one, two and three passes are ~2.2, 4.4and 6.6 respectively.

3.3 Experimental procedures

3.3.1 Initial sample

The material used in this study was commercially pure Al .Table (3.1) shows the chemical composition of the Al used in this study.

Table 3.1Chemical composition of the Al samples

element	Wt%
Al	96.33
Si	0.308
Fe	1.54
Cu	0.81
Mn	0.138
Mg	0.130
Cr	0.022
Zn	0.72
Ti	0.002

For the preparation of test samples, initial aluminum ingots were prepared; the ingot was cut into cube blocks. The blocks were exposing to turning (cutting and face-cutting) by lathe machine to achieve the final dimensions as below:

Cylindrical tubes of 20 mm in outer diameter, 2.5 mm in thickness, and 35 mm in length. In order to obtain a fully re-crystallized homogeneous microstructure and to remove the internal residual strain, all samples were annealed for 2 hour at 350 °C, prior to the processing.

Fig 3.2(a) and (b) shows AL samples before and after testing is done.

Figure3.2 (a): the schematic of initial tube.

Figure3.2 (b): the schematic final tube.

During the process, the tube diameter increases and returns to the initial size at the end of TCAP process. As shown in this figure, the cross section area of the tube before and after TCAP process remains constant though the tail part has some inconsistency.

3.3.2 Used Equipment

To perform the test, a tensile testing machine, 30-ton Model 4208 (Figure 3.3) was used. It belongs to the experimental stress analysis Laboratory of Mechanical Engineering Department of Tehran University. The device counts the quantity of force versus displacement such that the maximum force is the same required tonnage to perform each test.

Figure 3.3 schematic of instron a tensile testing machine, Model 4208

Since the process is carried out within high-pressure, probability of a meshed tube and mandrel inside the die is too much. For the purpose of reducing the force and probability of jamming tube and mandrel inside the die, a high temperature lubricant was used. The lubricant is in powder form and is sprayed on the surface. This lubricant maintains its lubricating properties at the temperature range 20℃ - to 400℃ (Figure 3.4).

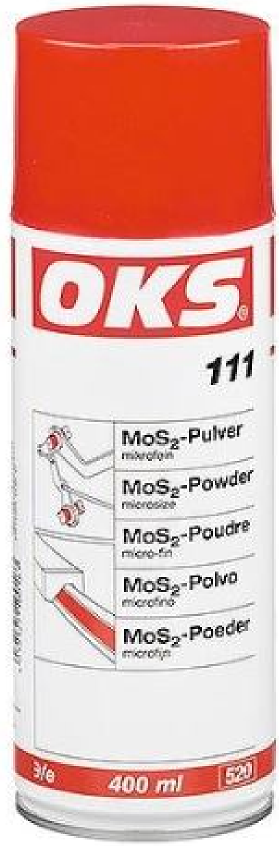


Figure 3.4 lubricants used in the experiments

3.4 TCAP die

3.4.1 Material and performance of die

The main advantage of this scheme compared to the previous dies [42] is, having two separate pieces or split-off. Exist of this section, cause prevention of meshed tube and mandrel into the outer wall.

The only problematic possibility with the die was meshing of the tube to mandrel. This was elevated with increasing the mandrel length. In this scheme, due to the longer length of the mandrel after applying the first pass, the second tube is placed into a die and simultaneously, with compression, the first tube is guided down the mandrel. By putting out the first tube from the mandrel the process is repeated. In this way, the first tube is placed in the die again and this time, the first tube simultaneously plays the role of mandrel for to the second tube, with compression, the second tube is guided into the bottom.

With this process, the first tube is under 2 pass and the second tube is under 1 pass. Similarly, the process is repeated until the tubes are produced with 1, 2, 3 passes.

3.4.2 Die Components

1) Two separate pieces or split off - 2) outer die – 3) Mandrel – 4) hollow punch – 5) lower support-6) upper support and pins.

Figure 3.5 shows the image of each component of the die.

a) Punch

b) Two separate pieces or split off die

c) Outer die

Figure 3.5, die components, a) punch b) two separate pieces or split –off die c) outer die

3.4.3 Method of making dies

For making die, at first outer surface of the outer die was machining. Inside the external die is in the shape of the frustum, in which by set the angle face cutting is done.

In order to produce two pieces of die, we maintain the same angle of machining outer frustum, and then we create the middle hole that is the location of Mandrel inside the frustum.

We do the heat treatment operation on die; with these operations we hardened it to 60-67 HRC. Then the frustum was cut by wire cut machine.

Head and bottom of half cone and middle hole were supposed to do grinding and machining operations for purpose of fitting the die.
A TCAP die was manufactured from hot worked tool steel and hardened to 60-67 HRC. Die parameters and their values are shown in table 1.

Table 3.2 Die parameters and their values

Parameter	φ_1, φ_3	φ_2	ψ_2	ψ_1, ψ_3	R/R_0	R_0	t_0
Value	135°	90°	90°	0°	1.2	8.75 mm	2.5 mm

The TCAP experiments were carried out with an INSTRON press under a pressing speed of 20 mm/min at room temperature. Experimental setup is shown in Fig3.6.

Figure 3.6 Experimental setup

Due to the experience gained during manufacturing of die, we considered an echelon, under the middle groove of two pieces die until the bottom fig (3.6)

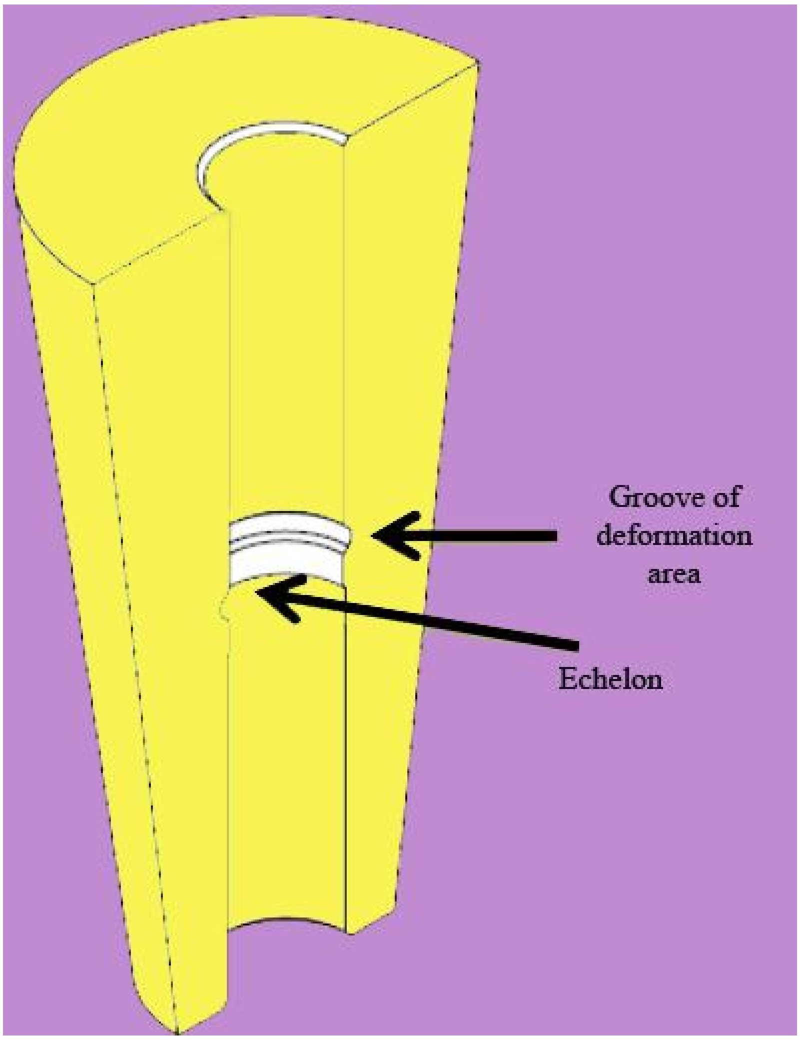


Figure3.7 cross-section of die

In this region, shaping is completed and with this way, tube motion in die is more convenient and applied force is reduced.

In mandrel also, we considered an echelon, under the lower area lobes until the bottom of mandrel, because after deformation of the tube and back to the initial state, due to a difference between inner diameter of tube and outer diameter of mandrel, tubes easily can be separated from the mandrel. Figure (3.8).

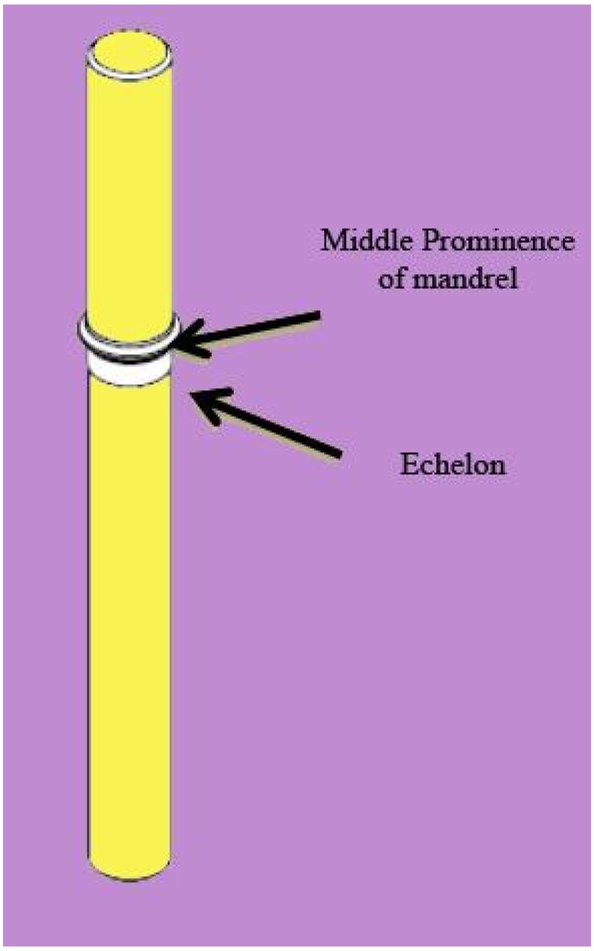


Figure 3.8 mandrel

The length of lower support is considered the same as the length of tube, until the tube has passed the process back to that.

Also lower support has a groove that covers the outer die and it lead to have an integrated die. Initially, we have designed the TCAP die without upper support, but with doing the tests we found that after applying the force, the second half of the die jumps up and cause die releasing and subsequently it stopped the operation.

Therefore an upper support was designed and by three pins was attached to the outer die. After improving the design, tests were successfully completed with no errors.

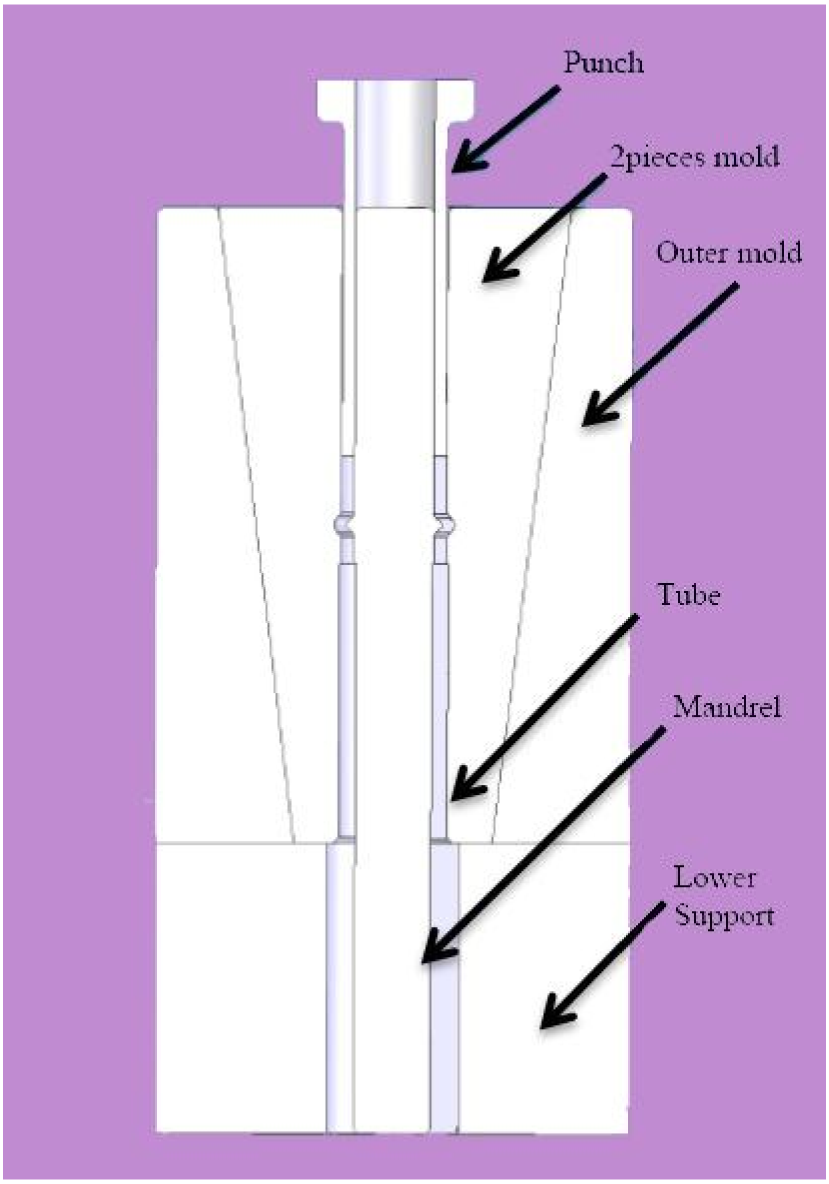


Figure 3.9 Original design without upper support

3.5 Design tests on samples

In this study, effects of number of passes in a constant temperature on the microstructure and mechanical properties are evaluated at constant strain rate.

Therefore, aluminum tubes under 1, 2, 3 passes in ambient temperature and a speed of 10mm/min are produced. Thus, after production of tubes, with doing micro hardness tests, pressure tests and metallographic tests we will investigate mechanical and microstructure change of aluminum tubes.

3.5.1 Vickers Micro-hardness test

From the produced tubes, rings were extracted from the center portion of the pressed materials. with a height of 10 mm , the cross-sectional sample was cold mounted, grounded until 2400 grad and polished with alumina , at room temperature .after polishing, the samples were analyzed by micro-hardness testing.

Shimadzu Micro-hardness test machine does belong to Metallographic Laboratory's Amir Kabir University School of Mining and Metallurgy (Figure 3.10).

Figure 3.10 Shimadzu Micro-hardness test machine

The samples were placed under the load of 100 grams for 10 seconds. Each sample was tested from three points, we gain the mean hardness.

Figure 3.11 micro-hardness test of 1 pass sample

3.5.2 Compression Test

Since the tube samples have a short length, we could not separate standard sample of tensile test, hence we have done stress testing on samples. Thus, from the processed tubes, rings with same length, diameter and thickness was prepared so that the rings have equal status in terms of dimensions. Stress-strain diagram of the separate rings of tubes under compression test at ambient temperature and strain rate of 10^{-3} were obtained. To reduce the effect of friction, a thin layer of Teflon and silicone lubricant was used between the two ends of the ring and also two plates of machine. Compression test sample and its dimensions are shown in Fig (3.12)

Figure 3.12 Compression test sample and its dimensions

For doing this test, Tension Testing Machine model 8502 belonging to Mechanical Properties of Materials Laboratory- Mining and Metallurgical Engineering faculty-Amir Kabir University (Figure 3.13) were used.

Figure 3.13 Tension Testing Machine model 8502

3.5.3 Metallographic Test

Metallography is used in the purpose of investigating the microstructure and the grain size. This operation includes; grinding, polishing, etching and observation in any device that we can do metallographic test such as optical microscopy, Scanning Electron Microscopy (SEM) - Transmission Electron Microscopy (TEM) and so on.We did grinding operation, with SiC Grinding Papers of the order 360, 400, 800, 1200, 1500, 2000 and 2400.In various stages of grinding, in each step, samples are rotated 90 degrees compared to the previous step. Grinding operations at each stage continues, until the complete disappearance of effect and along lines of the previous stages.

3.5.3.1 Polishing

At this stage fine scratches and very fine distortion layers remaining from previous section are separated.Before starting polishing, firstly, we thoroughly soaked polishing cloth with water and lubricant liquid, and then we spread some diamond paste on polishing cloth and polished the grinding surface. During the polishing, we use water and lubricants liquid. If this step and the previous step can be performed with sufficient accuracy, we will have a surface free of scratches and almost without any recognizable distortion metal layers.

3.5.3.2 Etching

Usually in metallography data structure after the final polishing is not clear under the microscope. Reflection of light will be so that the observation of microstructure, that is the main goal of metallography, is not possible.

In order to make visible grain boundaries, metallographic specimens are etched. This operation is carried out with immerge polished surface into a solution. As a result of this action, some of the metal is dissolved and goes out from the surface. If the etching solution is used appropriately, the metal surface is not uniformly resolved. Sometimes etch factor, attacks to the grain boundaries faster than the grain surface. After etching border appears like shallow steps in the surface. The vertical walls of these stairs do not reflect the light such as relatively smooth crystal surfaces, to the object lens of a microscope and thus the crystal boundaries are visible under a microscope.

3.6 TEM analysis

In addition, transmission electron microscopy (TEM) observations were performed to investigate precipitates. The disc specimens were prepared by an electrical discharge machining (EDM), mechanical grinding to 100 μm thickness of foil, and electro polishing using a 30% nitric acid solution in methanol, 25 volts for 20 seconds at - 30 ℃.

Figure 3.15 FEI Tecnai TF20, Transmission Electron Microscope

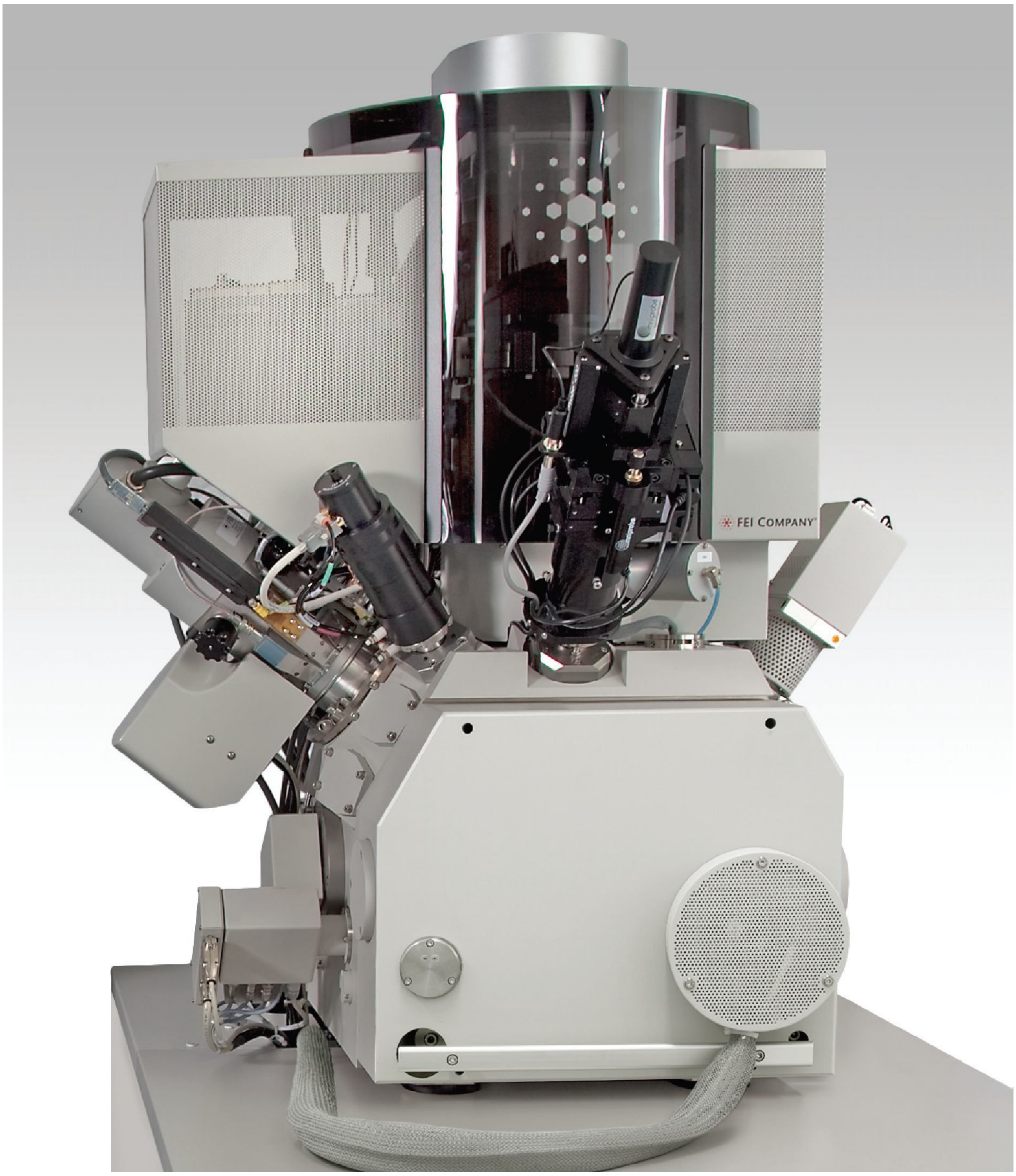


Figure 3.16 FEI Helios NanoLab 600iDualBeam

3.7 Grain Size Measurements

Grain structure is usually specified by giving the average diameter. Grain size can be measured by two methods.

(a) Lineal Intercept Technique: This is very easy and may be the preferred method for measuring grain size.

(b) ASTM Procedure: This method of measuring grain size is common in engineering applications.

3.7.1 Lineal Intercept Technique

In the mentioned method, lines are drawn in the photomicrograph, and the **number of grain-boundary intercepts**, N_l, is counted along a straight line.

- The mean lineal intercept is then given as:

$$\bar{l} = \frac{L}{N_l M} \qquad (3\text{-}2)$$

Where L is the length of the line and M is the magnification in the photomicrograph of the material.

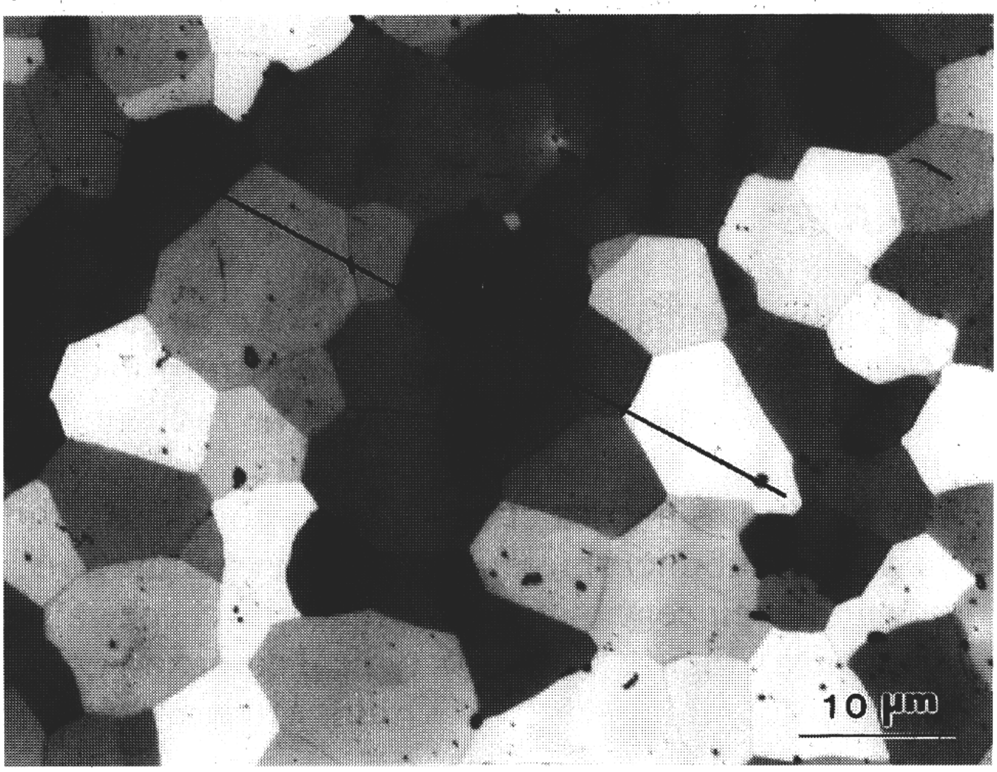


Figure 3.17 Micrographs showing polycrystalline TiC

In Figure (3.17) a line is drawn for purposes of illustration.

The length of the line is 6.5 cm. The number of intersections, N_l, is equal to 7, and the magnification M = 1,300. Thus,

$$\bar{l} = \frac{100\ X\ 10^3}{7\ X\ 1300} = 11\mu m$$

Several lines should be drawn to obtain a statistically significant result. The mean lineal intercept l does not really provide the grain size, but is related to a fundamental size parameter. S_v is define as grain-boundary area per unit volume, by the equation,

$$\bar{l} = \frac{2}{S_v} \qquad (3\text{-}3)$$

The most correct way to express the grain size (D) from lineal intercept measurements is:

$$D = \frac{3}{2}\bar{l} \qquad (3\text{-}4)$$

Therefore, the grain size (D) of the material of Figure (6-1) is:

$$D = \frac{3}{2} X\ 11 = 16.5 \qquad (3\text{-}5)$$

3.7.2 ASTM Procedure

With the ASTM method, the grain size is specified by the number n in the expression $N=2^{n-1}$ where N is the number of grains per square inch,when the sample is examined at 100 power micrograph.

Example

In a grain size measurement of an aluminum sample, it was found that there were 56 full grains in the area, and 48 grains were cut by the circumference of the circle of area 1 in^2. Calculate ASTM grain size number *n* for this sample.

Solution

The grains cut by the circumference of the circle are taken as one-half the number. Therefore,

$$N = 56 + {}^{48}\!/_{2}$$
$$= 56 + 24 = 80 = 2^{n-1}$$

$$But \quad n = \left({}^{\ln N}\!/_{\ln 2} \right) + 1$$
$$\therefore \quad n = \left({}^{\ln 80}\!/_{\ln 2} \right) + 1$$
$$= \left({}^{4.38}\!/_{0.69} \right) + 1 = 7.35$$

CHAPTER FOUR

RESULT AND DISCUSSION

4.1 Investigation of changes in mechanical properties

Figure (4.1) show the aluminum workpiece before and after TCAP processing.

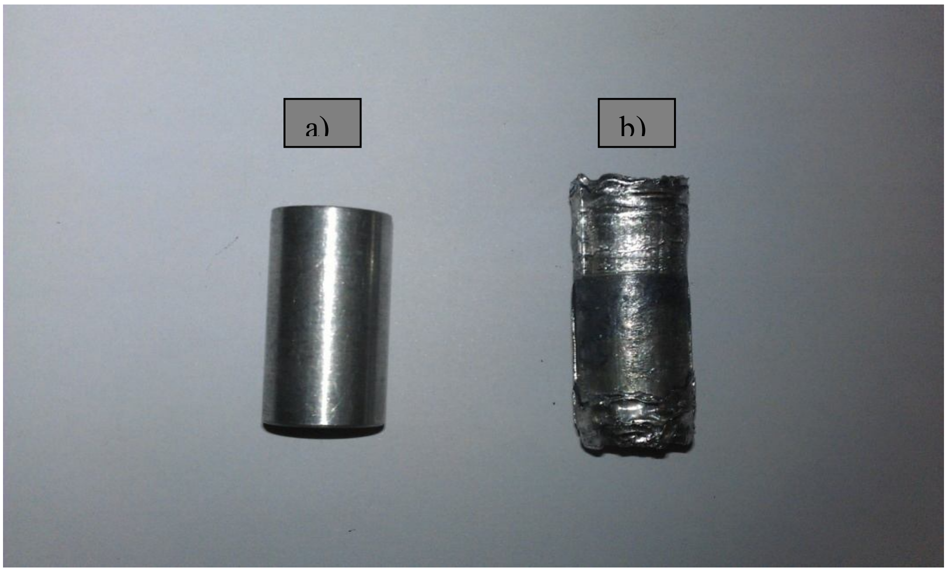


Figure 4.1: aluminum workpiece before(a) and after (b)TCAP processing

During the process, the tube diameter increases and returns to the initial size at the end of TCAP process. As shown in this figure, the cross sectional area of the tube before and after TCAP process remains constant though the tail part has some inconsistency.

4.2 Compression test

In this study, the effect of number of passes on aluminum tubes is investigated. Figure 4.2 shows the true stress–strain curves obtained from the compression tests of specimens subjected to 1–3 passes. For comparison, the flow stress–strain curve of a specimen before TCAP is also plotted.

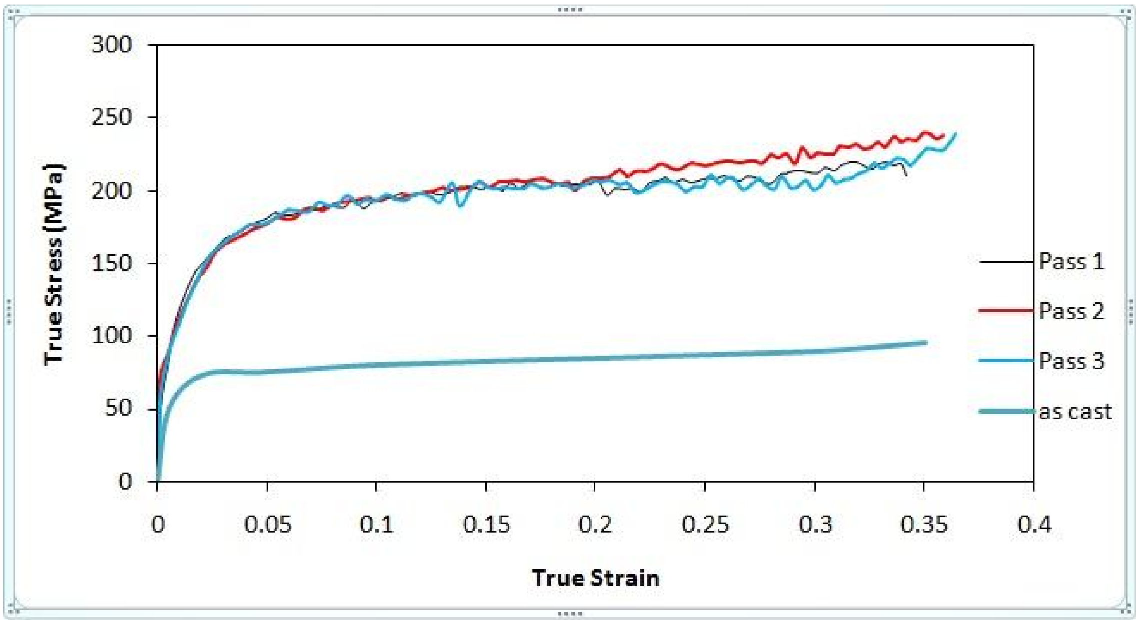


Figure 4.2 Stress-strain curve of 1, 2, 3 pass samples with accompany of as-cast sample

The graph illustrates changes in stress and strain of pure aluminum before and after doing TCAP process.

As you can see the curve of the as-cast aluminum is quite different from the samples that have undergone the process.

It is obvious that by doing the passes in our as cast samples the amount of stress increases enormously.

After the first pass, yield and ultimate strength of the specimens increases significantly.

It should be noted that the improved mechanical properties of pure AL at room temperature, simultaneously is concerned to shrink the size of the grain (about nm 350) and β-phase distribution that it limit the dislocation motion. Also 1 to 3 pass samples have similar properties.

Overall, the graph shows how the amount of stress increased dramatically while number of passes is go up. From the above diagram yield strength and ultimate strength are mined.

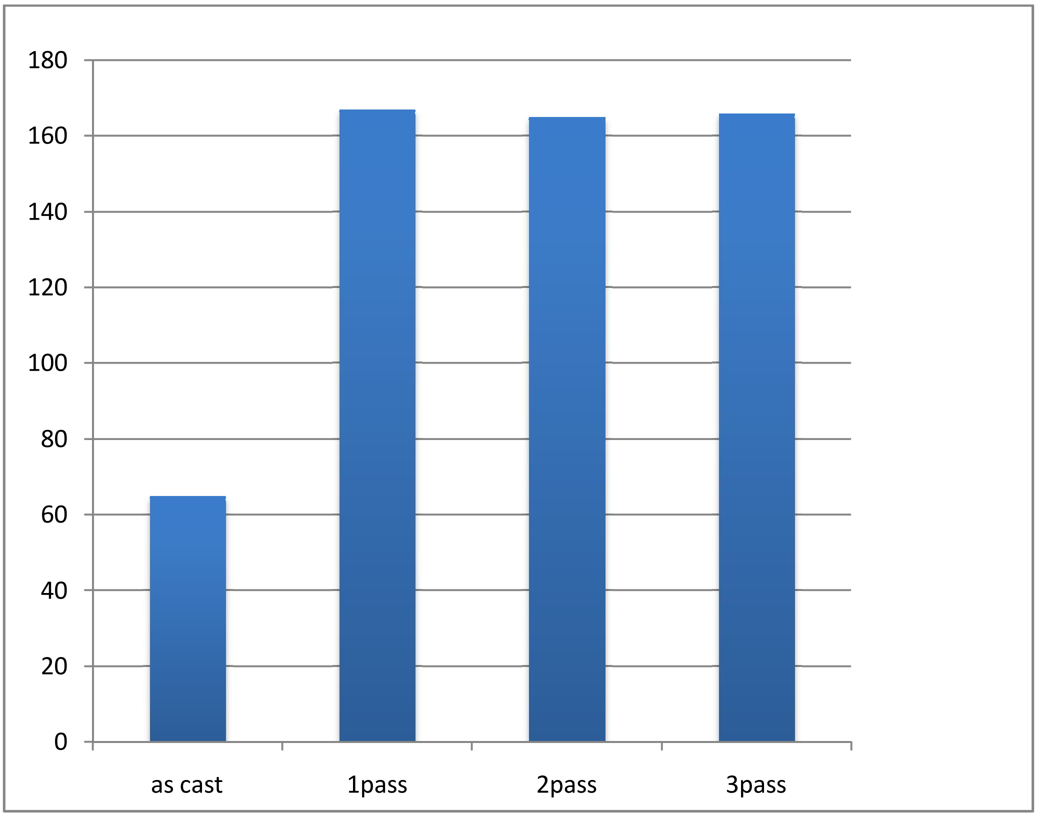


Figure 4.3 Variation of yield strength during consecutive passes.

Figure (4.3) shows the yield strength of the 1 to 3 pass samples with accompany of as-cast sample.

As can be seen from the chart, the highest increase in strength can be seen in the first pass. Yield strength of the samples has risen from65 Mpa in as-cast sample , to 165 in 3pass sample., However, changes in the yield strength of samples 1, 2, 3 Pass, is little and is almost linear, therefore the next consecutive passes don`t have tangible impact on improving the mechanical properties of the alloy.

This can be attributed to the microstructure of the samples. The microstructure of the tube cannot have a significant change after the first pass.

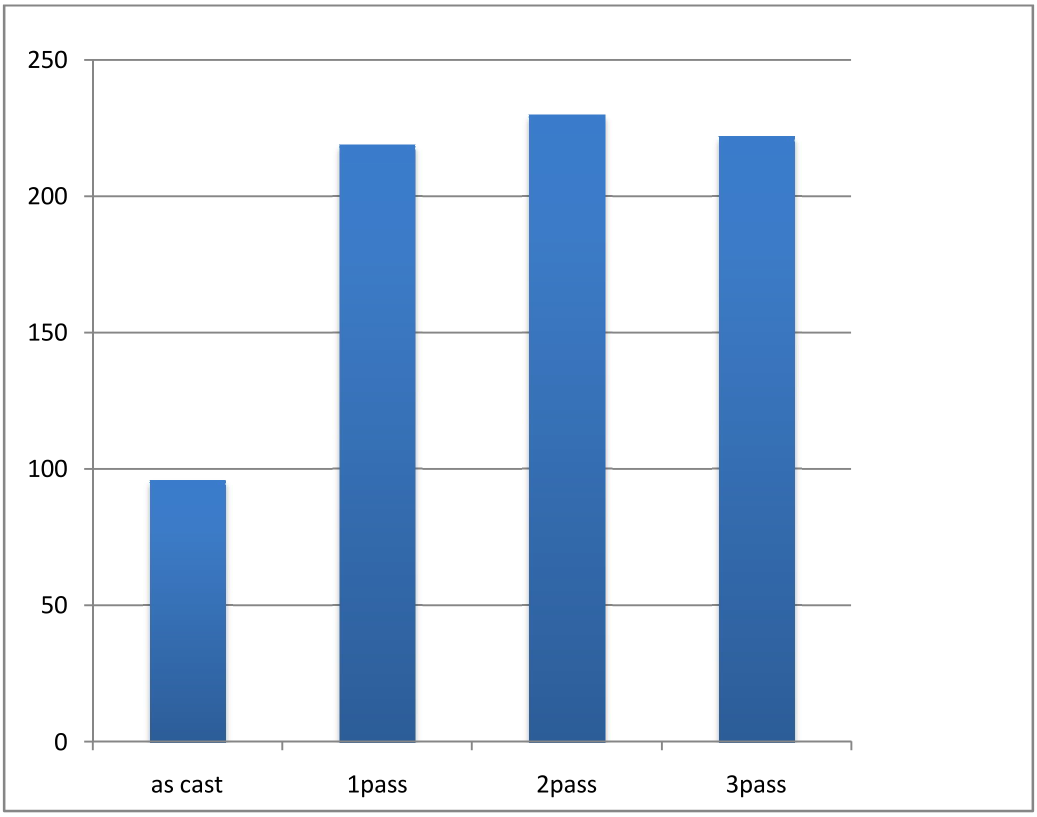


Figure 4.4 ultimate strength changes during the consecutive passes

Figure (4.4) illustrates the ultimate strength results of samples. The ultimate strength of sample 1 is increase to 219 Mpa from its initial amount that is around 96 Mpa. In the other words we have 123 Mpa increasing ultimate strength in the first pass in comparison to as-cast AL.

Figure 4.5 compression test result for sample 1p

Figure 4.6 Compression test result for sample 2 p

Figure 4.7 compression test result for sample 3p

4.3 Micro-hardness test

As described in the previous chapter, after cutting the identical profiles from sample and polishing, the samples were prepared for microhardness testing. Results of 1 to 3 Pass samples with accompany of as-cast sample can be seen in figure 4.8

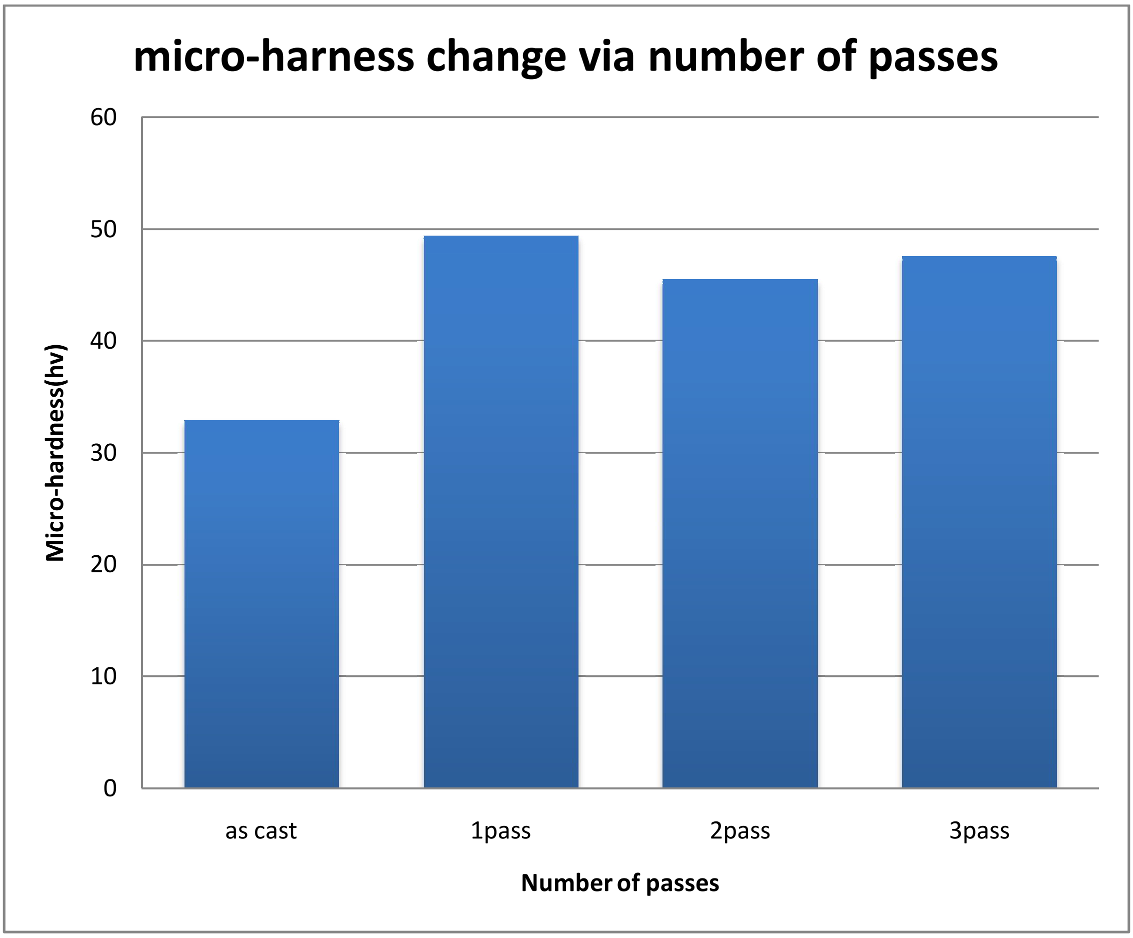


Figure 4.8 micro-harness changes via number of passes

The microhardness values of the samples increase significantly with the number of passes, but the amount increasing for each passes is different. The increase in hardness after the first pass is remarkable. TCAP process increased the hardness of samples from 32.99 in as-cast sample to 49.4 in first sample.

That means a 50% increase in micro hardness after the first pass in comparison to as cast sample. It must be noted that a slight decrease in micro hardness in 2 and 3 pass in comparison to 1st pass is due to measurement error. Or May this fact is due to the formation of cracks in this samples. An increasing trend in the micro-hardness values with increasing number of passes shows the enhancement of the TCAP process as the number of passes increases.

Increase in the number of passes in TCAP process after one pass, has no significant impact on the micro hardness of the samples. This condition can be seen also in ECAP process for cu[43]and for AL[44].

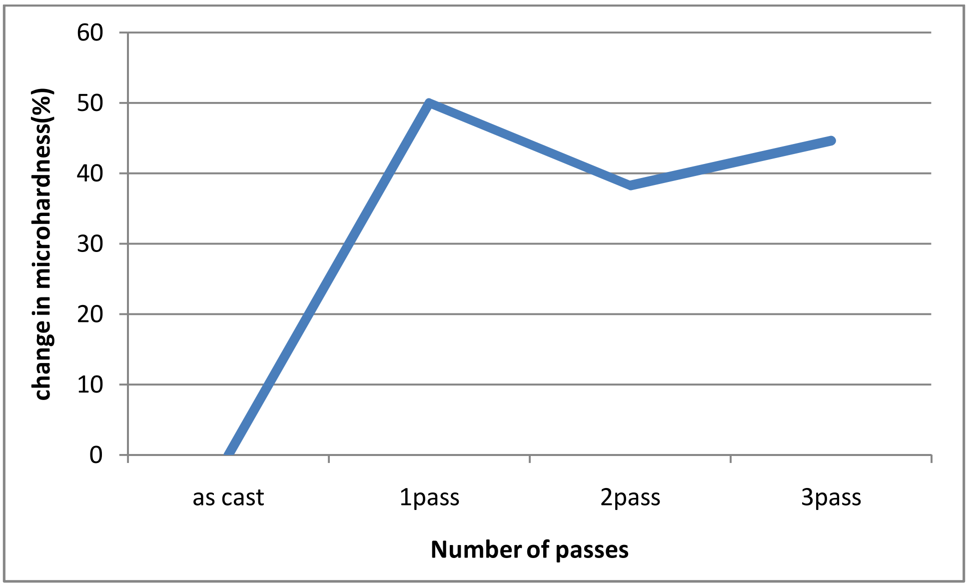


Figure 4.9 micro-harness change via number of passes (%)

As illustrated in figure 4.9, 50% growth in hardness is the main benefit of doing passes on samples. This increase fluctuated in the 2nd and 3rd pass but the most obvious results of this graph are that passes have a positive effect on amount of hardness.

To investigate hardness dispersion or strain distributions, on the cross-section of the TCAPed samples, Standard deviation (SD) as a statistical parameter, was used by equation (4-1): [45].

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i - M)^2}{n-1}} \qquad (4\text{-}1)$$

Where X_i ,M ,n are respectively the hardness values in i`th measurements, average hardness measurement and number of hardness measured.

In general, lower values of SD result in better dispersion hardening and strain distribution is more uniform.

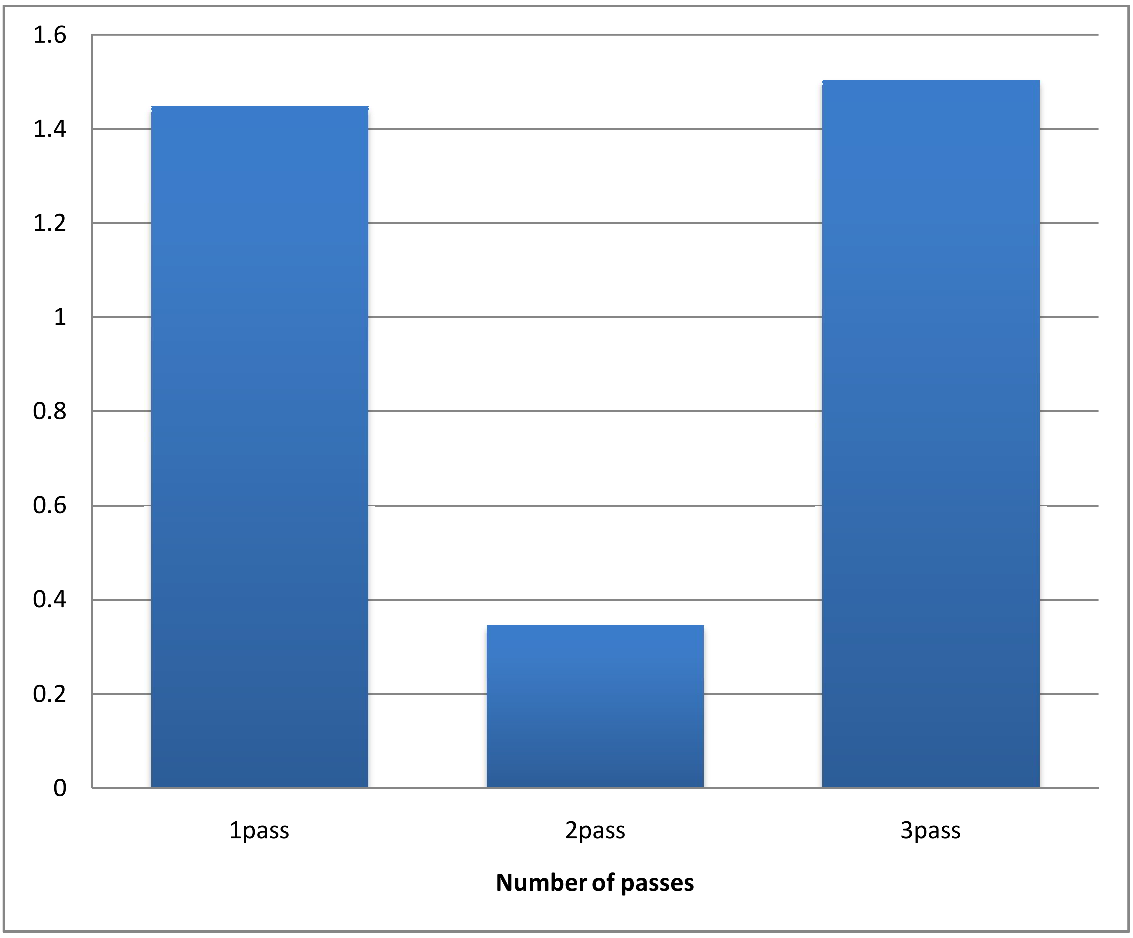
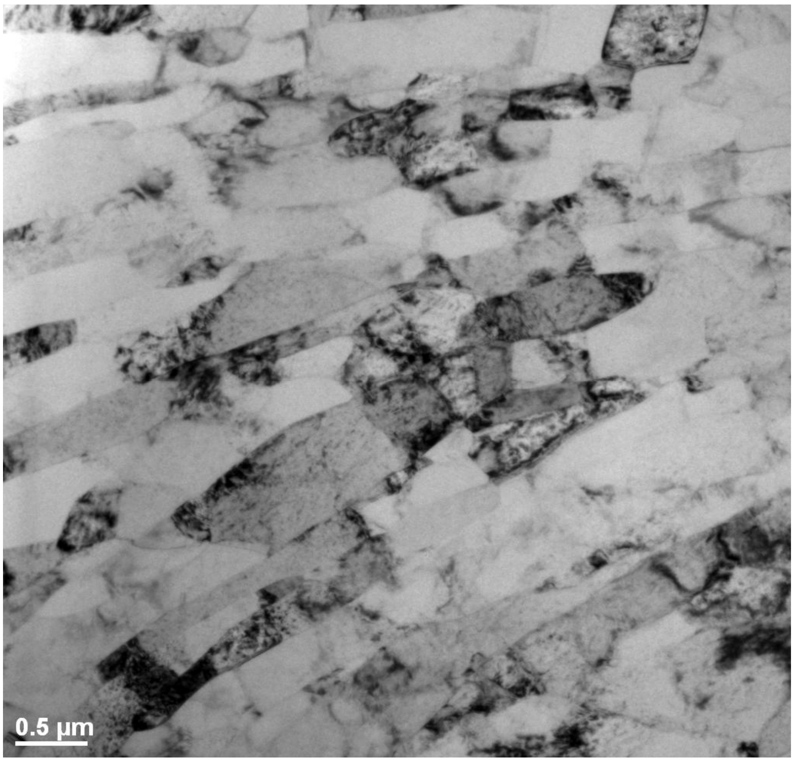


Figure 4.10 SD values of hardness distribution in different regions of the 1, 2, and 3 pass samples.

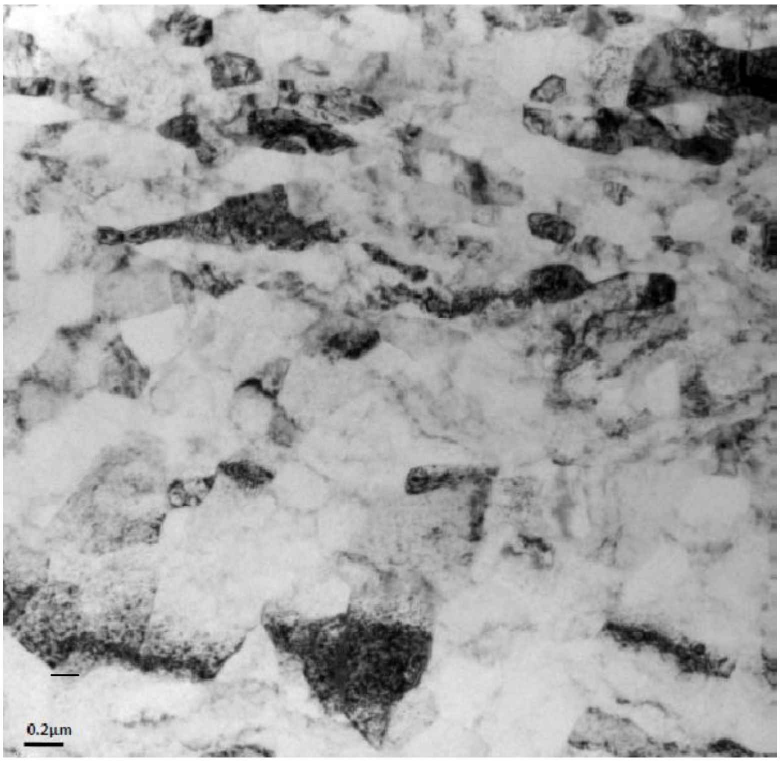
From Figure 4.10 we can see that the maximum value of hardness distribution is belonging to 3pass sample with amount of 1.504 and lowest value is belonging to 2pass sample with 0.346. So we can say that the 2pass sample has more uniform distribution than others.

4.4 Evaluation of microstructure

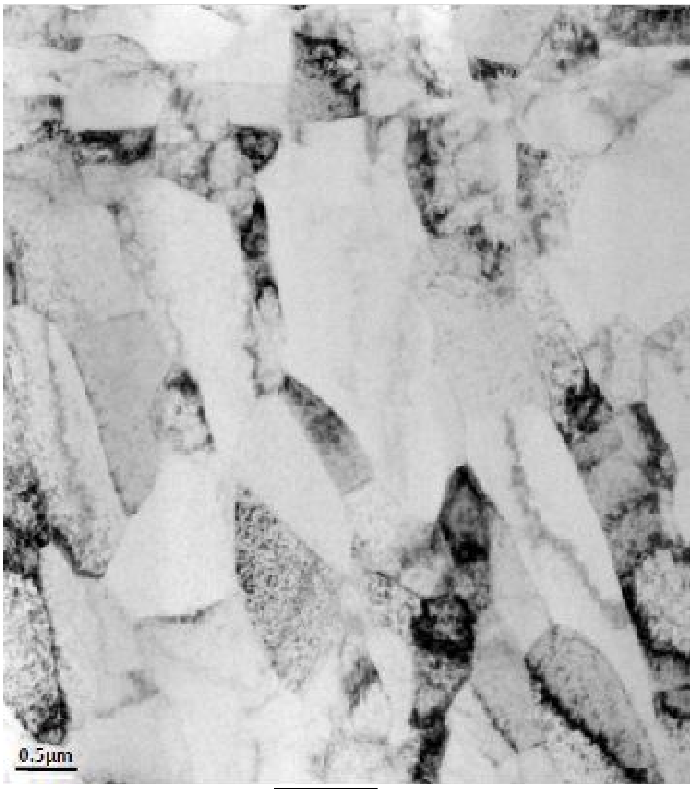
Figures 4.11(a)-(c) give the TEM micrographs of the cross-sectional microstructure of the TCAP processed tube after one-three passes respectively. As can be seen, the TCAP process could significantly refine the microstructure. From this figure, we can see that one pass of the TCAP process could refine the microstructure and new subgrains with 350 nm in size were formed from the initial annealed microstructure with grain size about 56 μm. TEM micrograph of the first pass TCAP processed sample contains an elongated and subgrain structure with tangled dislocations in which distinct regular-shaped walls was observed[46]. Increase in the number of passes to the second pass cause to change the elongated subgrains with ~350 nm size to a combination of new grains and subgrains with about 320 nm in size. When the number of TCAP passes increases to three elongated grains and subgrains are almost disappeared, and equiaxed grains with grain size about 300 nm are formed. The SAED patterns shown in figure 4.12 represent that there are predominantly high-angle boundaries after TCAP processing through two and three passes[47], but a higher fraction of low-angle boundaries when processing through one pass.



(a)



(b)



(c)

Figure 4.11 Bright field TEM micrographs of the cross-sectional microstructure of the TCAP processed tube after (a) one, (b) two and (c) three passes.

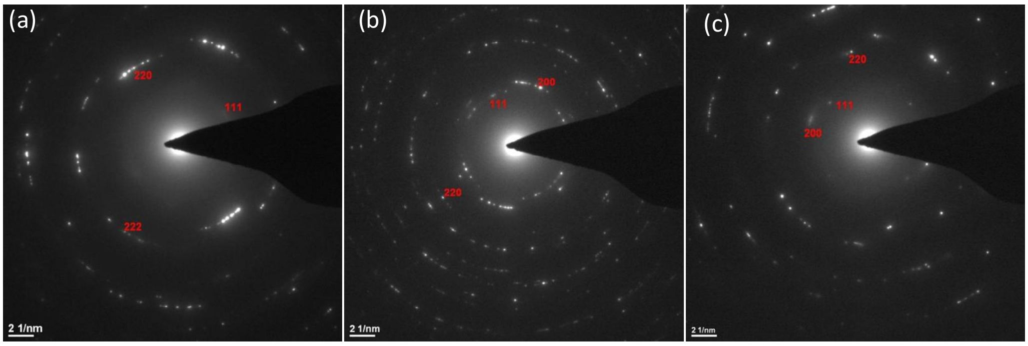


Figure 4.12 Corresponding SAED pattern of TEM micrographs Fig. 4.11 (a) - (c).

CHAPTER FIVE

CONCLUSION AND SUGGUSTIOS FOR FUTURE WORKS

5.1 SUMMARY

TCAP process of pure aluminum in this study during three consecutive passes at room temperatures was studied.

The die was designed and built .After doing designed TCAP test, in order to investigate the mechanical and metallurgical properties of production tubes, compression, micro hardness and metallographic tests were performed on them.

The results show that the first pass has the greatest impact on improving the mechanical properties, such that yield strength of the 1pass sample in comparison to raw sample was associated with an increase around 100 Mpa. And hardness of samples was increase 16.5HV.

The process of changes in yield strength and hardness of the samples through the passes 2 and 3 in comparison to 1st pass show little change.

Study of TEM images of the samples shows that the grain size of the initial annealed was about 56 µm , while 1passes sample has a grain size of about 300-400 nm.

Significant improvement in the mechanical properties of the first pass can be caused by decreasing grain size in it`s microstructure.

5.2 SUGGESTIONS

- Perform process simulation for all passes in order to determine the applied strain and required force for pressing process.

- Modifications of the process, in order to produce pipes with bigger diameter and length for using in the Industry.
- Improvements designed to reduce the force required for the process.
- Investigate grain refinement mechanisms in the process.
- Perform TCAP process on dual-layer pipes

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