

Quantification of residual stresses in relation with
microstructural changes during hot rolling of titanium alloys

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In

Production Engineering

By

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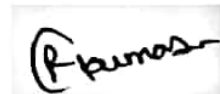
August 2019

CERTIFICATE

I hereby declare that the work done in the Dissertation entitled “Quantification of residual stresses in relation with microstructural changes during hot rolling of titanium alloys” submitted towards partial fulfilment of requirement for award of degree of Master of Engineering in Production Engineering, Thapar Institute of Engineering and Technology, Patiala, is an authentic record of the work carried out by me under the supervision and guidance of Dr. Gulshan Kumar, Assistant Professor, BITS Pilani Dubai and Dr. Sachin Singh, Assistant Professor, Mechanical Engineering Department, Thapar Institute of Engineering and Technology, Patiala.

The matter embodied in this report has not been submitted in part or full to any other university or institute for the award of any degree.

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Dedicated to
My loving
Parents for
their love and
support

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ABSTRACT

A fully recrystallized titanium alloy (Ti-6Al-4V) was hot rolled at different reduction rates of 20%, 40% and 60% deformation. The samples were prepared with the help of mechanical polishing followed by electro-polishing for subsequent characterization. A gradient in microstructure have been observed which is reflect in residual stress development across the thickness of the rolled sheet. The above measurements were done using EBSD technique and X-ray diffraction, respectively. It has been observed that the as received material contains bimodal grain structure. A grain refinement has been observed across the thickness of the rolled sheet with increase in percentage of deformation. Finer grains were observed at 60% deformation at mid-thickness ($T/2$). The stress gradient in the rolling direction (RD) increases from top surface (T_0) to mid-thickness ($T/2$) of the rolled sheet. However, it showed a decrease in gradient in the perpendicular direction. The stresses were compressive in nature throughout the thickness.

Keywords: Titanium alloy, Hot rolling, Deformation, Microstructure and Residual stress.

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NOMENCLATURE

Ti -	Titanium
Al -	Aluminium
V -	Vanadium
CP Ti -	Commercial Pure Titanium
HCP -	Hexagonal Closed Packing
BCC -	Body Centre Cubic
SEM -	Scanning Electron Microscopy
RS -	Rolling surface
NS -	Normal surface
TS -	Transverse surface
EBSD -	Electron Backscatter Diffraction
XRD -	X-ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 FORMING: It is a process of getting the desired shape and size of material through the plastic deformation [1, 2]. The load applied to the material may be either compressive, tensile, shear or a combination of any of these. To obtain the plastic deformation, the load should be such that it is greater than the yield strength of the material but should not reach the fracture strength of the material. The forming process is economical because good product quality and dimensional accuracy can be easily achieved without any loss of material [3]. Earlier the products used to form by the forming process and the product finishing is given through the machining process. But now tools and dies are made by the hard materials so that the product can be effectively made having great dimensional precision with the surface quality so that machining time can be effectively limited.

1.2 CLASSIFICATION OF FORMING PROCESS

1.2.1 Hot forming: The forming process in which the working temperature of the material is above its recrystallization temperature. A large amount of plastic deformation takes place without any strain hardening effect. There is a decrease of strength of the material due to which machine with low force can easily convert the material into the desired shape and size. There is also a high coefficient of friction in the hot forming process [3]. When the load is applied then the grains elongate in the normal direction of applied load. During the grain recovery process the residual stress is decrease [1].

1.2.2 Cold forming: The forming process in which the working temperature of the material is room temperature. The strength of the material is high compared to the hot forming process. Strain hardening effect came into the material which makes the material brittle. The coefficient of the material is also low as compared to the hot forming process. There is a large amount of force required in the cold forming process to convert the material into the desired shape and size [3]. When the reduction takes place then residual stress increase because of the increase in the stiffness of the material.

1.2.3 Warm forming: The process in which temperature of the material is above its room temperature but below its recrystallization temperature. The advantage of worm forming is that

there is a reduction of force required compared to cold forming. Also, there is less oxidation compared to hot forming [4].

A brief introduction to various type of forming processes such as forging, rolling, extrusion, drawing and bending is given below:

Forging: In this forming process material is placed between the tool and die as shown in figure 1.1. A compressive force is applied to convert the shape and size of the material. In forging the die used may be either closed die and open die. Typical parts made by this process are like gear, turbine disk and automotive engine component.

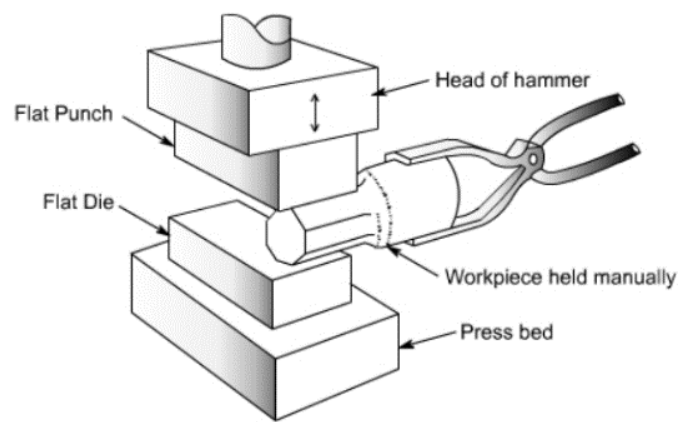


Figure 1.1 – Forging [5].

Rolling: In this forming process, the material is passed between the rollers(see figure 1.2). The reduction of material dimension depends upon the gap of rollers. This process is mainly used for the production of railway track, angel, landing gear and fan blades in aerospace industry etc.

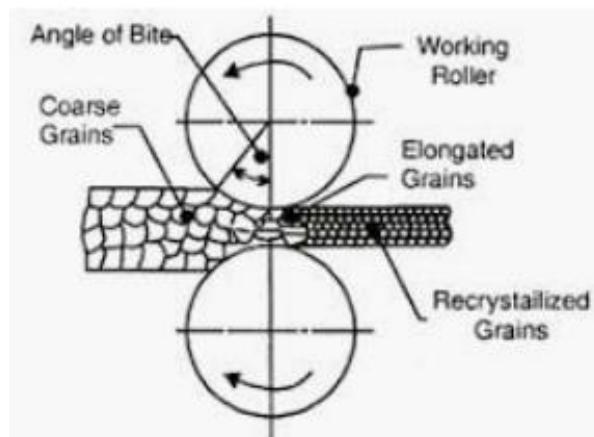


Figure 1.2 – Rolling process [5].

Extrusion: In this forming process the material is placed in a closed chamber and force is applied to the material to come out through an opening known as die having the constant cross-section. Thin walled cans, tubes and cups are made using this process.

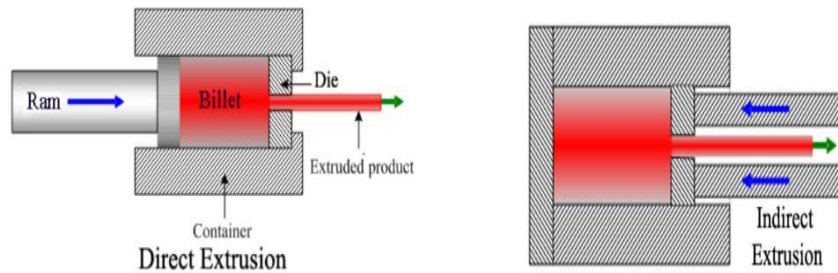


Figure 1.3 – Extrusion process [5].

Drawing: In this forming process the material is pulled out through the orifice of a die to reduce the cross-section. When there is a large reduction of cross-section required than the number of passes are increased as shown in figure 1.4. Musical strings and seamless tubes are made using drawing process.

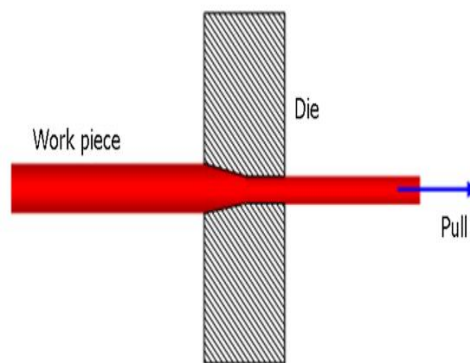


Figure 1.4 – Drawing process [5].

Bending: In this forming process metal is bent with the help of punch and die to get the desired shape of the material [3]. The process is shown in figure 1.5. Tubes and other hollow sections are bent by the bending process.

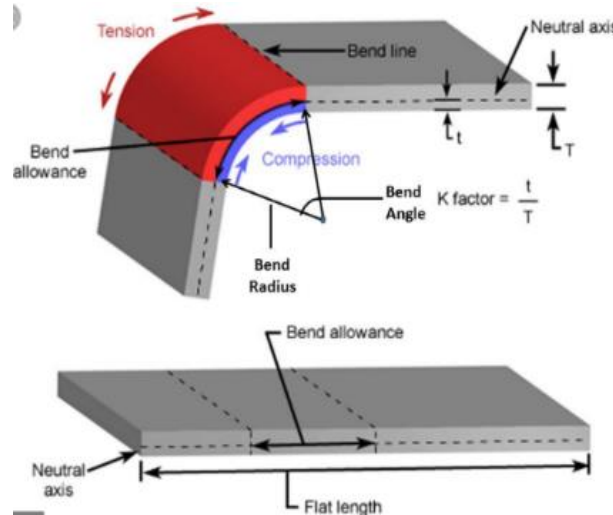


Figure 1.5 – Bending process [5].

1.3 ROLLING: During the 14th century, the goldsmith used the hand-driven rolls [6] for the rolling of soft metals like silver, gold etc. The process of rolling emerged well with the development of engineering and technology. By the end of the 16th century, the rolled sheets of gold and silver having uniform thickness were made by a Frenchmen named Brulier.

Among all the forming process that discover till now rolling is one of the most used forming process [6]. Near about 95% of all the ferrous and non – ferrous alloys used rolling for their working shape. The useful shapes of metal which can be made by rolling are sheet, strips and plates. It can also be used to make railway tracks, angle, bar etc. [6]. As shown earlier in figure 1.2, the material is passed between the rotating rolls the compressive stresses are applied to the material to deform it plastically. The shape of the rollers can be flat and grooved [6] which are placed at a fixed distance and driven by the electric system (gearbox, motor etc.). To get the desired shape the material is passed several times through the rollers having gap lesser than the cross-sectional area of material at the entrance of the mill. As the number of passes increases the gap between the rollers decreases to achieve the subsequent deformation [6].

1.4 CLASSIFICATION OF ROLLING MILLS: The assembly of the rollers is placed on the bearings which are held by the bearing blocks and these bearing blocks are held in the housing with all other facilities like roll driving arrangement and roller adjustment facility. This whole set up is called a rolling mill stand. Rolling mills are classified as given below:

1.4.1 Two-High rolling mills: Two opposing rolls are used in this rolling mill. The direction of rollers can be changed in reversing mill and the workpiece can enter from both sides. In this

rolling mill, the productivity is high. While in case of non-reversing mill, the direction of the rollers cannot be changed and the work piece cannot enter from both sides as shown in figure 1.6.

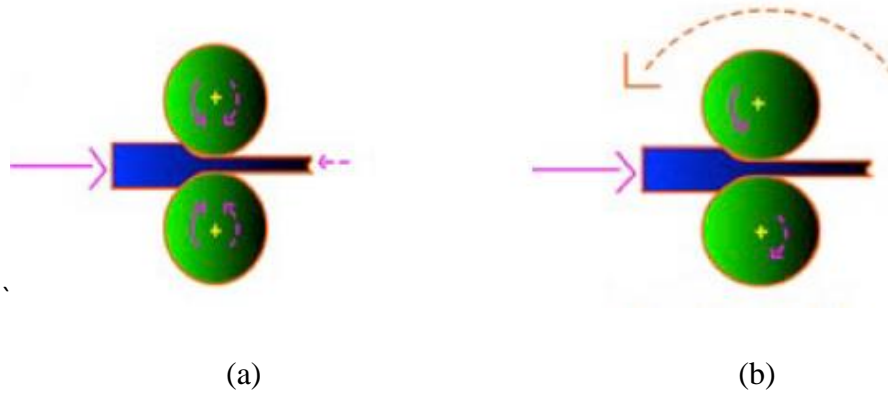


Figure 1.6 – Two-High rolling (a) Reversing (b) Non-Reversing mill [1].

1.4.2 Three-High rolling mills: Three rollers are used in this rolling mills. The material enter from the one pair of mill and then the workpiece is reversed and passed through the other pair (see figure 1.7).

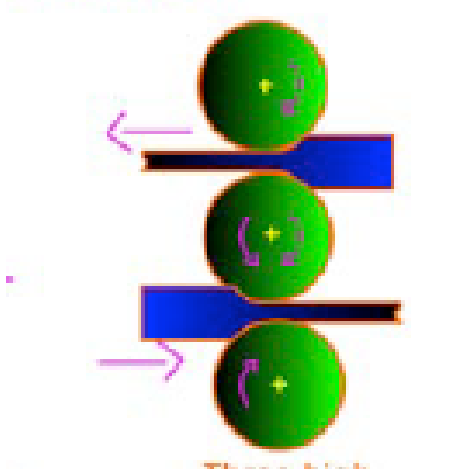


Figure 1.7 – Three - High rolling mill [1].

1.4.3 Four-High rolling mills: Small diameter rollers are used in this rolling mills for reduction and therefore input power required is less. Since the strength of small diameter rollers is poor so the large diameter rollers are used to backup as shown in figure 1.8.

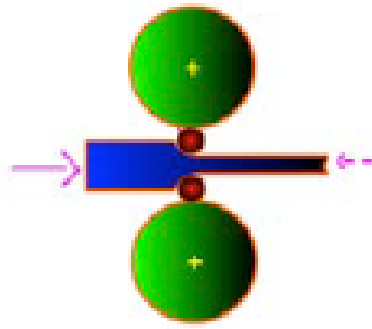


Figure 1.8 – Four-High rolling mill [1],

1.4.4 Plantary mills: In this rolling mill, a large heavy roller is covered by many small diameter rollers around the circumference of it. A slab can be directly converted into a strip in a single pass [1] as shown below in fig

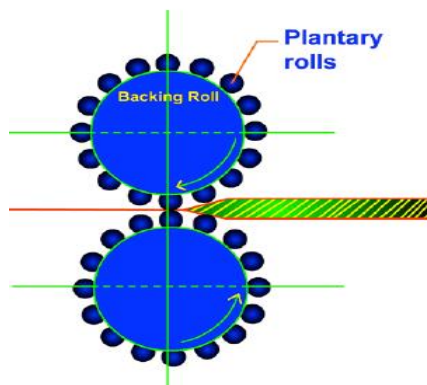


Figure 1.9 – Plantary mills [1].

1.5 CLASSIFICATION OF ROLLING PROCESS: The rolling process can also be categorized based on the condition given below:

- (1) Material temperature – hot rolling, cold rolling
- (2) Rolling direction – longitudinal and transverse rolling
- (3) Product shape – flat, hollow and section rolling

1.5.1 HOT ROLLING: In this rolling process, the rolling temperature of the material is higher than the recrystallization temperature of the material. The raw material used for the rolling process is ingot which is made up of molten metal [6]. The ingot is rolled into bloom and then bloom are converted into billet. The large sized products made from the bloom

includes plates, beams, railway tracks and angle section and the small size products are bars and strips.

Advantages of hot rolling:

- (1) The strength of the material decrease through which the compressive stress required in rolling is less and thus low capacity rolling stand used.
- (2) There is a large amount of plastic deformation takes place without any strain hardening effect.
- (3) Less energy consumption and low cost.
- (4) The processing performance of metal and alloys can be increased by hot rolling.

Disadvantages of hot rolling:

- (1) In hot rolling, mechanical and microstructure of the material are non-uniform.
- (2) Oxides are formed on the surface of the material [6].

1.6 TITANIUM: Iron was considered as the most useful material on the earth because of its different mechanical and physical properties but Fe has some limited issue like its heavyweight and less corrosion resistance property. The researcher lookout for another material and they found Titanium (Ti) has many advantages compared to Fe in many engineering applications [7]. It has high strength, low density and high corrosive resistivity compared to Fe.

In 1791, Reverend W. Gregor a mineralogist discovered a material sandy and black in colour. He calculated that the material has 50% magnetite property and remaining is reddish brown powder which gives a yellow substance after dissolving with acid. So he named it Menachanite. In 1793, Marlin Meinrich who found few other elements named it Titanium which means Earth. The name of Titanium also refers to the Greek mythology name titans. Titanium is found in Rutile and ilmenite oxide form in earth crust [7].

Due to its unique properties of high strength to low density ratio and high corrosion resistance, Titanium and their alloys have a wide range of applications in the aerospace industry, medical surgery as well as in the automotive industry [7]. There are many grades of titanium but Ti-6Al-4V is the most usable alloy because of its good machinability and excellent mechanical property [7]. Having its weight reduction ratio, it is used in aerospace and automotive. Among

all the Ti-alloys, Ti-6Al-4V (mainly 50%) and CP titanium (20-30%) used in aerospace and automotive industries.

1.7 CRYSTAL STRUCTURE: Transformation of one crystal structure to another crystal structure is called allotropic transformation and the temperature at which it takes place is called transition temperature. Like other metal, titanium also shows the different crystal structures at different temperatures. Pure titanium and titanium alloy have HCP crystal structure [7]. The transition temperature is $882 \pm 2 \text{ }^\circ\text{C}$ and the allotropic transformation is BCC crystal structure as shown in figure 1.10. The young's modulus of Ti is 145GPa.

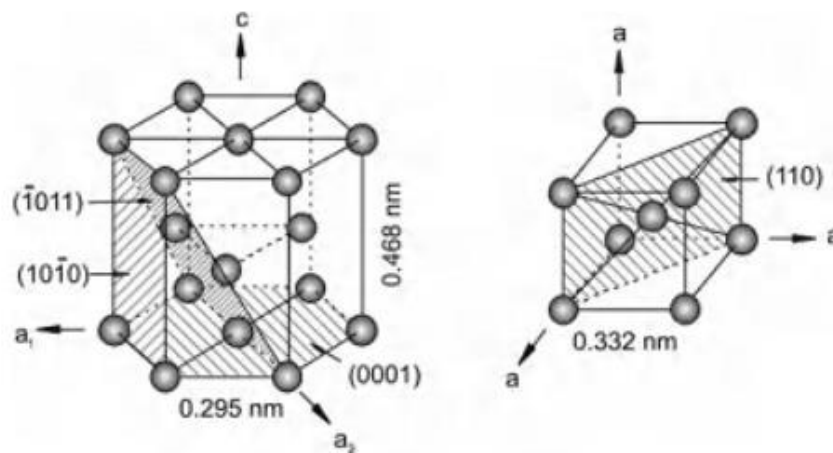


Figure 1.10– Crystal structure of HCP ($\alpha - Ti$) and BCC ($\beta - Ti$) [12].

Alloying element effect – the addition of several elements can increase the mechanical properties of titanium.

- (1) Addition of Al can increase the temperature of titanium by stabilizing the $\alpha -$ phase i.e $\beta -$ phase came.
- (2) Some element like Tin and Zirconium have little effect on the transition temperature but increase the strength of $\alpha -$ phase [7].

1.8 CLASSIFICATION OF TITANIUM ALLOYS: The presence of α and β tells the classification of Ti- alloy at room temperature. They include α , near α , $\alpha - \beta$, β .

1.8.1 Alpha alloys: When the commercially pure titanium is alloyed with the stabilizers like oxygen then the hardness and strength of titanium increases. By changing the amount of stabilizers various grades of titanium can be produced with strength varying from 290 and 740 MPa. The structure present in the material is alpha but due to some amount of iron stabilizers,

some microstructure of beta is also present. Aluminium is the most alloying element added as an alpha stabiliser and found many titanium alloys.

1.8.2 Alpha – Beta alloys: The elements like V, Fe, Mo developed the alpha-beta phase by stabilising the beta phase. These materials have moderate strength of 620-1250 MPa. Thermomechanical and heat treatment technique is used to prevent the material from fatigue failure and fracture so that the material mechanical properties will become high and use in a wide range of applications.

1.8.3 Beta alloys: When a beta stabilizer is added to the titanium then the beta alloy is produced. These materials are cold workable as compared to the alpha-beta alloy. They have high strength so they can be heat treatable at high temperature and has high corrosion resistance to other alloys [8].

1.9 APPLICATION OF TITANIUM ALLOYS:

1.9.1 Automotive Industry: Titanium alloy is used in the making of F-1 car’s engine parts but its use is restricted due to its’s high cost. So titanium is used only for special purpose car and racing cars [9]. Ti-6Al-4V is also used in making of intake valves, armour, and making connecting rods. Some of the commonly used titanium alloys in the automotive industry are given in table 1.1 –

Table 1.1 – Application of Titanium alloy in automotive industry [9].

Ti-6Al-4V	Suspension springs, Armor, connecting rods
Grade 2	Exhaust system
γ (TiAl)	Turbocharger rotors

1.9.2 Aerospace Industry: Titanium alloys are used in the field of aerospace mainly in the making of airframes and engine due to light weight of titanium as compared to steel and aluminium. It also has properties like space limitation (size to carry required load the wing envelop), high corrosion resistance and temperature which make the titanium suitable for the aerospace industry. Landing gear of Boeing 747 and 757 are the best example of space limitation and using of titanium alloy makes the solution [9] as shown in figure 1.11. Some of the commonly used titanium alloy in aerospace sector are given in table 1.2 –

Table 1.2 – Application of titanium alloys in aerospace industry [9].

CP-Ti	The environmental control system, clips and brackets
Ti-6Al-4V	Landing gear, wing, fuselage
Ti-5Al-2Sn-2Zr-4Mo-4Cr	Fan and compressor disc having temperature below 400°C
Ti-13V-11Cr-3Al	Used in SR-71 aeroplane for making body skin, frames and wings
Ti-811	Fan blades in engines

1.9.3 Medical Field: Titanium is also used in biomedical fields because of its high biocompatibility, high strength with reduced elastic modulus. CP-Ti and Ti-6Al-4V are the most commonly used titanium alloys for different applications given in table 1.3. The various tools used in medical fields made from titanium alloys are shown in figure 1.11.

Table 1.3 – Application of titanium alloys in medical fields [9].

Heart valve and connectors	CP-Ti
Dental, knee and hip joints	Ti-6Al-4V

[9]



Figure 1.11 – Application of titanium alloy in various industries [14].

1.10 RESIDUAL STRESS: These are the stresses present in the body after manufacturing/material processing in the absence of external loading [10]. The generation of residual stress can also take place from thermal gradients, inhomogeneous plastic deformation in the material etc. There are the following type of residual stress which are as follows –

1.10.1 Macroscopic: These type of residual stress occurred in several grains. By measuring the magnitude of strain residual stress can be measured at any point [10]. These type of stress are known as Type 1 residual stress as seen in figure 1.12. These type of residual stress can be measured by continuum mechanics. These stresses neglect the underlying microstructure. There is uniform dislocation takes place in the material [11].

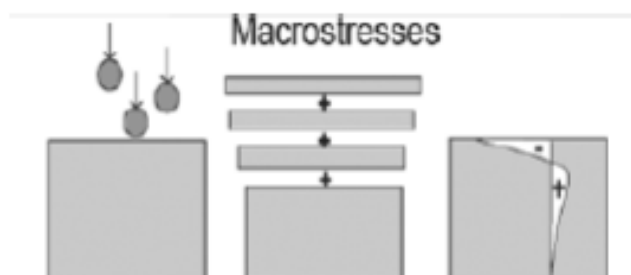


Figure – 1.12 Macroscopic residual stress in shot peening Type I [10].

1.10.2 Microscopic: These type of stresses occurred in different grains with different magnitude. There are two type of microstructure i.e. Type II in which the stress occur due to dislocation of grain from the other. Type III stress occur at sub-microscopic level i.e. point defect and line defect [11] as see in figure 1.13.

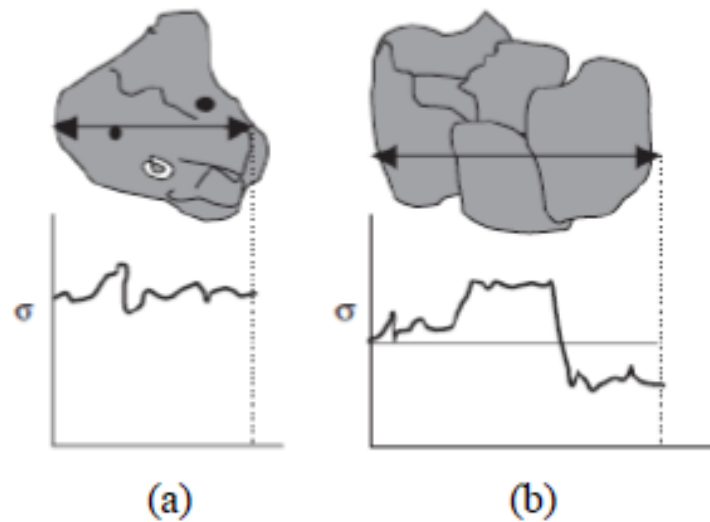


Figure 1.13- Micro-stress inside the grains (a) Type II and (b) Type III [10].

1.11 ORIGIN OF RESIDUAL STRESS: The development of residual stress takes place during the manufacturing process like machining operation, heat treatment and plastic deformation in which shape and mechanical properties changes [13]. The origin of residual stress takes place in two steps – (1) misfit- the difference in interplaner spacing and (2) constraints which oppose the retaining of misfits [10]. The macroscopic stress have more dimension of misfits and constraints as compared to the microscopic stresses. The macrostresses are located in different parts of the component. Both the steps are produced by chemically, thermally and plastically [10]. There is two types of stresses generate like tensile and compressive residual stress. Tensile stress is harmful to the component because fatigue failure and crack propagation take place. While the compressive residual stress is beneficial for the component.

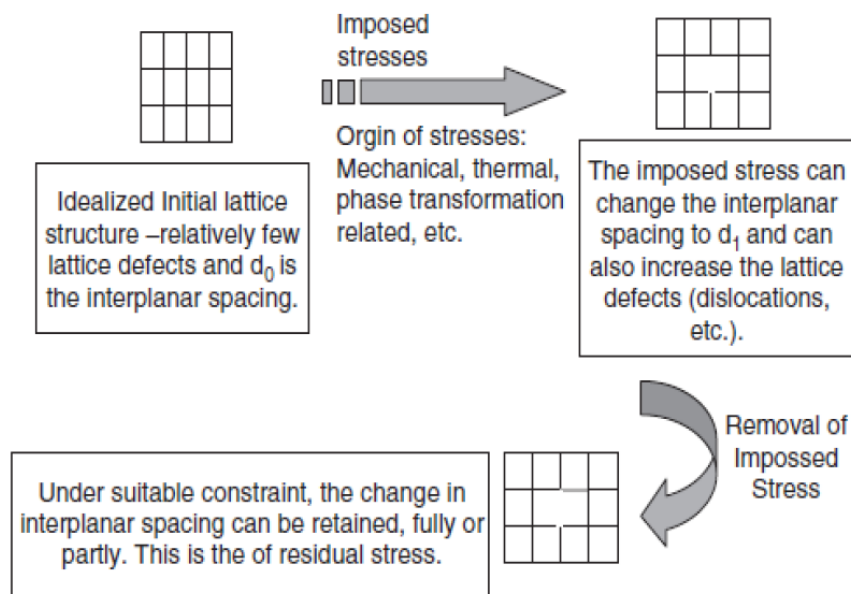


Figure 1.14 – Material imposed of stress due to which dislocation defect produced [10].

1.12 RESIDUAL STRESS MEASUREMENT: Last few years, there are many quantitate and qualitative methods are developed to measure the residual stress. There is two types of techniques used i.e. destructive and non-destructive.

1.12.1 Destructive Techniques:

(a) **Hole drilling:** A small hole is drilled 1-4 mm diameter and same height. Surface strain present in the material is measured by the three-element rosette.

(b) **Curvature and layer removal:** A simple geometry is used to measure the residual stress in the sample. In this method quick and simple calculation required to relate the curvature with residual stress. The stress present in the body is calculated with elastic properties and then the layer is removed after that the next layer stress is measured.

1.12.2 Non – Destructive Techniques: In this technique the residual stress is measured with the help of crystallographic and physical parameter relationship. There is no need for destruction of material.

X-Ray diffraction: When the x-ray fall on the measurement area then they should go back to the detector without hitting any obstruction. The only limitation of this technique is sample geometry as see figure 1.15.

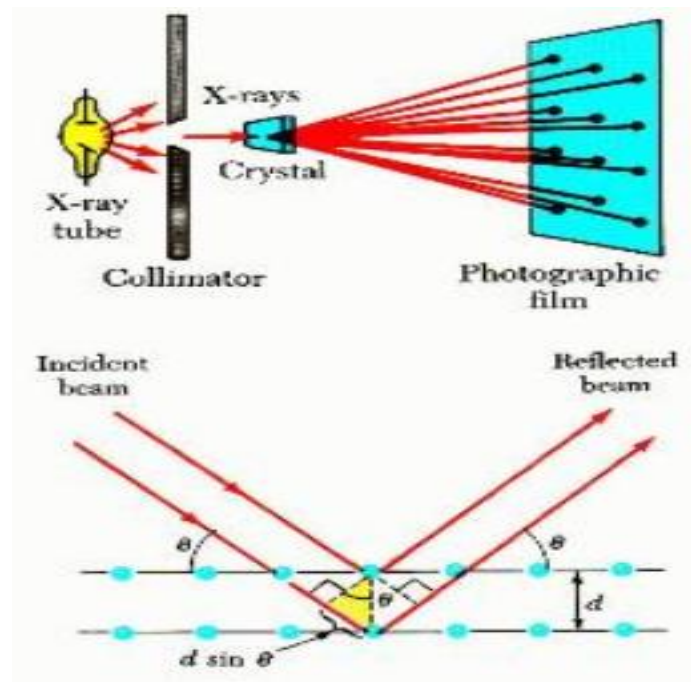


Figure 1.15 – X-ray diffraction [14].

Neutron diffraction: This technique depends on the elastic deformation in polycrystalline material in which spacing of lattice takes place. The large penetration of depth can take place in neutron diffraction as compared to the X-Ray diffraction. The measurement of residual stress can take place from 0.2 – 100 mm in Al. A complete 3-D map of residual stress can be taken from this [12].

1.13 MOTIVATION: Now-a-days, the main challenge in automotive, aerospace industry is to reduce the weight and get high strength of material. A lot of research has been carried out on the materials like Aluminium, Steel and Titanium. It is found that Ti is the only material which have high strength, low weight density ratio, high corrosion resistance and can withstand high temperature. Titanium is used in aerospace industry to manufacture wings, landing gears and fuselages etc. By the manufacturing of these components with various process like machining, forming and thermal loads there is tensile residual stress occurred which is undesirable for the components and cause fatigue failure which decrease the life of components. To avoid this material is hot rolled to get desirable deformation, reduce spring back effect and bring compressive stress. Since the stress has been gone through the thickness so that the research work is done to “Quantification of residual stress in relation with microstructural changes during hot rolling of titanium alloys.”

CHAPTER 2

LITERATURE SURVEY

2.1 GENERAL: In this chapter, the detail study of literature is done with regards to changes of microstructure, dynamical and mechanical properties of material during the hot rolling of titanium alloys.

2.2 Effect of hot rolling temperature on microstructure and dynamic mechanical properties of Ti-6Al-4V alloy

Wang Min et al. [15] use the relation between relative velocity (V_r), the rotational velocity of driver roll (n_1) and feed of idle roll (v) to make the motion parameter of rolls. The other method which was developed to compare the motion of rolls was 3-D FE model which is made in Abaqus/explicit. By using these relations results show that the relative velocity can be increased by the decreasing of the rotational velocity of roll or increasing the feed of idle roll which makes increase Δh_i . It is found that the feed of idle roll has a more bulging effect on the roll force as compared to the rotational velocity of driver roll. By increasing the rotational velocity make the end plane quality and there is a uniform distribution of strain and temperature in the deformed ring but there is an opposing effect on the roll force.

J. Kohler et al. [16] the study shows that the generation of residual stress takes place due to distortion produced in a large component in the aerospace industry. For the measurement of residual stress, an indirect method i.e layer removal method is used which is better than the X-ray diffraction.

Tripathi et al. [17] Have shown the fatigue behaviour of commercial pure titanium during cold rolling. In this study, the samples were cold rolled to a thickness reduction of 2.5, 5, 7.5 and 10%. The cold rolling significantly improved fatigue life of the material. It is observed that there is dual slope in the as received condition where as there is only single slope in the cold rolled material. Fatigue life of the material is considerably improved at low deformation

Yi Chen et al. [18] examined the changes of crystallographic texture in the β -Ti alloy (Ti-15Mo-3Al-2.7Nb-0.2Si) during the hot rolling process at 1023K. It is found that due to the shear strain fading of texture intensity on the surface area and making of key texture gradients through-thickness. It is observed that the dynamic recrystallization takes place in the hot rolling and the formation of its grains is analysed with the help of EBSD. From this analysis, it is observed that the weakening of texture is due to the rotation of grains. There is a gradual change

in the microstructure along the longitudinal direction and as the deformation increase (56% to 84%) the fibre microstructure becomes more important.

S.K Sahoo et al. [19] done a review on the reduction of commercially pure titanium of 50%, 70%, 80%, and 90% through-thickness along with unidirectional rolling by hot rolling. There were two types of twins were observes in the microstructure i.e tensile twins and compressive twins. When there is a 50% reduction of thickness in the sample and it is found that these twins were in abundance in the sample. There is also texture intensity increase when there is an increase of the degree of rolling deformation.

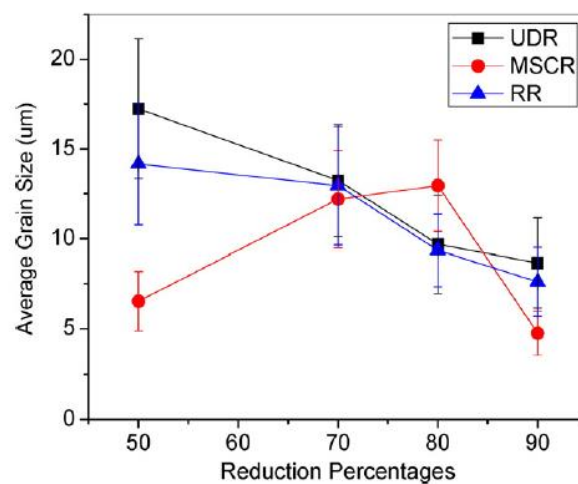


Figure 2.1 – Average grain size of CP titanium at different reduction.

Z. X. Du et al. [20] show the effect of hot rolling and heat treatment on the microstructure of Ti-3.5Al-5Mo-6V-3Cr-2Sn-0.5Fe which is a high strength β titanium alloy having excellent tensile properties. It is examined from microstructure and solution treatment of alloy in $\alpha + \beta$ rolled alloy have finer β grain size as compared to the all heat treatment conditions. By increasing the temperature the strength of alloy decrease and ductility increase or vice-versa. There is better ductility in $\alpha + \beta$ field with the help of rolling and solution treatment as compared to the rolling and solution at the β field.

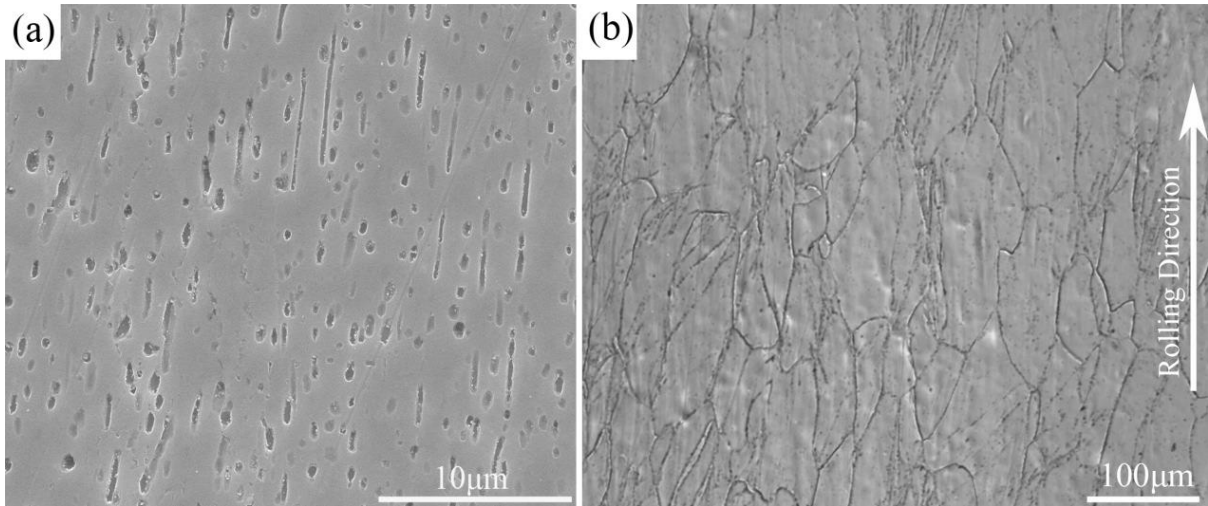


Figure 2.2 – Microstructure of the alloy rolling at 790 °C and 870 °C.

Luo Yumeng et al. [21] done a review on the changes of microstructure and dynamic mechanical properties of Ti-64 alloy by the hot rolling temperature. Since Ti-64 have high strength and low density due to which recrystallization and phase transformation occur when the rolling temperature is greater than the recrystallization temperature. To study the microstructural changes the polishing and etching are done after that plate was examined on the Normal surface (NS), Rolling Surface (RS), and Transverse Surface (TS) with the help of Scanning Electron Microscopy (SEM). The SEM results show that the elongation of the α equiaxed grains are more elongated along the rolling direction as compared to the normal direction when the rolling temperature reached 900°C. To study the texture measurement of Ti-64 alloy X-ray diffraction using Cu $K\alpha$ radiations and results shows that change of rolling temperature from 840°C to 930°C there is a variation of texture due to which there is a change of mechanical properties anisotropy.

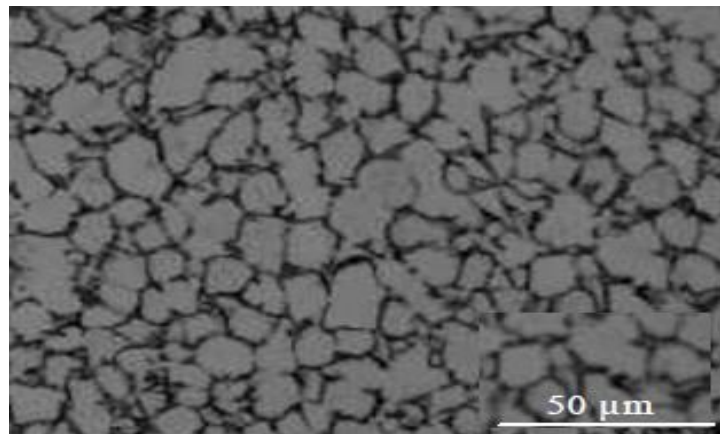


Figure 2.3 – Microstructure of Ti-6Al-4V before rolling.

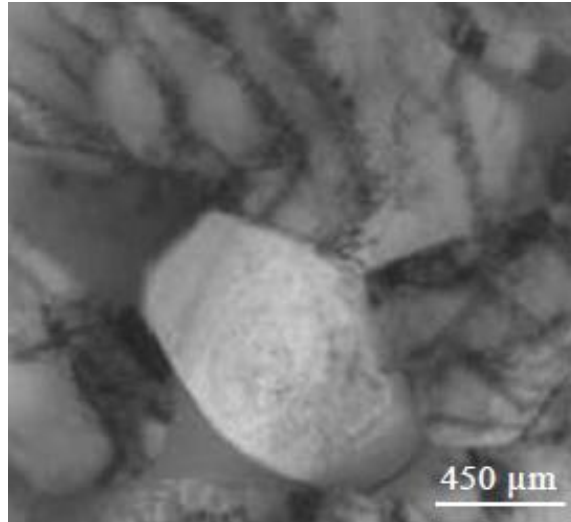


Figure 2.4 – low density dislocation after hot rolled.

Shubo Gao et al. [22] A ingot 760*380*900mm of High-Nb TiAl was fabricated by hot pack rolling method is used in which controlling the rolling temperature there are three microstructure obtained – Duplex(DP), near lamellar(NL) and fully lamellar(FL) by which different mechanical properties get. As compared to the heat treatment process, the hot rolling process is used to control the microstructure and mechanical properties. Results show that after hot rolling strength of high-Nb TiAl alloy sheet improves at elevated temperature and rolling temperature.

G.Y Deng et al. [23] done a review on the change of microstructure and stress-induced in HSS during the hot rolling. Observing the microstructure study shows that there is a void formation on the surface of the material due to spalling of carbide at grain boundaries which can act as the origination of cracks during further heating and cooling. The stable temperature is achieved is 630°C which is used as a reference temperature to investigate high temperature. There is large compressive stress is observed in the workpiece due to contact with hot-rolled strip but no tensile stress is observed because of low temperature. X-ray diffraction technique is used to analyze the residual stress in the workpiece.

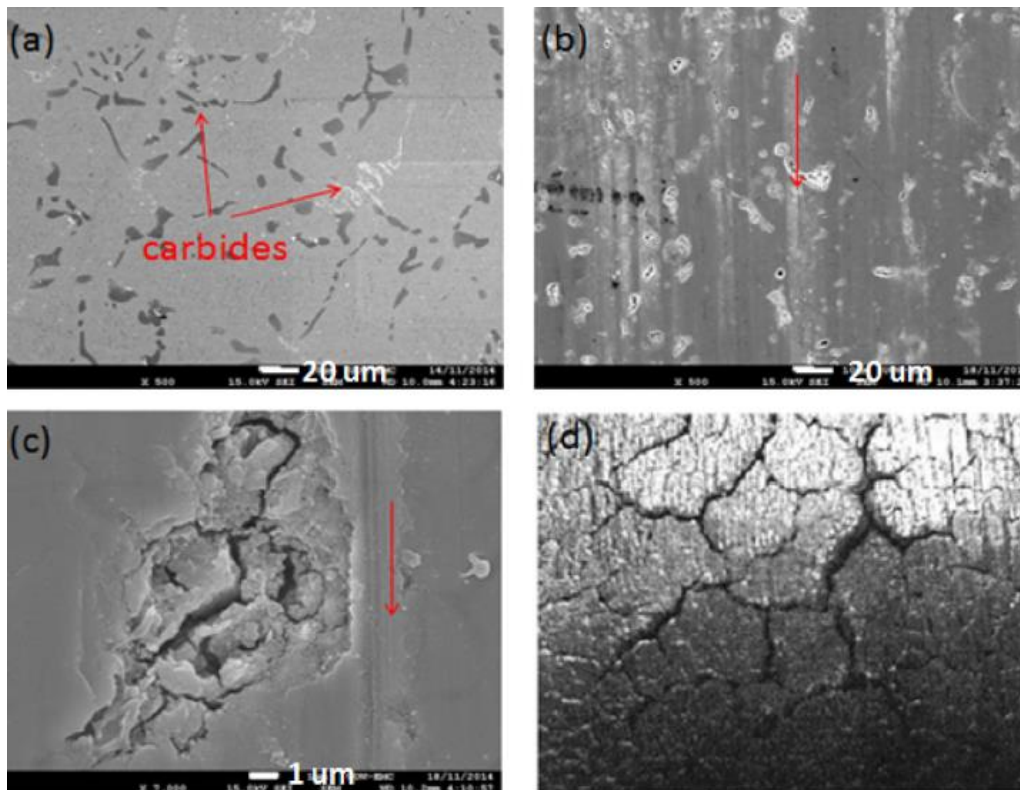


Figure 2.5 – Microstructure observation at HSS work roll surface (a) before hot rolling (b) after hot rolling (c) magnification of b (d) thermal crack pattern after hot rolling.

Jian Lan et al. [24] bearing race can also be manufactured by a cold rolling process with high precision and without removing material but there is the development of uneven residual stress. So this paper review on the control and measurement of residual stress which is measured by X-ray. The results show that the residual stress is present at the outer surface of bearing race while its magnitude starts decreasing along with the depth.

K.L Mothosi et al. [25] residual stress is produced during the manufacturing process. Compressive residual stress is beneficial while the tensile stress is harmful. In this study, roll speed was taken 10 rpm and the roll gap was taken of 20, 50 and 100 mm. X-ray diffraction is used to measure residual stress. The results show that the compressive residual stress was found in all specimen. The value of maximum principal stress was -55 MPa having 83% compaction density. As the density of specimen decrease than the value of residual stress decrease i.e less compressive residual stress.

Jie Zhao et al. [26] this paper review that with the help of dynamic recrystallization the microstructure of near- α was observed in the $\alpha + \beta$ phase. The microstructure was observed

at the temperature at 800°C. The Electron Beam Scattering Diffraction(EBSD) was used to analyze the deformed and undeformed stage of TA-15.

Qiong Wu et al. [27] done a review on the ring rolling. It is found that some material is removed during the manufacturing process of ring parts having less rigidity. The research object used is a 2219-Al alloy which is used in aerospace ring rolling. The finite element method is used to simulate the results.

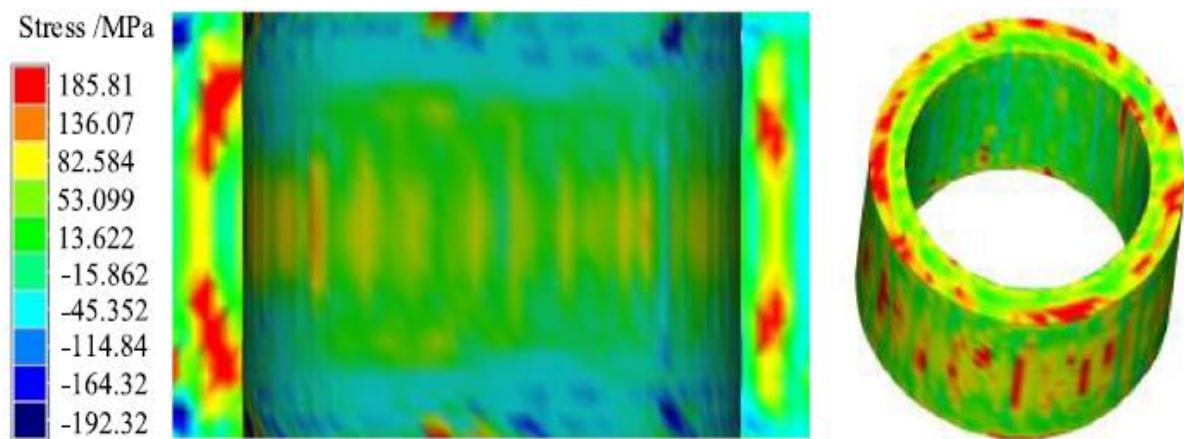


Figure 2.6 – Simulation results of residual stress in ring rolling.

Silvia Brunbauer et al. [28] done a review to find the residual stress, temperature and phase transformation of a low quenched seamless steel tube with the help of finite element method. In comparable condition there is discontinuous cooling effect is negligible in residual stress but the micro-hardness effect came in effect. From this review it is concluded that the residual stress distribution is plastically due to thermal misfits.

V. Velay et al. [29] done review on the superplastic forming process in which Ti-6Al-4V is used. The condition for the superplastic forming process in this alloy is high temperature ($> 900^{\circ}\text{C}$) and low strain rate ($\dot{\epsilon} < 10^{-3} \text{ s}^{-1}$) but for the industrial cost there must be some refinement so the microstructure refinement is needed so condition become (800 and 10^{-3}). the objective of this review the changes of microstructure evolution under the mechanical and thermal loading condition from different microstructure having equiaxed grain size $0.5 \mu\text{m}$ and $3 \mu\text{m}$ with $670 - 800^{\circ}\text{C}$ and strain rate $10^{-4} \text{ s}^{-1} - 10^{-2} \text{ s}^{-1}$. There is a strain hardening effect is also observed.

2.3 RESEARCH GAP: A lot of work has been done on the microstructure and mechanical properties of titanium alloy during the hot rolling process but no work has been reported on the study of change of microstructure and effect of residual stress during the hot rolling. So the present work is done on the “Quantification of residual stress in relation with the microstructural changes during the hot rolling of titanium alloy”. Following are the limitation observed during the literature review:

- Change in microstructure and variation of grain size at different reduction rate
- Residual stress distribution along the rolling direction and perpendicular to rolling direction

2.4 OBJECTIVE OF PRESENT WORK: After literature survey the objective of present work was as following:

- Hot rolling of Ti-6Al-4V alloy at different reduction rate
- Study of microstructure throughout the thickness
- Determining the residual stress throughout the thickness

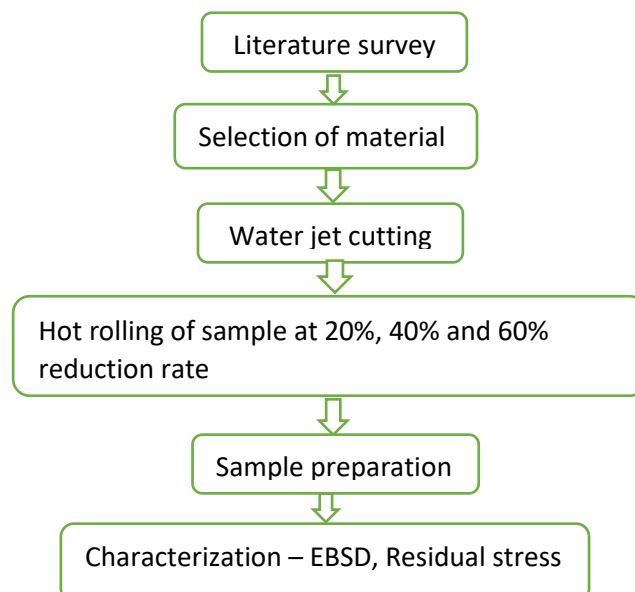
CHAPTER 3

DESIGN OF EXPERIMENT

3.1 INTRODUCTION: In this present work, the change in microstructure and residual stress are presented after the hot rolling for different plastic deformation of 20%, 40% and 60%. The microstructure has been examined across the - thickness (T/2, T/4, T/8 and T₀). T is the thickness of deformed sample. EBSD and XRD characterization techniques were used to characterize the material.

3.2 MATERIAL SELECTION: A rectangular bar (with dimension length 160mm, width 35mm and thickness 6mm) of Ti-6Al-4V alloy has been used to study the through-thickness residual stress development. The material was in fully-recrystallized state.

3.3 METHODOLOGY: The starting material were subjected to a plastic deformation of 20%, 40% and 60% in a laboratory rolling mill at a high temperature of 950°C. The rollers are made up of steel forged alloy having diameter of 360mm and rotating at a uniform rotational speed of 7 rpm. The specimen were then mechanically polished using a SiC paper with grit sizes of 200 μ m, 400 μ m, 600 μ m, 1000 μ m and 1500 μ m. The sample were subjected to electro-polishing using an electrolyte of methyl alcohol and perchloric acid (80:20 by weight) at 27V for 10 sec. The sample were then characterize using X-ray and Electron diffraction along the thickness at T/2(mid-thickness), T/4, T/8 and T₀ (at the top surface). The Quanta 3D-FEG is used for EBSD scans and analysed by OIM EBSD software package. An area of 100 X100 μ m² was used in EBSD with a step size of 0.1 μ m. The residual stresses were measured using the X-ray diffraction.



3.4 MACHINE AND EQUIPMENT:

3.4.1 Water jet cutting machine: Water jet cutting machine is used to cut 4 pieces of equal width of Ti-64 material along the rolling direction. See figure 3.1. Wide variety of tools are cut using a high pressure of water (30000 – 90000 psi) with the mixture of abrasive substance. The main advantage of the water jet cutting machine is that no heat is produced during the cutting process. Composite of tungsten carbide are used in the manufacturing of nozzles. Removal rate of material is effected by the distance between the nozzle and material which should be minimum 3.2 mm.



Figure 3.1 – Water jet cutting machine (focal point, Patiala).

3.4.2 Rolling mill: Two-high rolling mill is used for rolling of Ti-64. Before rolling, the material is placed in the furnace at 950°C for 30 min to achieve the recrystallization state. Then the material is passed through the rollers. The roller speed is 7 rpm and repetitive reduction option is used to achieve 20%, 40% and 60 % reduction.



Figure 3.2 – Rolling mill (I.I.T Bombay).

3.4.3 Grinding: Abrasive paper (Sic) is used for grinding the specimen. The grit size used of Sic is 200, 400, 600, 1000 and 1500. Scratch marks are removed using this paper along the rolling direction. Suitable pressure and speed are used to avoid deep scratch and overheating.



Figure 3.3- Abrasive paper

3.4.4 Polishing: To get the scratch-free and mirror finish polishing is done with the help of soft cloth (velvet). The medium used for polishing is alumina powder and diamond paste. The cloth is washed with water to remove the contaminants and specimen was held on the rotating disc.



Figure 3.4 – Polishing machine (I.I.T Bombay).

3.4.5 Electro-polishing: This is used to remove dust and scratch from the sample. The electrolyte used is methanol 80% and perchloric acid 20% at 27V, 4.5 A for 10 sec.



Figure 3.5 – Electro polishing machine (I.I.T Bombay).

3.5 ELECTRON BACK SCATTERED DIFFRACTION (EBSD): It is a technique in which primary accelerated electron of a beam of a scanning electron microscopy gets diffracted from the atomic layer of the crystal structure of a material. In this technique, a crystalline/polished specimen is placed in the SEM chamber at an angle of 70° to the horizontal. When the electron beam falls on the specimen and diffracted from its strike to the fluorescent screen which is equipped with a camera. The rays give the geometry of the lattice plane present in the crystal through which crystal structure and orientation of grains are obtained. In the present work, all the orientation of grains is obtained from EBSD which is done on QUNATA 3D FEG machine.

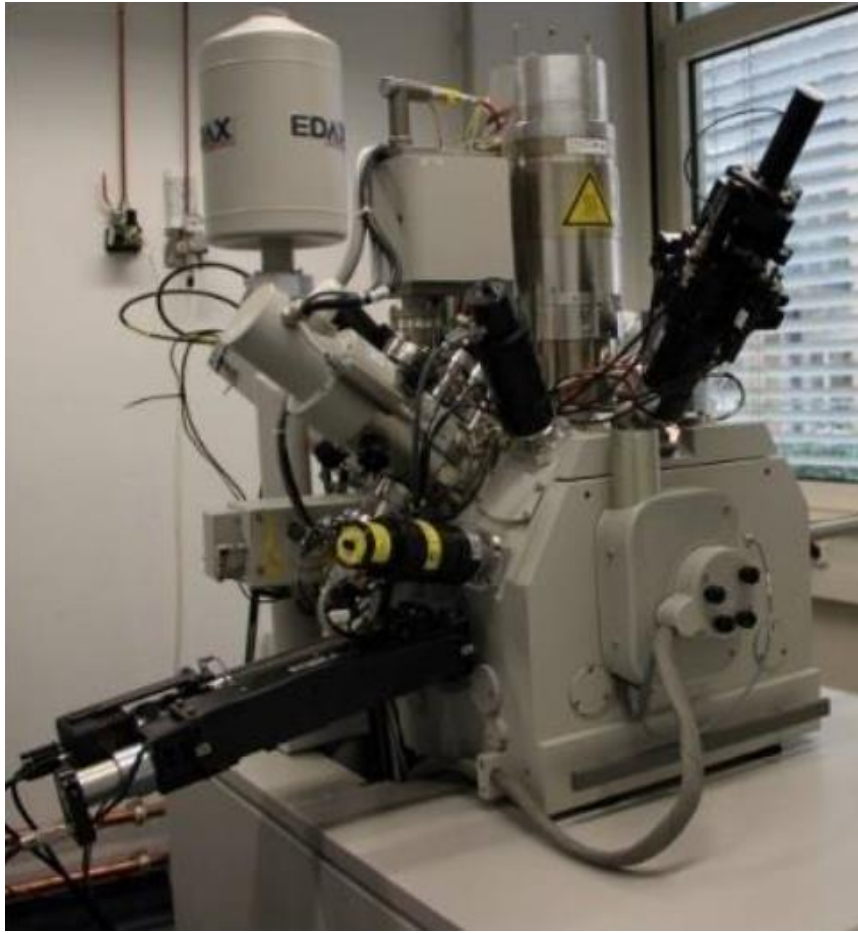


Figure 3.6 – EBSD machine (I.I.T Bombay).

3.6 X-RAY DIFFRACTION (XRD): It is a powerful technique which provides the information of micro strain, interplaner spacing and degree of orientation of crystalline phase etc. in this technique X-ray fall on the specimen and get diffracted to a different angle with different intensities. By measuring the angle and intensity we can measure the interplaner spacing and width of the diffracted beam tells about the micro strain. The Bruker X-ray is equipped with stepper motor with an optical encoder to give precise value.



Figure 3.7 – X-ray diffraction machine (I.I.T Bombay).

CHAPTER 4

RESULT AND DISCUSSION

4.1 INTRODUCTION: In this chapter, as received sheet of Ti-64 (grade 5) subjected to a laboratory hot rolling deformation 20%, 40% and 60% respectively. The rolled sheets were characterized across the thickness at different thickness location of T0 (top surface), T/8, T/4 and T/2 (mid-thickness) respectively, where T is the thickness of rolled sheet. This chapter also describe the various EBSD and residual stress (measured through XRD) analysis obtained for different percentage of deformation at different thickness location. The change in grain size and development of residual stress throughout the thickness of material is given below:

4.2 GRADIENTS IN MICROSTRUCTURE: EBSD technique was used to analyse the change in microstructure of the material. It has been observed that the as received material has bimodal microstructure having few small grains and coarser grains size (see figure 4.1). the average grain size at mid-thickness (T/2) of the sheet. After the hot rolling at various reduction rates the coarse grain start converting into finer grains and elongation of grains takes place. the top surface (T0) showed less change in grain structure compared to other thickness locations. As shown in figure 4.1 the coarse grains are present at the surface of as received material and at 20% reduction the coarse grain start converting into finer grains. At 40% reduction, the finer grains were observed at mid-thickness of material. At 60% reduction, the plastic flow of material was higher and finer grains were observed at all the thickness location of material. However, the mid-thickness showed more prominent changes in grain structure at higher percentage of deformation.

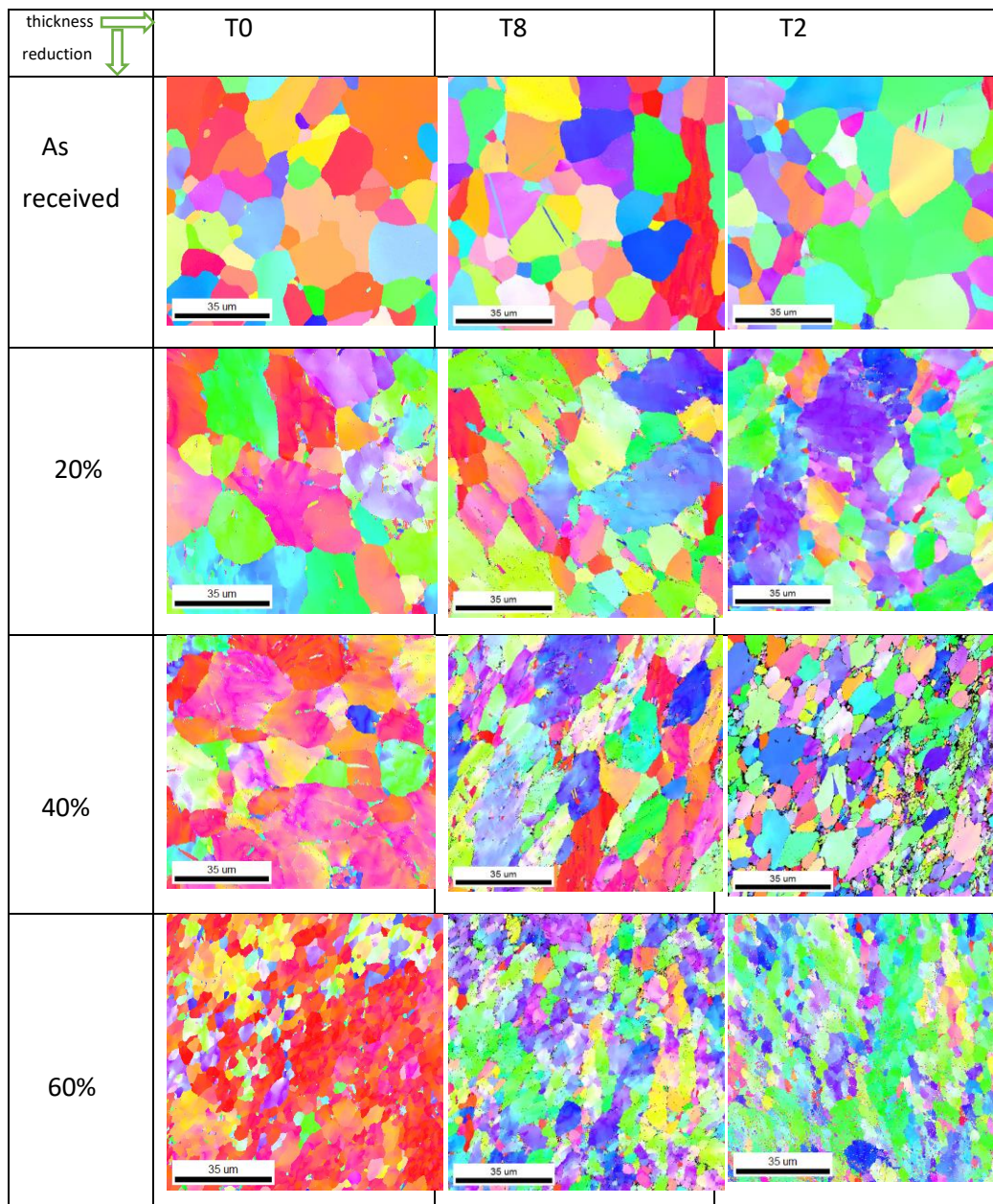
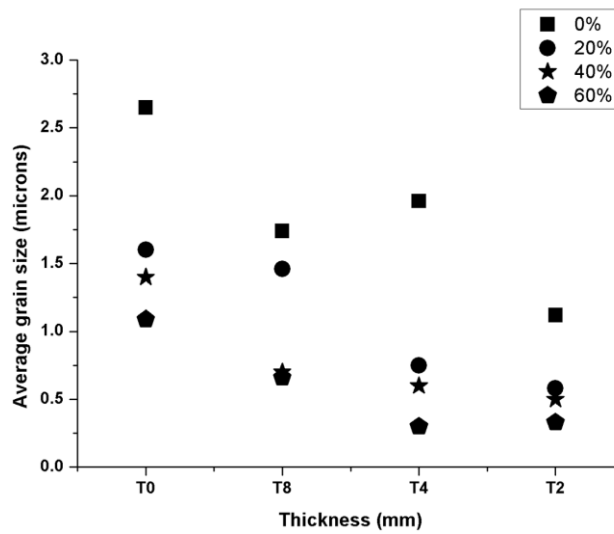


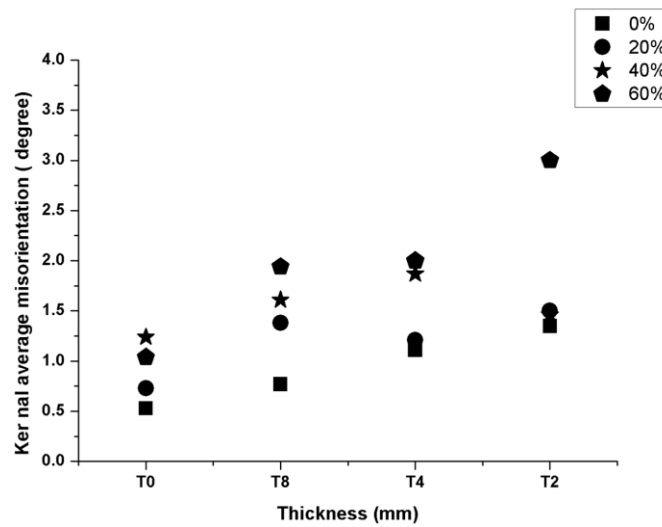
Figure 4.1 – Through thickness microstructure development during hot rolling.

From the analysis of microstructure the average grain size and Kernel average misorientation can also be analysed. When the hot rolling takes place then the variation in grain size takes place throughout the thickness of material. As seen in figure 4.2 (a) the grain size of the as received material was 2.60 microns on the surface (T0). After the hot rolling, the grain size decrease and gradients in grain structure at the surface of as received and 20% reduction was high and it decreases with deformation reduction on the surface of material. The grain size on

the surface of material at 60% reduction rate is 1.06 microns. The average kernel misorientation reflects an indirect parameter of plastic strain of the material.



(a)



(b)

Figure 4.2 EBSD data for different thickness location T0, T8, T4 and T2 (a) average grain size and (b) Kernel average misorientation.

4.3 GRADIENTS IN RESIDUAL STRESS: X-ray diffraction technique was used to measure the residual stress developed during the hot rolling of Ti-64 (grade 5). Bruker D-8 Discover™ machine was used for the measurements. The as received material has almost negligible stress gradient. When the material passed through the rollers a compressive residual

stress was developed throughout the thickness of material. It was observed that both the normal (σ_{11}) and shear stress (τ_{13}) varying throughout the thickness. It is to be noted that a tensile residual stress of magnitude 10MPa was present in the surface of as received material but throughout the thickness it become compressive at T/4 and after the tensile at 4 MPa T/2. However the variation in the values is negligible compared to the variation in the values for different percentage of deformation. Hot rolling of the material was done at different reduction rates of 20%, 40% and 60% respectively. It was observed that a highly non-uniform compressive residual stress was present throughout the thickness. The compressive residual stress along the rolling direction throughout the thickness was -30 MPa at the surface and -150 MPa at T/2 of 20% reduction. A highly compressive residual stress was also observed for 60% reduction which vary from -150 MPa at surface to -250 MPa at the mid-thickness of material (see in figure 4.3)

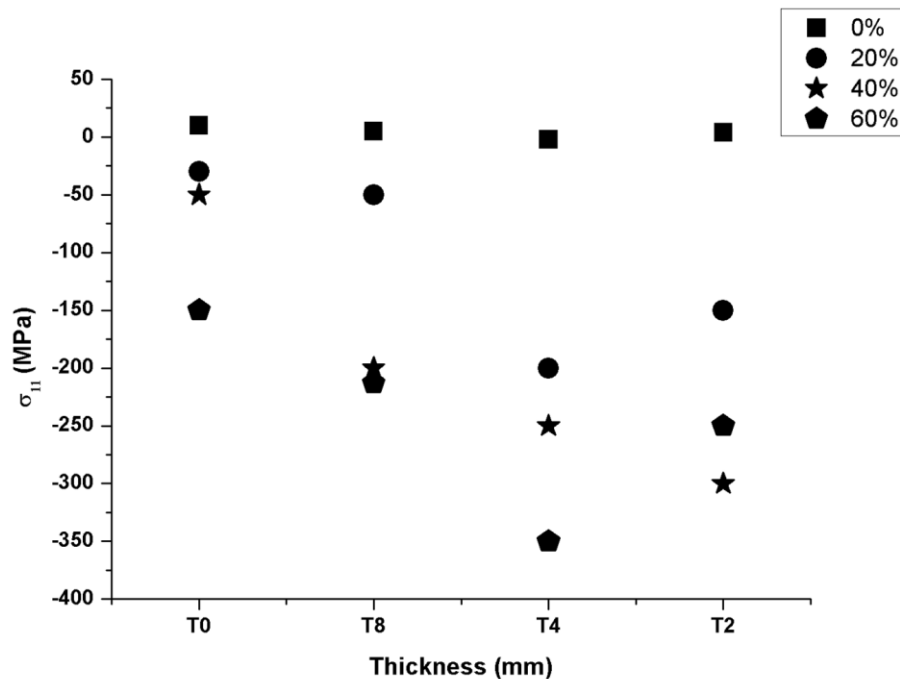


Figure 4.3 – Residual stress (σ_{11}) signatures as function of thickness

Similarly, the compressive residual stress perpendicular to the rolling direction was also measured which is denoted as σ_{22} . The compressive residual stress vary from -250 MPa at the surface of 20% reduction rate and it goes tensile residual stress at 70 MPa at the mid-thickness of material at 60% reduction as shown in figure 4.4. It is also to be noted that the trend in stress gradient for rolling direction and its perpendicular direction is opposite.

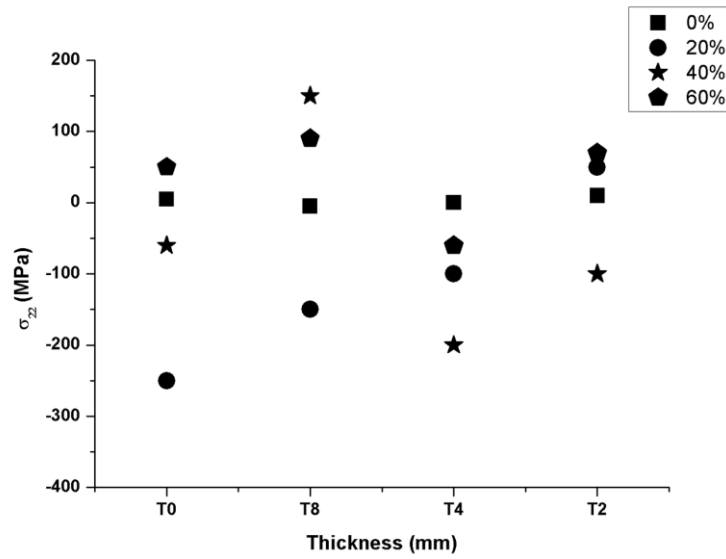


Figure 4.4 – Residual stress (σ_{zz}) signatures as function of thickness

Shear stress of the material was analysed from surface of the material to mid-thickness of material. Shear stress of the as received material vary form 2 MPa at surface to 10 MPa to mid-thickness. While at different reduction rate the shear stress vary form 30 MPa at surface of 20% reduction rate to -15 MPa at the mid-thickness of material at 60% reduction rate as shown in figure 4.5.

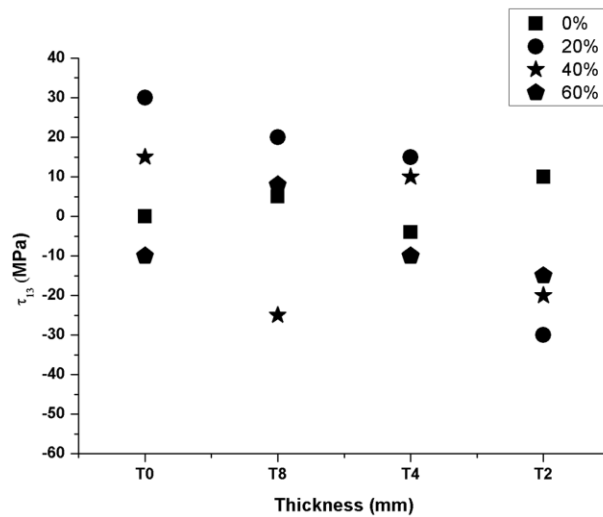


Figure 4.5 – Residual stress (τ_{13}) signatures as function of thickness

CHAPTER 5

CONCLUSION AND FUTURE SCOPE

5.1 CONCLUSION: In this chapter, the results obtained by doing the hot rolling of a fully recrystallized titanium sheet (6mm thickness) with different reduction rate (20%, 40% and 60%) are concluded by doing the experimental measurements in terms of microstructure evolution and residual stress development across the of sheet. Following were the main observations:

- Evolution of microstructure - from coarse grains to finer grains throughout the thickness with different reduction rate.
- A highly compressive residual stress along the rolling direction (σ_{11}) and normal to rolling direction (σ_{22}).
- Shear stress was also analysed during the hot rolling.
- A clear stress gradient was established in this study.

5.2 FUTURE SCOPE:

- A microstructural origin of residual stress
- Correlation between texture development and residual stress distribution
- Numerical study of stress gradient during hot rolling of titanium alloy using anisotropic yielding
- Effect of different rolling process parameter on the through thickness residual stress gradients

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