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Quantitative analysis of stress residuals in conjunction with microstructural modifications in hot rolled titanium alloys

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ABSTRACT

Hot rolling was performed on a completely recrystallized Ti-6Al-4V and 22%, 44%, and 66% reduction rates was used. For further characterization, the samples were prepared using two types of polishing i.e., mechanical and electro-polishing. A slope in microstructure of Ti-6Al-4V was observed, and this slope is highlighted due to development of residual stress across the thickness of sheet while rolling took place. The EBSD and X-ray diffraction were used, for investigation of above-mentioned problems. It has been noted that the material's as-received grain structure is biomedically compatible. Along with an increase in the percentage of deformation, a grain refinement has been seen across the thickness of the rolled sheet. At half of thickness and 66% distortion, finer granules were seen. From the top surface to the middle thickness of the rolled sheet, the stress gradient in the rolling direction rises. The stress gradient is decreased in the lateral direction.

Keywords: Titanium Alloy, Residual-Stress, Permanent Deformation, Microstructure, Hot Rolling, Electron Backscatter Diffraction (EBSD), X-Ray Diffraction (XRD).

INTRODUCTION

Through a process called plastic deformation, a material is transformed into product of desired shape and size as per our requirement [1, 2]. A substance may be subjected to shear/compressive/tensile, or a combination of any of these three. For yielding to occur, force must be in such a manner that it exceeds the yield strength while being below its fracture strength in order to cause plastic deformation. Since better quality and dimensional accuracy of product can be easily attained without any material reduction in volume. And forming process is cost-effective [Amitabh Ghosh and Ashok Kumar Malik, 2010]. In the past, the items were formed using the forming process, and they were machined to finish level using the machining process. However, today's tools and dies are composed of strong materials, allowing for the successful production of products with high levels of dimensional precision and surface excellency so that efficient reduction of machining time can be obtained.

Rolling operation: The metal is repeatedly inserted between rollers at the mill's entrance with gaps which is smaller than the cross-sectional area of the metal to get required form. As space between the rollers narrows and as the number of passes rises, deformation of output metal increases the rollers' gap determines how much material is reduced. In the aerospace industry, this procedure is primarily utilized to make fan blades, landing gear, and railway track. For rolling soft metals like silver, gold, and other such materials throughout the 14th century, goldsmiths employed hand-driven rolls [Siddhartha Ray, 2016].

Rolling is one of the most popular forming processes out of many previously discovered other manufacturing processes so far. Most of ferrous and non-ferrous alloys can be rolled. Metal may be rolled into sheets, strips, and plates, which are all practical shapes. Additionally, it may be used to create bar, angle, and railway tracks.In doing so, stress of compressive type is applied to distort the molecules of the material so that plastic deformation can be obtained. Flat roller or groove type roller can be used for such operation [Siddhartha Ray, 2016].

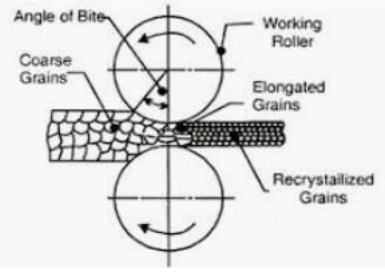

Figure 1: Reducing the cross-sectional are using rolling operation

Categorization of rolling operation

Following conditions can be utilized to in rolling operation:
(1) The longitudinal and transverse rolling based on directions of rolling
(2) Material`s temperature decides selection of process; either cold rolling or hot rolling
(3) Product shape: rolling, hollowing, and flat.

Hot rolling:

The material is rolled at a temperature greater than the temperature at which it would recrystallize. Ingots produced are used as raw material utilized in the rolling process where these ingots are made from molten metal. These ingots are formed into bloom, which is subsequently transformed into billet. Iron bars, railway tracks, and section (C/L/square) are formed from bloom using hot rolling operation. Smaller size products can also be made. [Siddhartha Ray, 2016].

Benefits of hot rolling:

(1) As a material's strength declines, less compressive stress is needed while rolling, leading to the use of rolling stands with low capacities that results in less size of rolling machine.
(2) There is significant plastic deformation without any effect of strain hardening.
(3) Low cost and less energy use.
(4) Hot rolling improves the efficiency by getting more deformation of metals and alloys.

Hot rolling's drawbacks:

(1) The mechanical properties and microscopic structure of the material are not uniform during hot rolling.
(2) The material's surface develops oxides [Siddhartha Ray, 2016].

LITERATURE REVIEW

The motion parameter of rolls was created by **[Verlindenet al., 2007]** using the relationship among comparative velocity, roll`s turning velocity, and feed. The findings of employing these relationships demonstrate that the relative velocity may be raised by either lowering the roll's rotational speed or raising the feed of its idle roll, which raises change in hi. When the rotational velocity of the driver roll was compared, it was discovered that bulging effect was high due high feed that resulted in high roll force. Strain distribution was uniform and warmness in the plastically deformed ring are improved by increasing rotational velocity, but the roll force is adversely affected.

According to [**J. Kohler et al., 2016],** distortion caused by a sizable component in the aerospace sector causes residual stress to be generated. In indirect approach of layer removal method, is utilized for residual stress assessment and is superior to X-ray diffraction.

Pure Ti was cold rolled and its fatigue effect was shown by [**Tripathi et al., 2013].** The samples in this investigation were rolled below recrystallization temp. to reduce cross section of samples. Material's fatigue life was greatly extended by the cold rolling. It has been noted that the cold-rolled material has a single slope, but as in received state, it has a dual slope. At low deformation, the material's fatigue life is significantly increased.

[**Chen et.al.,2015]** investigated the changes in topography of crystal during the hot rolling operation in Ti alloy. It was discovered that shear strain causes important texture gradients to form through thickness and a faded topography was seen on the surface area. It was highlighted that changing re-crystallization occurred, and EBSD was used to examine how the grains are formed. This investigation shows that the rotation of the grains is what causes the texture to degrade. Change happens gradually.

The variations in Ti-64 alloy's microstructure and dynamic mechanical characteristics caused by the high rolling temperature have been reviewed by [**Luo Yumeng et al., 2018].** Recrystallization and phase alteration take place due to temperature higher than the re-crystallization temp. Ti-64 has a high strength and low density. Polishing and etching on the plate were carried out to analyze the microstructural changes using scanning electron microscopy (SEM) on the surface before rolling and after rolling. According to SEM findings, when the rolling temperature reached 890°C, the extension of the grains was greater in the direction of rolling compared to normal direction. Cu K radiations were used to analyse the topography of Ti-64 alloy, and the findings demonstrate that when temperature changes from 800°C to 900°C, fluctuation in topography of rolled part occurs that results in change in mechanical property.

Shubo Gao etal., 2108] Hot pack rolling was utilized to create an ingot of Nb rich Ti-Al alloy whose dimensions was as 760*380*900mm. By adjusting the rolling temperature, three microstructures were created and each of them had a distinct mechanical property. Micro-patterns and mechanical characteristics were controlled in hot rolling operation as opposed to the heat treatment technique. Results indicate that high-Nb Ti-Al alloy sheet's strength increases after-hot-rolling at higher temperature and rolling temperature.

A review of the changes in microstructure and stress on the hot rolling of HSS was completed.**[G.Y. Deng et al., 2107].** By observing the microstructure research, it reveals that carbide grain particles shrinks at grain boundaries due to which voids get developed, that lead to obtain starting point for fractures during additional heating and cooling. 600°C is a reference temperature, it is the stable temperature that may be attained. Because of contact with the hot-rolled strip, a significant amount of compressive stress is seen in the workpiece, but because of the low temperature, no tensile stress is seen. The residual stress in the workpiece was analyzed using the X-ray diffraction method.

Lan conducted a study where they found that cold rolling method can also be employed to manufacture bearing race at great accuracy. Although residual stress would build unevenly. Therefore, this study reviews the measurement and regulation of residual stress as determined by X-Ray. Findings indicate that outer surface of bearing race contain residual stress, where it gradually decreases in magnitude with depth.**[Jian Lan et.al., 2017]**

Residual stress is created throughout the production process, according to [**K. L Mothosi etal., 2017].** Tensile type stress is bad, while compressive residual stress is advantageous. In this investigation, Remaining stress was measured using X-ray diffraction. The findings demonstrate that all specimens included compressive residual stress. Maximum primary stress was -55 MPa with compaction density of 83%. Less compressive residual stress results from a reduction in residual stress value as a function of specimen density.

In this study, **Jie Zhao et al. [26]** discuss how dynamic recrystallization allowed for the observation of the near -alpha microstructure in the near alpha + beta phase. Microstructure was analyzed at 800 °C temperature. The distorted and Non-distorted stage were both subjected to an analysis using the Electron Beam Scattering Diffraction (EBSD).

The ring rolling has been reviewed by [**Qiong Wu et al. 2019].** It has been discovered that some material is eliminated during the production of ring sections with lower stiffness. The item in question is a Aluminum alloy used for aircraft ring made by rolling operation. Outcomes were simulated using the finite element approach.

Review of the Ti-6Al-4V-using superplastic forming method by **[V. Velay et al. 2016].** Experiment was conducted at high temperature (> 920°C) and low strain rate (203 s1) [29]. Manufacturing by rolling operation of any sample alloy was done at elevated temperature and minimum strain rate, The goal of this study is to examine how various microstructures with equiaxed grain sizes of 0.55 m and 3.2 m evolve under mechanical and thermal loading conditions at temperatures between 680 and 820 °C and strain rates between 105 s1 and 104 s1. Additionally, a hardening under strain effect was seen.

Research gap & purpose:

Research gap

Although much research on hot rolling process was conducted by many researchers, scholars, scientist and engineers on the micro-arrangement of grains & mechanical characteristics of Ti and its alloys but as per my knowledge no one has yet did the investigation on changes in micro arrangement of grains and the effect of residual stress. Therefore, current work is conducted on this research gap.

Purpose of the current investigation:

Following a review of the literature, the following goals were made during our current work:

(1) Studying of microstructure throughout the thickness,
(2) Reduction of cross section at different rate of Material Ti-6Al-4V
(3) Residual stress quantification at different level of thickness

Material:

Titanium:

Due to its variety of mechanical and physical properties, iron was once thought of as the most valuable substance on Earth. In their hunt for an alternative material, the researchers discovered titanium (Ti), which has significant advantages over iron (Fe) in many technical applications. In comparison to Fe, it is stronger, lighter, and more corrosive resistant.**[Yassin Mustafa Ahmed et al., 2014]**

Mineralogist Reverend made the discovery of a sand-like, black substance in 1791. According to his calculations, the material is made from magnetite and a substance that is like brown colour powder of some material, when dissolved in acid, produces a yellow solution. He gave it the name Menachanite because Marlin Meinrich discovered a few other elements in 1793 and he named it as Ti. "Titanium" is also a reference to the God of Greek. The earth's crust contains titanium in the forms of rutile and ilmenite oxide.Titanium alloys and pure Ti have a vast variety of usage in the space and aircraft sector, automobile sector and medical sector due to its special features of high strength to weight ratio. There are other titanium grades, but Ti- 6Al-4V alloy is preferred due to its excellent mechanical properties and good machinability and good resistance to corrosion. Aerospace and the automobile industries employ it because of its weight reduction ratio.Among Ti-6Al-4V (around 50% of all Ti-alloys) is employed in automobile and aircraft industries.**[Yassin Mustafa Ahmed et al., 2014]**

Table 1.Lists of some Ti-alloys in the Automobile sector, Aircraft & Medical [C. Veig1et al., 2012]

Alloys	Application Area
Ti-6Al-4V	Connecting rods, armour, and suspension springs
Grade-2	Exhaust System
γ (TiAl)	Turbocharger rotors
CP-Ti	Mechanism of control-system
CP-Ti	Mechanism of control-system
TI-6Al-4V	Docking gear
Ti-811	Propeller blades of engines
TI-6Al-4V, TI-6Al-7Nb	Prosthesis Implants

Crystal arrangement:

Allotropic conversion is the process of changing arrangement of one crystal into another, and operating temperature is known as the alteration temperature. Titanium has many crystal forms at various temperatures, just like other metals.

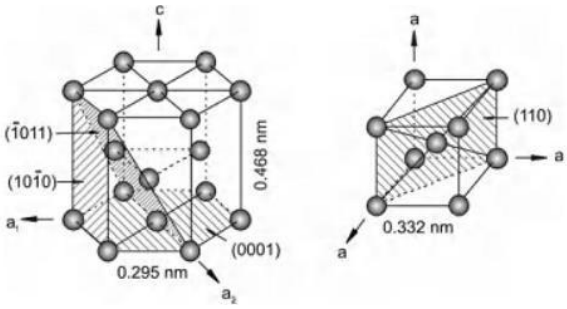

Figure 2 Crystal arrangement of $\alpha - Ti \& \beta - Ti$ [12]

Crystal arrangement of pure titanium and titanium alloys is HCP [7]. According to fig.2, the allotropic transformation is a BCC crystal structure, and the transition temperature is 882 2 °C. Ti has a young's modulus of 145 GPa. By adding or mixing other many elements into pure Ti, mechanical properties can be altered drastically
(1) Mixing of Aluminum stabilizes the alpha phase of Ti. So it can operate at higher temperature.
(2) Effect of Ti and Zr have minor effect on the alteration temperature but it definitely help in obtaining high strength of α – phase **[Yassin Mustafa Ahmed et al., 2014]**.

Selection of materials:

To analyse the through-thickness residual stress development, a rectangular bar made of the Ti-6Al-4V alloy (diameter: 35 mm, width: 160 mm, thickness: 6 mm) was employed. The substance had finished recrystallizing.

Residual stress:

Without any external loading, stresses that remain in the body after manufacturing or material processing are known as residual stress. Thermal gradients,uneven plastic deformation of the material, and other factors can also result in the creation of residual stress**. [Verlinden et al., 2007]**

Source of residual stress:
The two factors that cause residual stress are **(1)** mismatch (difference in interplanar spacing) and **(2)** limitations (prevent misfits from being retained). In comparison to microscopic stresses, macro stressors have a larger dimension of mismatches and limitations. The component's many portions have varied macro-stress locations. Chemically, thermally, and plastically are all used to achieve both stages. Tensile and compressive residual stress are two different types of stresses that are produced. As a result of fatigue failure and fracture propagation, tensile stress is bad for the component. While the component benefits from the compressive residual stress.**. [Verlinden et al., 2007**

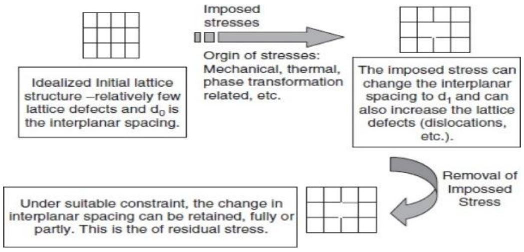

Fig.3: Imperfection due to dislocation.

Measurement of residual stress:
Numerous quantitative and qualitative approaches have been developed in past to assess residual stress. Both destructive and non-destructive procedures are employed.

Inspirational factor:
Obtaining lightweight along with high-strength materials is now biggest issue faced by the automobile and aerospace industries. Numerous studies have been conducted on materials including titanium, steel, and aluminium. Ti is the only material that has been proven to have great strength-to-weight ratio, good corrosion resistance, and the ability to sustain high temperatures without any deformation. In the aircraft sector, titanium is utilized to make things like wings, landing gear, and fuselages. Fatigue failure occurs due to residual-stress of tensile type that is bad for parts which results in shortens their lifespan.

METHODOLOGY

Initial material was exposed to 9600C when deformed plastically to the level of deformation of 22%, 44%, and 66% while rolling. The rollers have a diameter of 370 mm, are composed of steel forged alloy, while roller revolve at constant revolution of 7 rpm. Following that, Hand polishing was done using emery paper of grit sizes of 200, 400, 600, 1000, and 1500 respectively. Higher the number of grit size, finer the surface we get. The sample was electropolished for 10 seconds using an electrolyte and an acid by 85:15 ratio (by weight). The OIM EBSD software suite analyses EBSD images using the Quanta 3D- FEG. In EBSD, a 100 X 100 m2 area with 0.1 m steps was employed. The XRD was utilized for calculating the residual stresses.

Machines & equipment
Hydrodynamic Machining Using Jet Water Cutting Machine:
Ti-64 material is cut into 4 equal-width pieces using a water jet cutting machine in the rolling direction. Look at figure. Abrasive mixture and high-pressure water (30k–90k psi) are used to cut a wide range of tools. The biggest benefit of using a water jet cutting equipment is that there is no heat generated when cutting. Nozzles are made by combining tungsten carbide and other materials. The minimum 3.2 mm gap between the nozzle and the substance has an impact on the pace of material removal.

Fig.4: Hydrodynamic Machining Using Jet Water Cutting Machine(R.I.M.T., BAREILLY)

Compaction of billet using Rolling mill:

Ti-64 is rolled in a two-high rolling mill. The material is heated to 960°C for 35 minutes in the furnace before rolling to obtain the recrystallization condition. The material is then run over the rollers. Repetitive reduction is utilised to accomplish reductions of 22%, 44%, and 66% at a roller speed of 7 rpm.

Fig. 5: Compaction of billet using Rolling mill(R.I.M.T., BAREILLY)

Grinding:

The specimen is ground using abrasive paper (Sic). Sic is used with grit sizes of 200, 400, 600, 1000, and 1500. With the help of this paper, scratches may be erased in a rolling motion. To prevent severe scratches and overheating, the proper pressure and speed are utilized

Fig. 6: Abrasive paper [R.I.M.T., BAREILLY]

Mirror like Polishing using velvet:

Using a soft cloth (velvet), sanding is carried out to achieve a scratch-free and mirror finish. Diamond paste and alumina powder are the polishing mediums. The specimen was held on the revolving disc while the cloth was cleaned with water to eliminate impurities.

Fig.7: Mirror like Polishing using velvet (R.I.M.T., BAREILLY)

Electrically controlled polishing:

It is utilized to make sample clean from dust and scratches. At 29V, 4.3 A, for 18 seconds, an electrolyte of 82% methanol and 22% perchloric acid is utilized

Fig. 8: Electrically controlled polishing on specimen(R.I.M.T., BAREILLY)

EBSD:

In Scanning electron microscope, a beam of highly accelerated electron gets diffracted crystal arrangement of atoms` layer. In this method, a polished test subject is positioned in the compartment of S.E.M. machine at a 78-degree angle from plane. When a beam of electron strikes on test subject, it diffracts from the sample and lands on a fluorescent screen that has a camera. Crystal`s arrangement determines grain orientation and its placement, is provided by the rays. All of the grain orientation used in this study was determined using EBSD on a 3-D F.E.G. apparatus.

Fig. 9: Electron Back Scattered Diffraction machine (I.I.T., Roorkee)

X-RAY DIFFRACTION (XRD):

Powerful technology like XRD itself offers data on micro strain, interplanar spacing, degree of phase orientation of crystals, etc. In this method, X-rays strike the material and are diffracted to various angles and intensities. The interplanar spacing and beam width may be determined by measuring the angle and intensity, which provides information on the micro strain. To provide accurate value, X-ray has a stepper motor and an optical encoder.

Fig. 10: XRD machine (I.I.T., Roorkee)

FINDINGS AND DISCUSSION

Ti-64 sheet of 5th grade type alloy was hot-rolled in a lab and deformed by 22%, 44%, and 66%, respectively. The thickness of the rolled sheets (T) was measured at top surface, one fourth of thickness one third of thickness, and at half thickness correspondingly. Various percentages of deformation and various thickness locations are two parameters which are used to determine residual stress using XRD machine. Undermentioned tabular format of figures displays how the grain size and residual stress changed as the material's thickness decreased

Gradients in microstructure:

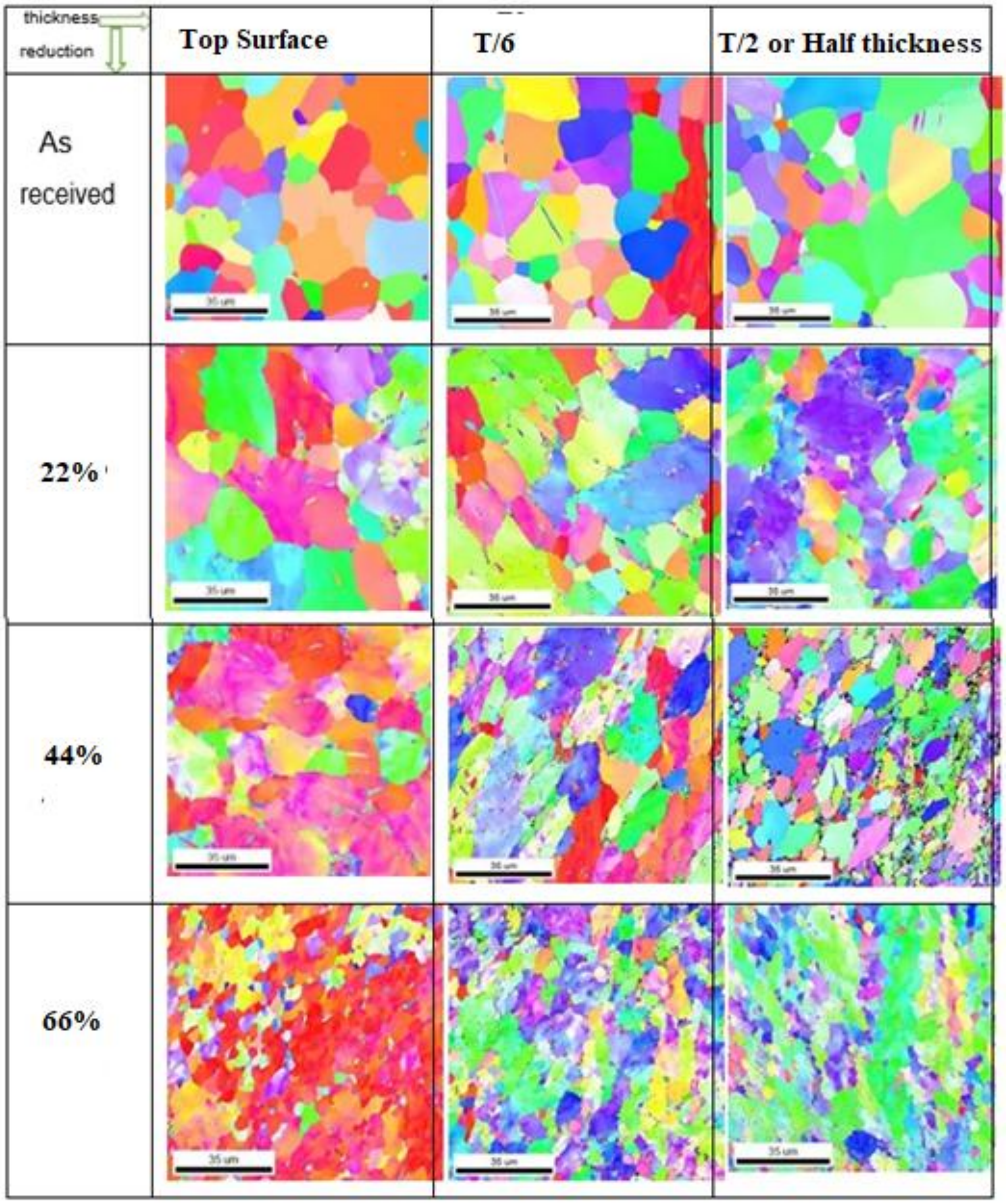

Figure 11: Through thickness microstructure development during hot rolling

The variation in the crystal`s micro-arrangement was examined using the EBSD technique. The material as received has been found to have a biomedical compatible microstructure with a limited number of tiny grains and larger grains at the sheet's mid-thickness (T/2) as demonstrated in fig. Following hot rolling at different reduction rates, coarse grains start to elongate and transform into finer grains. Compared to other thickness sites, the top surface exhibited minor variation in grain arrangement. The surface of the material as received has coarse grains, which start to change into finer grains with a 22% reduction, as shown in figureabove. At 44% reduction, the finer grains were visible close to the material thickness's midway. As permanent deformation of material increases grains of very small size can be visualized across the thickness distribution of the material at a 66% reduction. While mid-thickness displays more dramatic alteration in arrangement of grains, it may be due increment in percentage of permanent deformation. The EBSD method was used to investigate how the material's microstructure changed. And It was discovered that the material had a bimodal microstructure, consisting of both a larger and smaller particles as displayed in abovementioned figure.

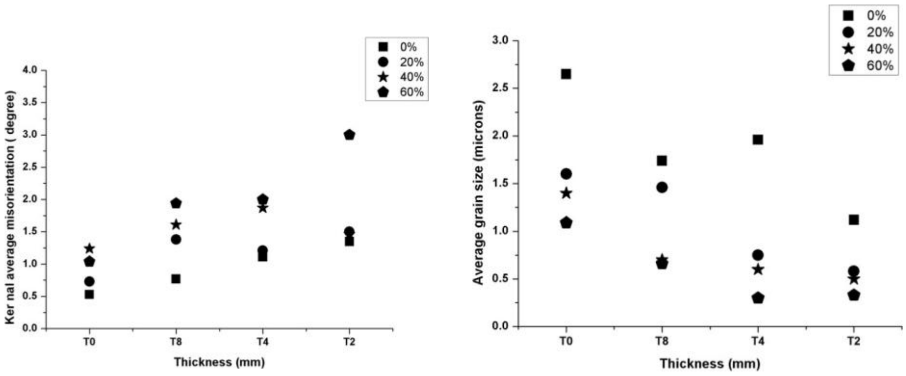

Figure 12 Grain Size and orientation graph at different thickness location

The average grain size and average misorientation of the kernels may also be determined from the examination of micro-structure. The fluctuation in grain size occurs across the thickness of the material during hot rolling. Material`s particle size was 2.90 microns at top surface, as displayed in figure.Following hot rolling, there was a considerable 20% reduction in grain size and grain structure gradients at the surface of the material, and this reduces when surface deformation is reduced. The granularity at a 66% reduction rate, the surface of the material is 1.26 microns. The average kernel misorientation serves as a proxy for the material's plastic strain.

Slopes in residual stress:

The residual stress that was created during the hot rolling of Ti-64 was measured using the X-ray diffraction method. “Machine model name Discover” was employed for the measurements. Material's stress gradient is essentially non-existent as received. A compressive residual tension was created across the thickness of the material as it was moved through the rollers. Both the normal as well as the shear stress were seen to change over the thickness.

It should be noted that the surface of the material as received had a residual tensile stress of magnitude 12 MPa, but as the thickness changes to T/3 and following the tensile becomes 5 MPa at T/2. But the difference in the numbers is insignificant when compared to the difference in the readings for various percentages of deformation. Different reduction rates of 22%, 44%, and 66% were used when hot rolling the material.

It was found that at top surface, it had a significantly non-uniform compressive residual stress. and at T/2 of 22% reduction, the residual stress of compression type was found in the orientation of rolling and their values are -32 MPa and -155 MPa, respectively. For 66% reduction, a very compressive residual stress was also noted, ranging from -156 MPa at the material's surface to -254 MPa at its midpoint in thickness.

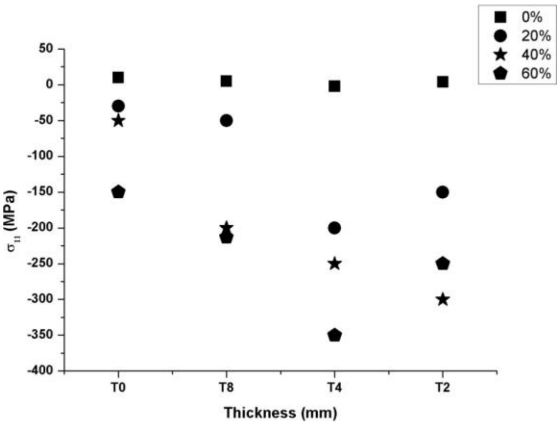

Figure 4.3σ11 Vs T (thickness level)

Likewise, compressive stress acts in perpendicular direction with respect to the rolling direction & it is represented by the σ_{22} and calculation is done. Following figure displays the compressive residual stress ranges from (-) 254 Mega Pascals to 72 Mega Pascals while its position also changes from top surface to half thickness of material, along with its reduction rate variation from of 22% to 66%. Additionally, it should be noticed that the stress gradient's tendency for the rolling direction and its opposing direction are the exact opposites

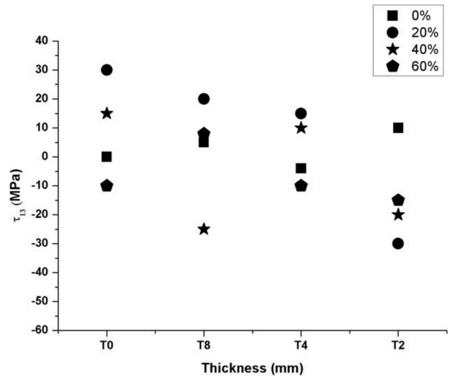

Figure 4.4σ_{22} Vs T (0 Thickness Level)

Shear stress investigation was carried out from top surface to half thickness of test subject. Material's shear stress ranges from 2.1 MPa at the top surface to 11.2 MPa at half of the thickness. As shown in figure mentioned below, the shear stress varies at different reduction rates, from (-) 32 Mega Pascals at the surface while having reduction rate of 22% to (-)19 Mega Pascals at the half of the thickness of material while having reduction rate of 66%.

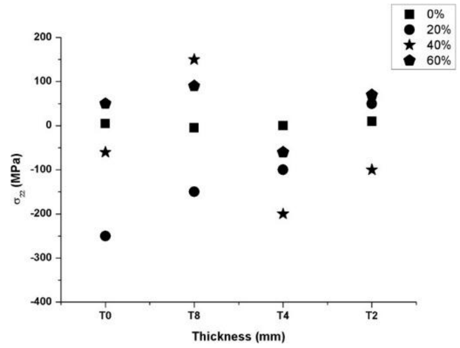

Figure 4.5σ_{11} Vs T (Thickness Level)

CONCLUSIONS AND POTENTIAL OPPORTUNITY

A fully recrystallized Ti-sheet of thickness of 7 mm was obtained from hot rolling and its outcomes at reduction rates of 22%, 44%, and 66% are summarised through experimental measurements of the development of the sheet's microstructure and residual stress. The key findings are as follows:

(1) Microstructure evolved, going from coarse to finer grains at various reduction rates across the thickness.
(2) A strong compressive residual stress that runs to both parallel and perpendicular direction of rolling (σ_{22})..
(3) Shear stress was also examined when the rolling was hot.
(4) In this investigation, a distinct stress gradient was found.

POTENTIAL OPPORTUNITY

(1) Relationship can be established for growth of texture.
(2) Allocation of residual stress can be found on the basis of microstructure
(3) Anisotropic yielding can used in a numerical investigation of the stress gradient

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