AN INVESTIGATION ON EFFECT OF TEXTURE ON THE PLASTIC DEFORMATION OF CP-TITANIUM

A thesis Submitted in partial Fulfilment of the Requirements for the degree of

Bachelor of Technology

IN

METALLURGICAL AND MATERIALS ENGINEERING

BY

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DEPARTMENT OF METALLURGICAL AND MATERIALS ENGINEERING

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CERTIFICATE

This is to certify that the thesis entitled, "**AN INVESTIGATION ON EFFECT OF TEXTURE ON THE PLASTIC DEFORMATION OF CP-TITANIUM"** submitted by **ASHISH KUMAR (111MM0457)**, **SANTOSH KUMAR** (**111MM0619**) in partial fulfilment of the requirements for the award of **Bachelor of Technology Degree** in **Metallurgical and Materials Engineering** at National Institute of Technology, Rourkela is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

Date: 1/5/15

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ABSTRACT

Due to extensive research and development in the area of materials in the last few decades, many material came up with unique properties. These unique properties let them to be used in various fields. One such metal is titanium, which is extensively used aerospace and defence sectors. It is due to its properties like high strength and toughness, high fracture toughness, high corrosion and erosion resistance and its use at various temperature ranges. It has also found its use in medical applications.

In our present work, we have tried to co-relate the texture and the mechanical behavior of the cp-titanium. Here annealed sample was rolled upto 90 % reduction and then samples were made along the rolling direction. This sample was tested in UTM Instron 1195 for its plastic deformation. It was deformed upto ultimate tensile strength only and then EBSD and OIM study was done. Then elastic modulus, misorientation, taylor factor was measured.

KEYWORDS: Texture, misorientation, elastic modulus, taylor factor.

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CONTENT

1.0. INTRODUCTION

Titanium and its alloys are one of the most widely used materials for structural and industrial applications [1-14]. Due to their high temperature applications and formability it has been widely used in automotive industries [12]. Due to their high strength-to-weight ratio, and working capabilities in both high and low temperatures it is used in aerospace and petroleum industries [12-13]. Titanium is alloyed with iron, aluminum, vanadium and molybdenum to produce strong and light weight alloy for aerospace applications like jet engines, missile and spacecraft; military, industrial process (chemicals and petrochemicals, desalination plant, pulp and paper), automotive, medical prostheses, orthopedic implants, dental and endodontic instruments and files, dental implants, sporting goods, jewelry, mobile phones and other applications [13]. These metal/alloys have excellent high temperature stability, due to which they are also used in turbines engines [14].Metallurgy of titanium is very expensive. So the application of titanium is limited to those sections only where either high performance or where life cycle cost justify its selection and usage. Although Titanium and its alloys are expensive, it cannot be compromised on account of its extraordinary properties, low maintenance cost and long life use. As titanium has a strong affinity towards oxygen thus the extraction, downstream processing and extrusion becomes expensive. This limitation has started a considerable amount of scientific and technological interest in developing potentially viable and economically affordable manufacturing methods that can reduce the cost of titanium and can make it a material of daily use.

For titanium, the common slip modes are {1 0 -1 0}, {1 0 -1 1}, and {0 0 0 1} planes with <1 1 -2 0> as the slip direction. Due to limited slip systems operative during plastic deformation of titanium, twinning becomes the main factor for maintaining the deformation

capabilities [15-16]. Hence dislocation slip and twinning occur concomitantly during plastic deformation [17]. It has been well established that different thermo-mechanical processing of titanium and its alloys improve their mechanical properties or deformation properties through adequate texture evolution [16]. The present study is an attempt to establish the deformation or mechanical behavior of polycrystalline commercially pure (CP) titanium as a function of orientation or texture of individual grains.

1.1. Objective

Our objective of this project is to establish the effect of the texture on the mechanical responses of CP titanium during plastic deformation. The main objectives are:

1. Microstructural developments during uniaxial tension of CP-titanium sheet.
2. Study of texture and other properties through EBSD (electron backscattered diffraction) analysis.
3. Correlation of microstructure and property of CP-titanium under uniaxial tension.

2.0 LITERATURE REVIEW

2.1 Titanium and its background

Titanium is the ninth most abundant element in the earth's crust and the fourth most abundant element. The commercial production of titanium started in 1948 due to its huge demand in aerospace industries. World titanium sponge metal production reached 166,000 metric tons in 2008[19]. The main titanium-containing minerals are rutile, ilmenite and leucoxene (Table 1) [20]. Ilmenite is the most important source of titanium and will continue to be the chief ore for the production of titanium. Ilmenite ($FeO \cdot TiO_2$ or $TiFeO_3$) contains 40–65% TiO_2. Leucoxene ($Fe_2O_3 \cdot nTiO_2$) is a natural alteration product of ilmenite, typically containing more than 65% TiO_2.

Table 2.1

Titanium minerals and their chemical compositions.

Mineral	Composition	TiO_2 content (%)
Rutile	TiO_2 (tetragonal, twinned)	~95%
Anatase	TiO_2 (tetragonal, near octahedral)	~95%
Brookite	TiO_2 (orthorhombic)	~95%
Ilmenite	$FeO \cdot TiO_2$	40–65%
Leucoxene	$Fe_2O_3 \cdot nTiO_2$	N 65%
Arizonite	$Fe_2O_3 \cdot nTiO_2 \cdot mH_2O$	—
Perovskite	$CaTiO_3$	—
Geikielite	$MgTiO_3$	—
Titanite or sphene	$CaTiSiO_5$	—
Titaniferous magnetite	$(Fe \cdot Ti)_2O_3$	—

The extraction of titanium is mainly done by Kroll extraction process. Here first titanium ore is converted into titanium sponge by the following steps:-

1. Passing Cl gas through the ore resulting in colorless titanium tetrachloride.
2. Titanium tetrachloride is purified by fractional distillation.
3. The liquid form of titanium tetrachloride is reacted with either Mg or Na under an inert (Ar) atmosphere to obtain titanium sponge while Mg or Na is recycled.

The sponge titanium is melted along with its alloying elements in the briquette form. It can be done by the following process:-

1. Electroslag refining.
2. Vacuum arc remelting.
3. Electron beam melting.
4. Plasma arc melting.
5. Induction skull melting.

The crystal structure of Ti is HCP alpha phase at room temperature, but due to allotropic transformation at 882 degree centigrade its crystal structure changes into BCC beta phase. The alloying elements which are alpha phase stabilizers are Al, O and N and the beta phase stabilizers are Mo, V, W, Nb, Ta, Fe, Cr, Cu, Ni, Co and Mn. The alloying elements which are neutral towards phase transformation are Zr, Si and Sn.

Classification of titanium alloys:-

1. Alpha or near alpha titanium alloys:- It is generally called commercially pure (CP) titanium. It is non-heat treatable and weldable alloy. It has good corrosion resistance, medium strength and good creep strength.

2. Alpha-Beta titanium alloys:- It is heat treatable and have good forming properties. It has high to medium strength and also have good creep strength.
3. Beta titanium alloys:- It is also heat treatable and readily formable. It has very high strength and low ductility.

CP titanium is the main alloy which is used vastly and have various applications. It has HCP structure and the purity is 99.0 to 99.5. The main elements in unalloyed titanium are Fe and interstitial elements such as C, O, N and H. O content determines the grade and the strength. C, N and H present as impurities. H mainly give hydrogen embrittlement to titanium. The main properties of CP titanium are as follows:-

1. Lower strength, depending on the contents of O and N.
2. Corrosion resistance to nitric acid, most chlorine.
3. 0.2 % Pd addition improves corrosion resistance in HCl, H2SO4, H3PO4.
4. Less expensive.

The main applications of titanium are airframes, heat exchangers, chemicals, marine, surgical implants.

During crystallization from melt to solid state and thermo-mechanical processing of Ti alloys, it has been found that there evolve some texture in the material. Texture is very important as it influences the material properties. Indeed, it has been found that the influence of texture on material properties is, in many cases, 20%–50% of the property values. Depending on the orientation of the principal stress axis with respect to the dominant basal plane texture, there is a significant variations in yield and ultimate tensile strength, bend ductility and total fatigue life to

failure. In monotonic loading where stress is parallel to the transverse plate orientation, we found higher modulus and improved yield and ultimate strength.

2.2. Texture or Preferred orientation

It has been found that the properties of the crystalline materials depend on the properties of each single crystal. It also depends on the parameters characterizing the polycrystalline state. Texture or say preferred orientation plays an important role in determining the mechanical as well as the physical properties [21]. It is the distribution of crystallographic orientation of a polycrystalline material. Depending upon the amount there is no texture or strong texture. It is seen in almost all engineered materials that it has a strong effect on mechanical properties. This give anisotropic properties to the materials. It is mainly classified into two; deformation texture (due to any type of deformation in the material) and recrystallization texture (due to the heat treatment of the material). It has been found that if there is some texture in a material, the material would behave like a single crystal material. But it doesn't mean that there is only one type of preferred orientation. In any polycrystalline material there are more than one component of texture. Due to severe amount of deformation, as in rolling or wire drawing, material develop the texture where crystallographic planes tend to orient themselves in a preferred manner with respect to the direction of maximum strain. Texture can be determined by various methods. Some of them allow the quantitative analysis of the texture, other are only qualitative. XRD and EBSD are two main methods for calculating the texture present in the specimen. Texture can be represented by pole figure and inverse pole figure. Orientation distribution function can represent the texture of each component in precise manner [22-25].

2.2.1. XRD (x-ray diffraction)

It can be used for different kind of materials but the limitation is the analysis of materials with multiphase systems and low crystalline symmetry. Here relative intensities of diffraction peaks are used as preliminary indications of crystal texture. A stronger peak identifies a direction of preferred crystallographic orientation that is normal to the diffracting planes for the peak. The degree of texture is indicated by the full width at half maximum (FWHM) of the peak. XRD line profiles are broadened due to (1) Small crystalline sizes and (2) Lattice distortions. These two can be separated on the basis of different diffraction order dependence of peak broadening and it is based on FWHM of the peak. The XRD profile gives (1) Crystalline size distribution and (2) Dislocation structure in nanocrystalline materials. Here Fourier coefficients of the physical profiles are fitted by the Fourier coefficients of well established *ab initio* functions of size and strain peak profiles. It is a macroscopic analysis. Here texture goniometer and Geiger counters are used.

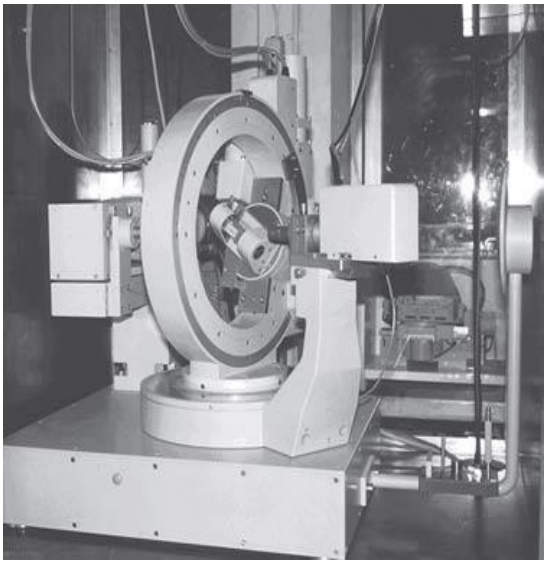

Figure 2.1. Goniometer

2.2.2. EBSD (electron back scattered diffraction)

Here backscattered electrons are studied to determine texture in scanning electron microscope. Within a single grain, electron beam is fixed at a point on the surface and due to the diffraction of beam at particular angles the intensity of the reflection is measured. This leads to the formation of a backscattered Kikuchi pattern made up of Kikuchi lines. Here the sample should have high surface quality, so polished by 1200 mesh papers and then electro-polished at room temperature at 17 V in a solution consisting of 25% H_2SO_4 + 15%HF + 60% CH_3COOH. Due to the location and symmetry of the backscattered Kikuchi pattern, we index the Kikuchi lines. These indexed patterns are used to describe the orientation of the grain within an experimental error of ±2.

The information stored by commercial software using this method includes specimen coordinates as well as the crystal orientation. So a two dimensional map of the orientation of grains on the surface of a polycrystalline material. Each grain orientation is described with reference to an external frame of reference. Here the orthogonal axes are (1) The directional normal to the plane of sheet and (2) the transverse direction parallel to the plane of the sheet. It is a microscopic analysis.

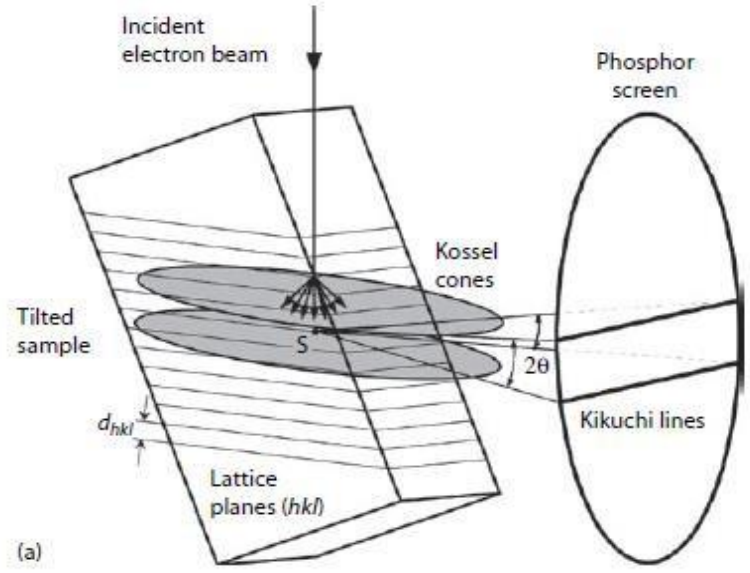

Figure 2.2. Kikuchi lines from an EBSD

2.2.3. Pole figure

Here projection of poles onto a pole figure from a reference figure is appeared. It is simply a stereogram with its axes defined by an external frame of reference with particular hkl poles plotted onto it from all of the crystallites in the polycrystalline material [26-27]. The external frame is defined by the normal direction (ND), rolling direction (RD) and transverse direction (TD) in the sheet. The position of the obliged pole on the circle is resolved as far as two angles: The angle α is the azimuth of the pole, where north pole is given by α = 0° and the angle β describes the rotation of the pole around the polar pivot, beginning from a specified reference bearing. While rolling of the sheet normal course ND is ordinarily decided to be in the north pole of the circle, with α = 0° for ND, and the rotation angle β is 0° for the rolling heading RD or, less every now and again, the transverse bearing TD. For other deformation modes or example geometries a fitting 3-D, ideally right-handed, coordinate framework must be determined.

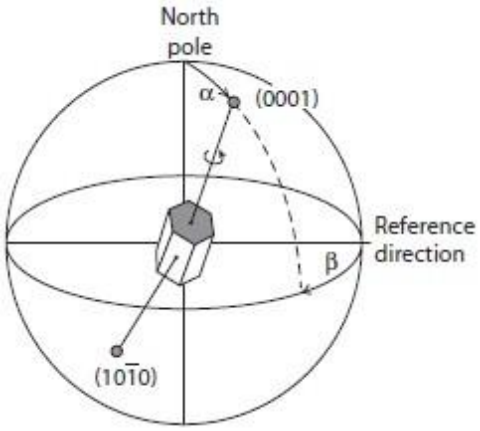

Figure 2.3. Orientation of titanium crystal of basal plane

A schematic of (100) pole figure is represented in figure 2.4. In figure 2.4a, the Stereographic projection of (100) poles is represented and the projection of (100) poles of one grain on the equatorial plane is represented in 2.4b. Projection of the (100) poles of a polycrystal where the grains are randomly oriented is represented in 2.4c. Figure 2.4d shows the projection of (100) poles of a textured polycrystal, and this is usually represented as contour maps – as shown in figure 2.4e.

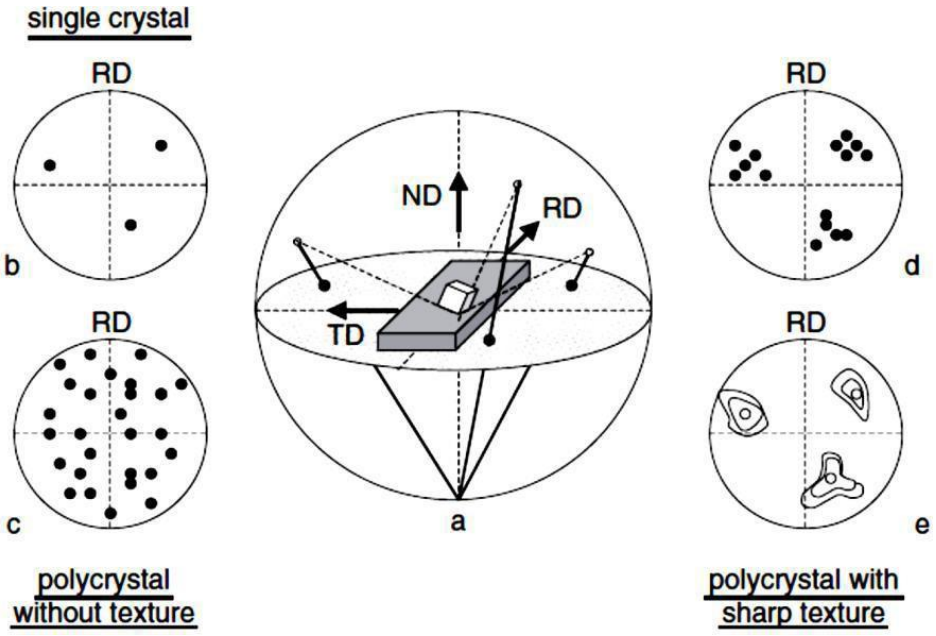

Figure 2.4. Schematic representation of pole figure of basal plane

2.2.4. CODF (crystal orientation distribution function)

The limitations present in pole figure representation of texture lead to the invention of orientation distribution function (ODF) , where it analyze all intensities separately and during projection it is always possible to miss certain orientations [26-27]. CODFs are constructed by combining the data obtained from several pole figures. It requires intense use of mathematics. It can be mathematically expressed as:-

$$(dV/V) = f(g)dg$$

Where g is the orientation of the grain and V is the volume fraction of grains in all intervals $g \pm \Delta g$. A sample in which there is no texture, the value of ODF would be zero otherwise it would have some maximum or minimum value [28-29]. It describe the orientation *g* of each grain relative to three Euler angles. Euler angles define the difference in orientation between the crystal axes and the deformation axes i.e. RD, ND and TD.

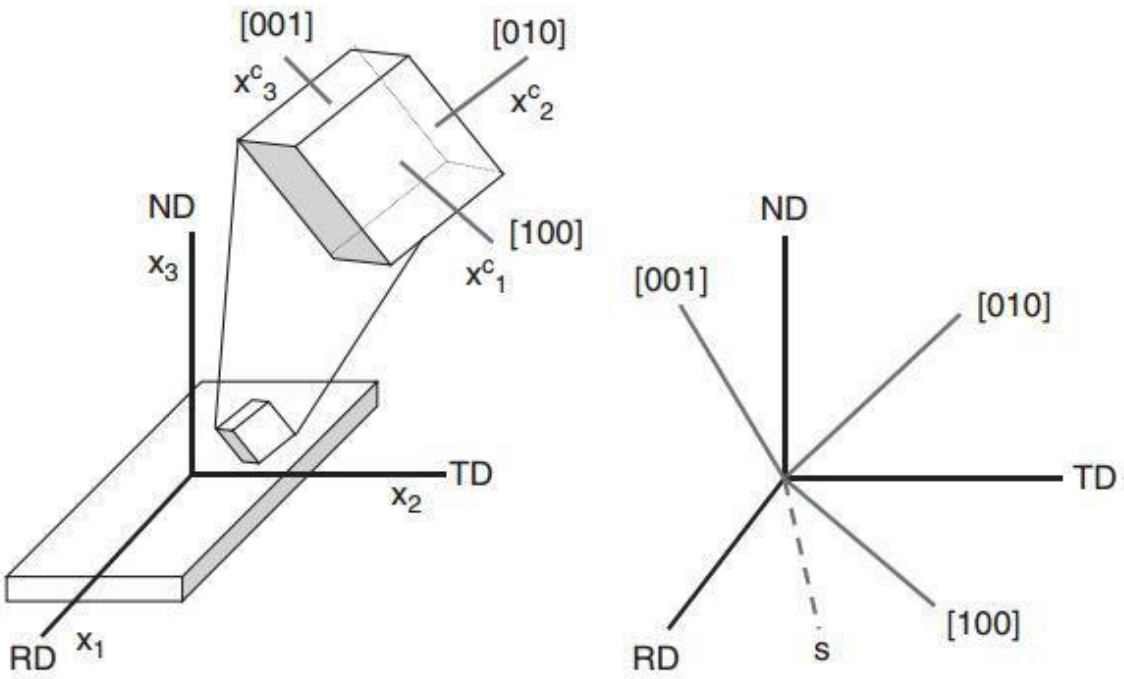

Figure 2.5. Orientation of the crystal axis system (Xic) and the sample axis system {RD, TD, ND}; *s* is the intersection of the planes (RD–TD) and ([100]–[010])

Now by using Euler angles in Cartesian co-ordinates, we form Euler space. There are three angles which are ϕ_1 , ϕ_2 and Φ. Φ is varied between 0 and 180 degrees and ϕ_1 and ϕ_2 can be varied between 0 and 360 degrees. We can look for individual orientations at several locations.

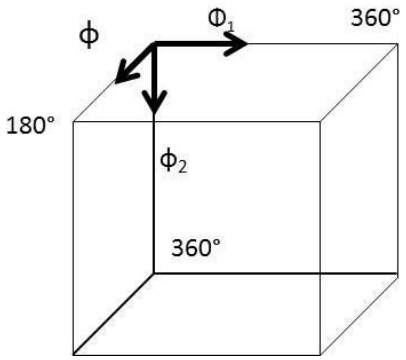

Figure 2.6. Euler space and Euler angles

2.3. Plastic deformation

Plastic deformation is a process in which enough stress is applied on metal that causes the object to change its size shape in a way that is not reversible. There are mainly two mechanism by which the plastic deformation takes place at room temperature. These are:-

1. Slip
2. Twin

In any polycrystalline material to be able for homogenous plastic deformation, minimum five slip systems are required. In HCP crystal system there are mainly three planes on which slip can occur which are (1) Prism plane (10-10), (2) Pyramidal plane (10-11) and (3) Basal plane (0001). Here slip occurs due to activation of <a> type Burgers vector which is maximum at {1 0 -1 0} <1 -2 1 0> prism plane, to some extent in {0 0 0 1} <1 -2 1 0> basal plane and least at {1 0 -1 1} <1 -2 1 0> pyramidal plane as the critical resolved shear stress increases in the same manner [30]. There is one more slip system that takes place due to <c + a> slip at the pyramidal plane having slip system {1 0 -1 0} <2 -1 -1 3> [31]. As the condition of five slip system is not fulfilled, introduction of twin system in the homogenous plastic deformation of cp-titanium is necessary [32]. So, here in cp-Titanium plastic deformation mainly takes place by slip as well as twinning, twinning playing the essential role in deformation [33]. When the deformation is very less, slip plays a major role in deformation. But as the amount of deformation increases, twinning start playing a major role. The grains which deform due to twinning get fragmented and the grain which deform by slip get elongated without fragmentation. Upto plastic deformation of 10% reduction, twinning plays important role. But the microstructure remains nearly same except for the formation of twins in a few grains. As the reduction increases to 20%, more twinning was activated and due to the formation of grains, because of fragmentation, grains with and without

twins are clearly identified. As the deformation increases upto 40% reduction, twinning plays important role in plastic deformation and significant refinement of grains takes place. Now due to the crossing of deformation twins and generation of secondary and tertiary twins, a twinned lamellar structure get developed of thickness of 1-5 micrometer compared to the initial grain size of 30 micrometer and the untwinned grains remain coarse due to slip.

Here the twinning are of two types:-

(1) Compressive twins {1 1 -2 2} (1 1 -2 -3) whose activation doesn't depend on the orientation of the matrix.

(2) Tensile twins {1 0 -1 2} (1 0 -1 -1) whose activation depends on the orientation of the matrix.

Twinning was observed only in grains whose basal poles were located within approximately 50 degree centigrade from the normal direction. In grains where basal poles are located 40 to 90 degree from the normal direction towards the transverse direction, slip mechanism is the main deformation mechanism. Beyond 40% reduction, due to the saturation of twinning, slip plays main role in deformation.

3.0 MATERIAL AND EXPERIMENTAL PROCEDURE

3.1. Material

Here the material used in the present study was cp-titanium plate of 0.5 mm thickness. Now these 0.5 mm plates were obtained from a plate having the thickness of 5 mm. To obtain a 0.5 mm plates, the 5 mm plates were cold rolled to a thickness reduction of 90 % in a rolling mill laboratory. Then annealing of these plates was done at a temperature of 600 degree centigrade of the time span of 1 hour. The chemical composition of the sample is given in table 3.1. Subsequently tensile test specimen (see Figure 3.1.) was prepared from the annealed plate for uniaxial tensile testing.

Table 3.1. Composition of the sample

Fe	C	N	H	O	Ti
0.034	0.004	0.004	0.0004	0.134	Balance

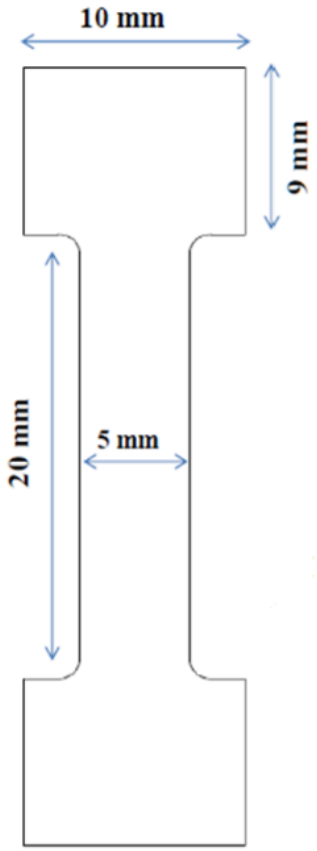

Figure 3.1. Sample specimen

3.2. Tensile Test

Tensile test of the sample was carried out in an Instron 1195. Tensile deformation of the sample which was in rolling direction was done beyond the yield point up to the ultimate tensile strength point.

3.3. EBSD

The metallographic and electro-polishing were carried out before EBSD analysis. For metallographic polishing, the standard procedure were followed. For electro-polishing, we do it in a Stuers polisher, LectroPol-5, at 25V for 20 seconds. Here the electrolyte used was methanol and perchloric acid both in the ratio of 80:20. These procedures were carried out at -20 degree centigrade. For EBSD analysis, Fei-quanta SEM (scanning electron microscope) was used which was attached with TSL-OIM. This whole setup is known as EBSD system. This EBSD scan gives us the grain size and grain average misorientation. EBSD scanning of an area 1 mm x 1 mm has done. We have calculated GAM (grain average misorientation), GOS (grain orientation spread), misorientation, elastic stiffness, low angle grain boundary, high angle grain boundary, Taylor factor and Schmid factor. Grain average misorientation is the average misorientation between the points in the grains. It can be seen as the amount of texture. If the GAM is high the texture will be less. Grain misorientation spread is the range of misorientation of grains by which it is misoriented. The angle between the grain boundary can vary between 0 degree and 180 degree. In low angle grain boundary, the angle varies from 0 degree to 10 degree and in high angle grain boundary the angle varies from 10 degree to 180 degree. In a polycrystalline material, to accommodate the slip five independent slip systems are required. Normally it has been observed that to activate these the yield stress should have a value two times of critical resolved shear stress which is the minimum value. And the factor by which the minimum CRSS get increased is called the Taylor factor. Thus the Taylor factor can be said as the ratio of the yield stress and the minimum CRSS.

4.0. RESULTS AND DISCUSSION

Figure 4.1. shows the inverse pole figure (IPF) map of the samples after plastic deformation till ultimate tensile strength. The figure clearly shows deformation heterogeneity of the grains/orientations: some grains were subjected to higher plastic deformation, some had undergone through fragmentation and some had twinned.

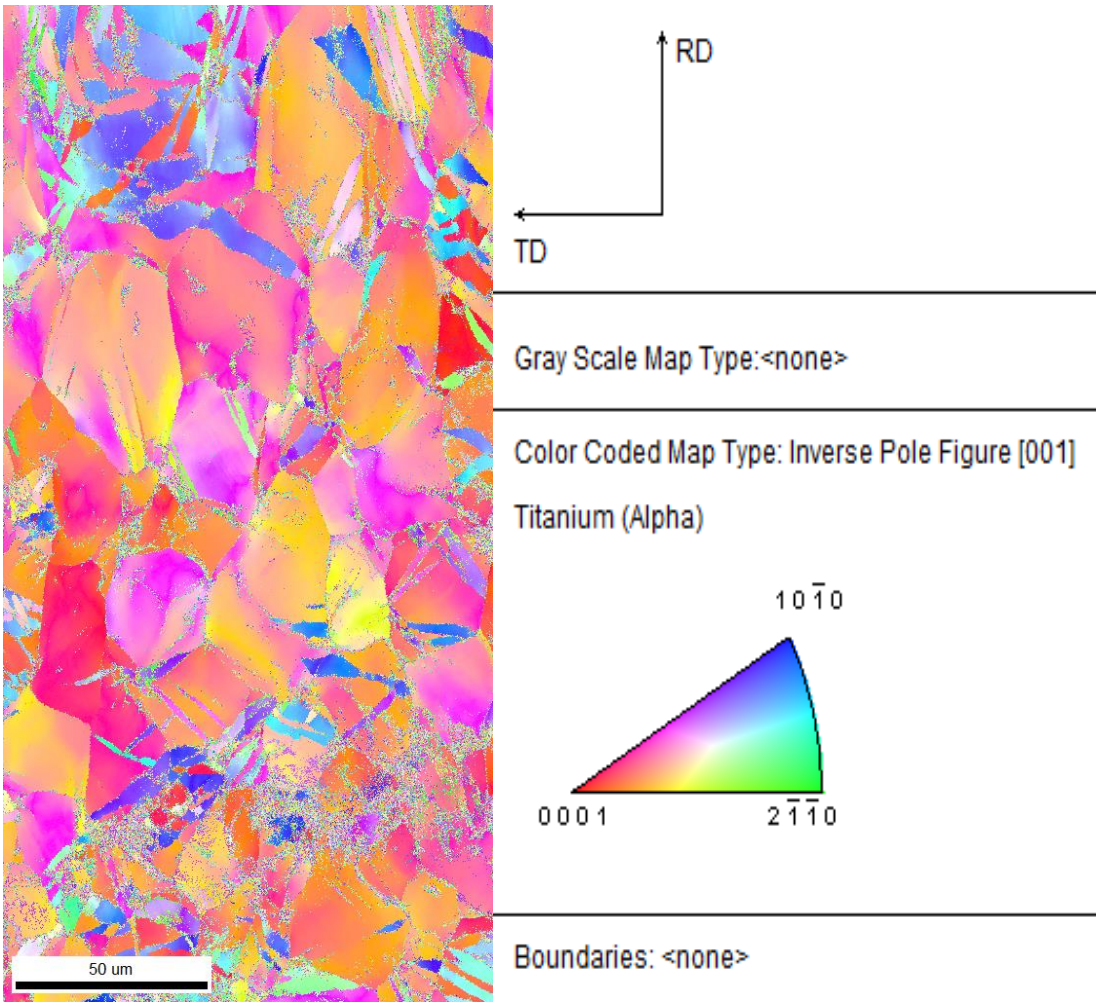

Figure 4.1 Inverse pole figure with grains

In figure 4.2 the value of GAM mostly lies between 0 and 2 degree. We have observed that number fraction of 0.25 have GAM value near 1 degree. It shows that majority of grains have very low misorientation i.e. majority of grains are oriented in the same direction.

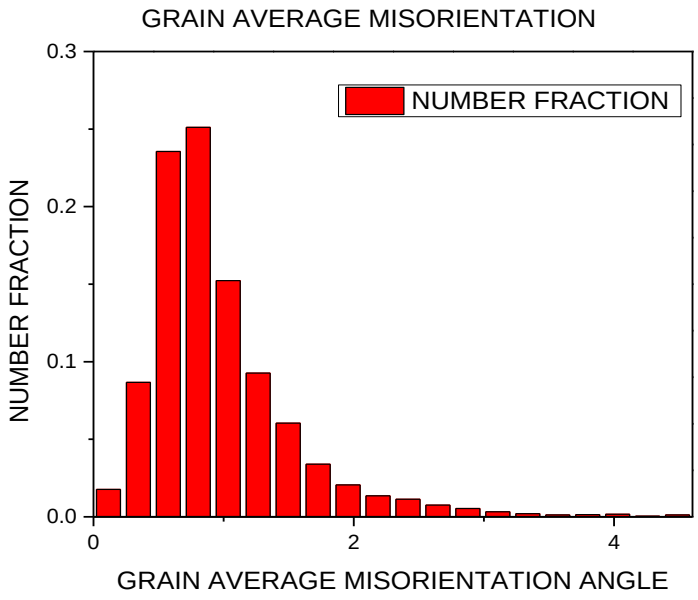

Figure 4.2 Number fraction VS GAM

From the figure 4.3 of GOS we can say that the spread of grain orientation is mostly less than 2 degrees having number fraction 0.7. It supports the previous observation of GAM that most of the grains are oriented in the same direction.

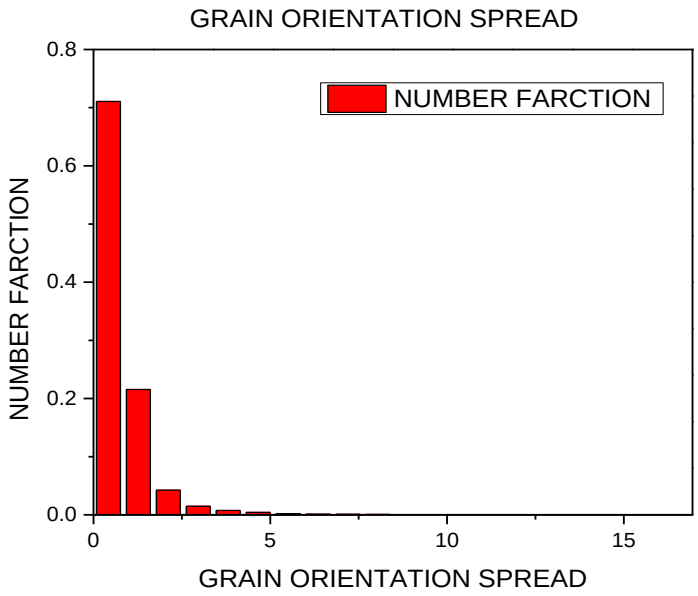

Figure 4.3 Number fraction VS GOS

If we see the low angle grain boundary in figure 4.4, it shows that 0.7 number fraction of grains have 1 degree of grain boundary. It means that majority of grains have low angle grain boundary. Number fraction of value 0.907 fall in low angle grain boundary.

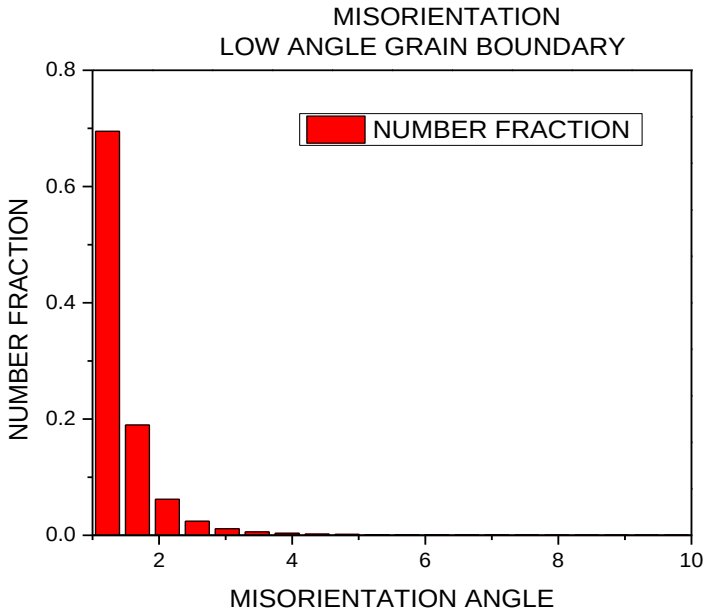

Figure 4.4 Number fraction VS LAGB

In the figure 4.5, the remaining grains, which fall in high angle grain boundary have two specified grain boundary angle. These are 94.8 degree and 64.3 degree having total number fraction of 0.093. These two are the two twins which are found in titanium. 94.8 degree and 64.3 degree corresponds for tensile twin and compressive twin having number fraction 0.006 and 0.0008 respectively. So we can say that tensile twin is the major one.

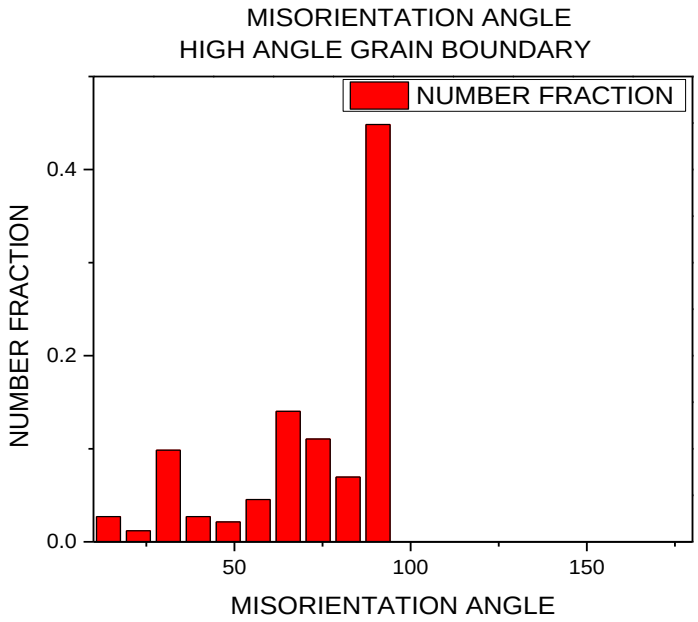

Figure 4.5 Number fraction VS HAGB

From the figure 4.6 given below, the value of elastic modulus mainly varies from 80 GPa to 110 GPa. The maximum number is near 0.15 having elastic modulus value near 90 GPa.

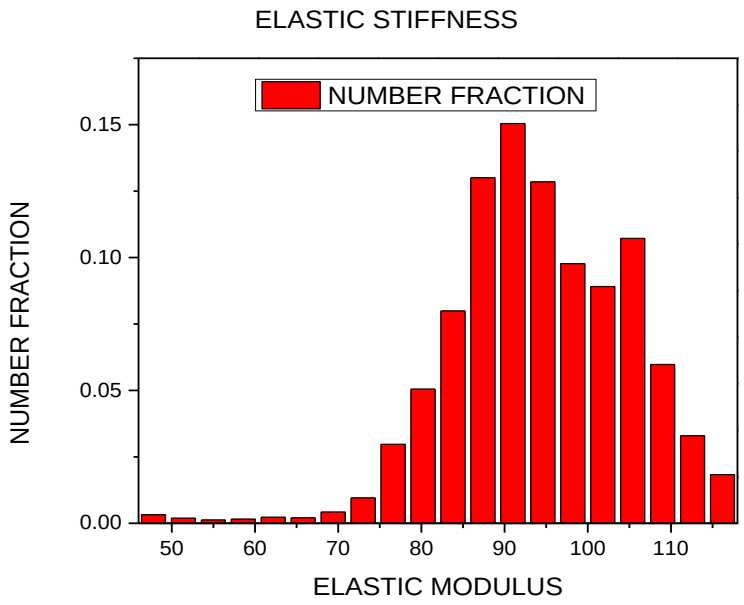

Figure 4.6 Number fraction VS elastic modulus

Now as we have analyzed each and every grain, we have observed these things.

It has seen in figure 4.7 that as we go from the basal plane through the near basal plane to the off basal plane the value of GAM increases.

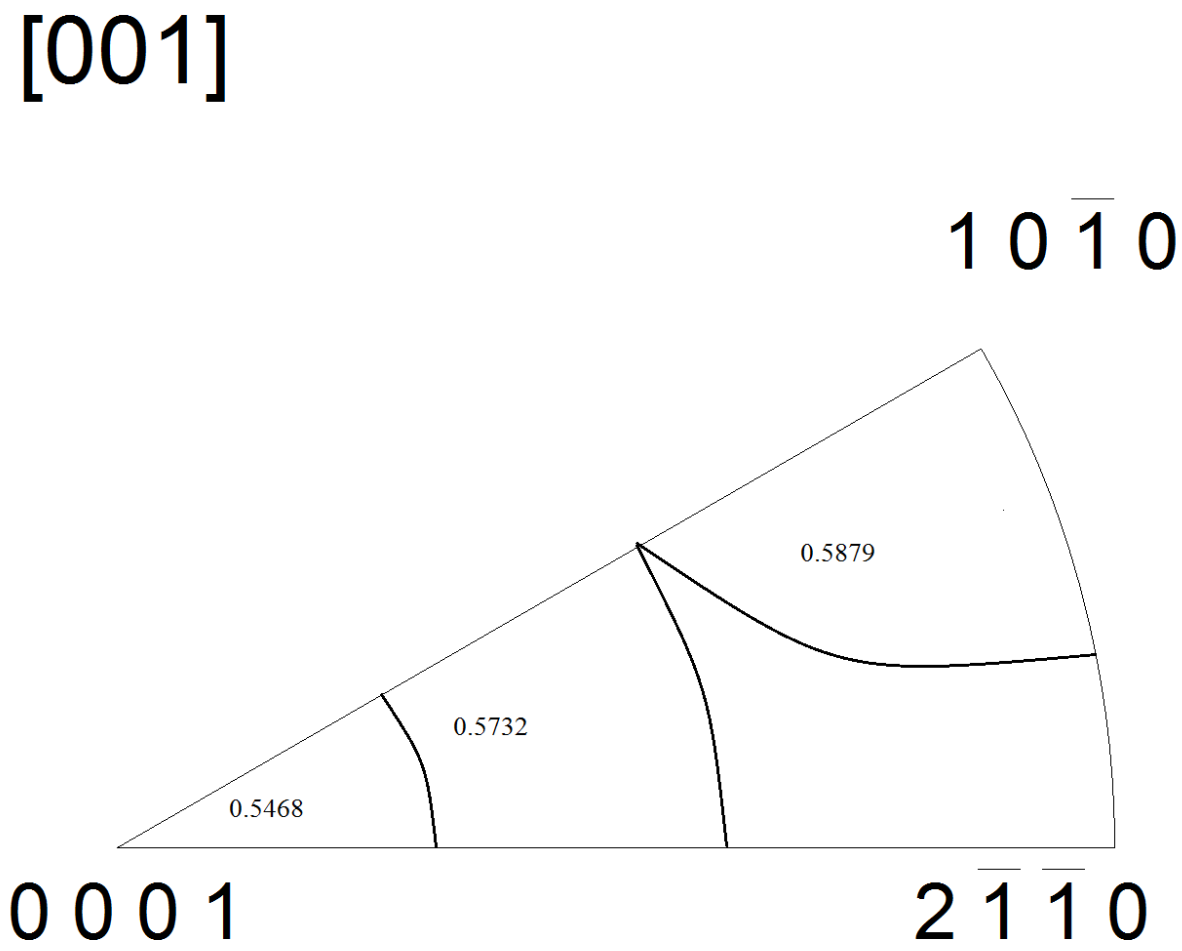

Figure 4.7 Inverse pole figure for GAM

As shown in the inverse pole figure 4.8 below, the Taylor factor as increases.

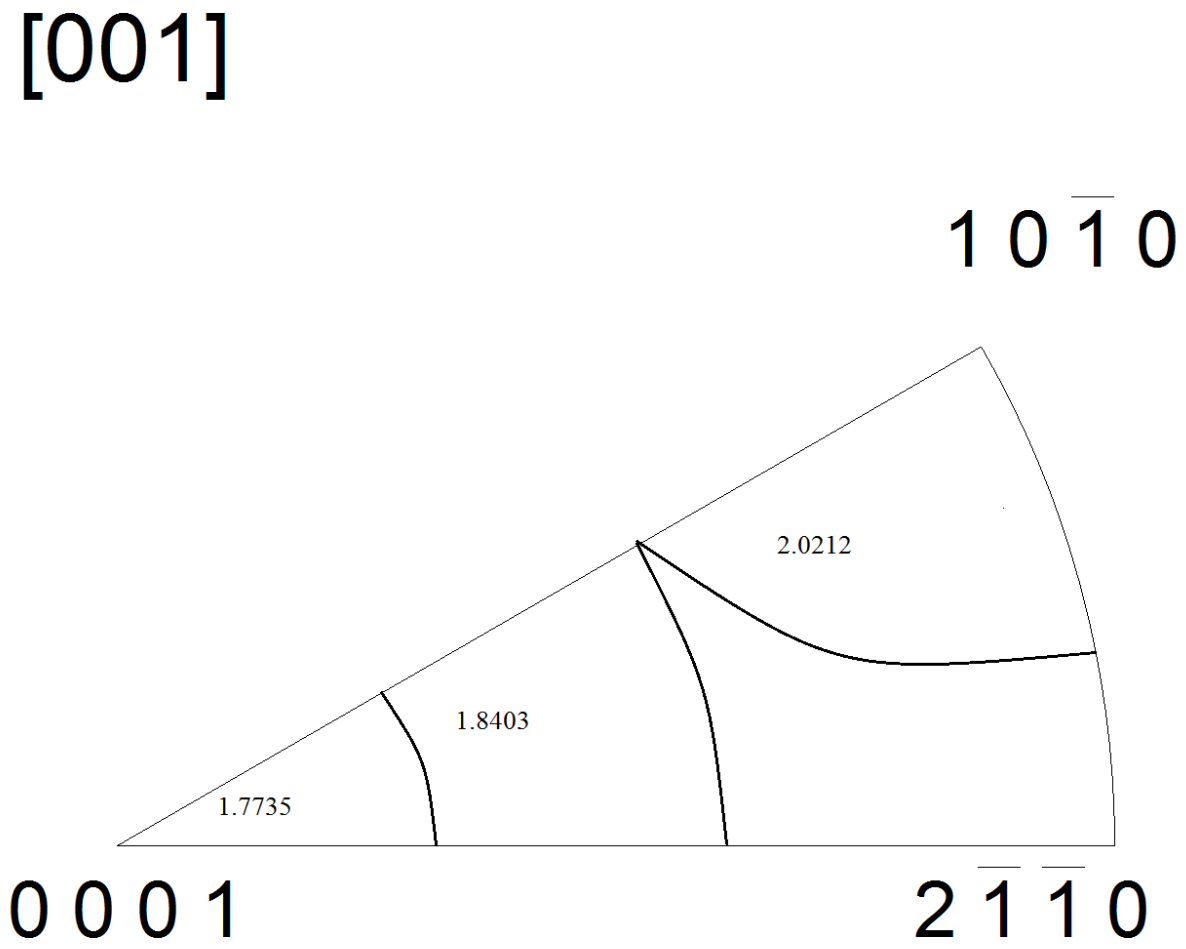

Figure 4.8 Inverse pole figure for Taylor factor

As seen from the figure 4.9 the value of GOS should also increase, but it increases upto near basal only and decreases in off basal region.

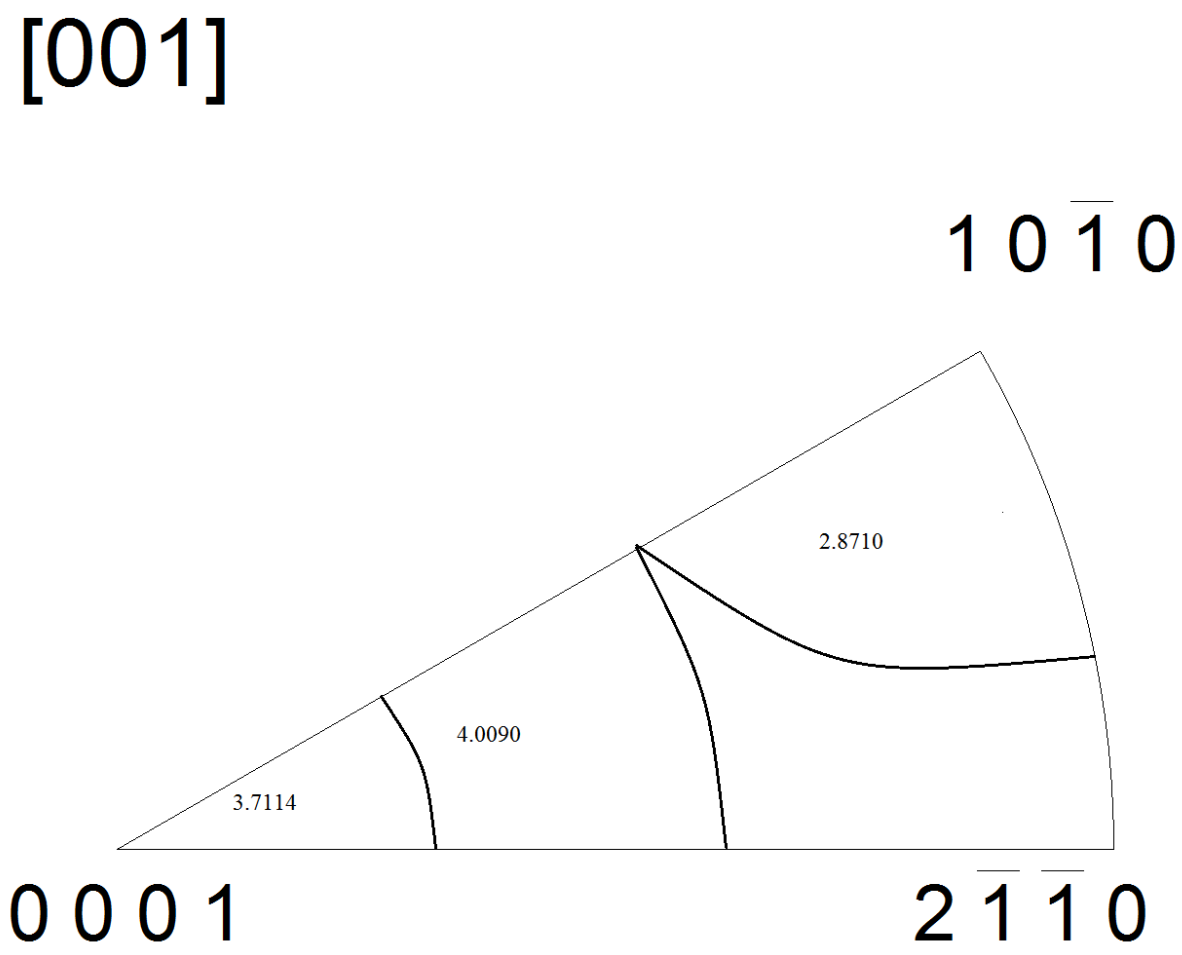

Figure 4.9 Inverse pole figure for GOS

Now we have observed from the figure 4.10, the elastic modulus value of the specimen increases as grains orientation shift from basal through near basal to off basal. We have also observed that maximum number of grains are in basal region.

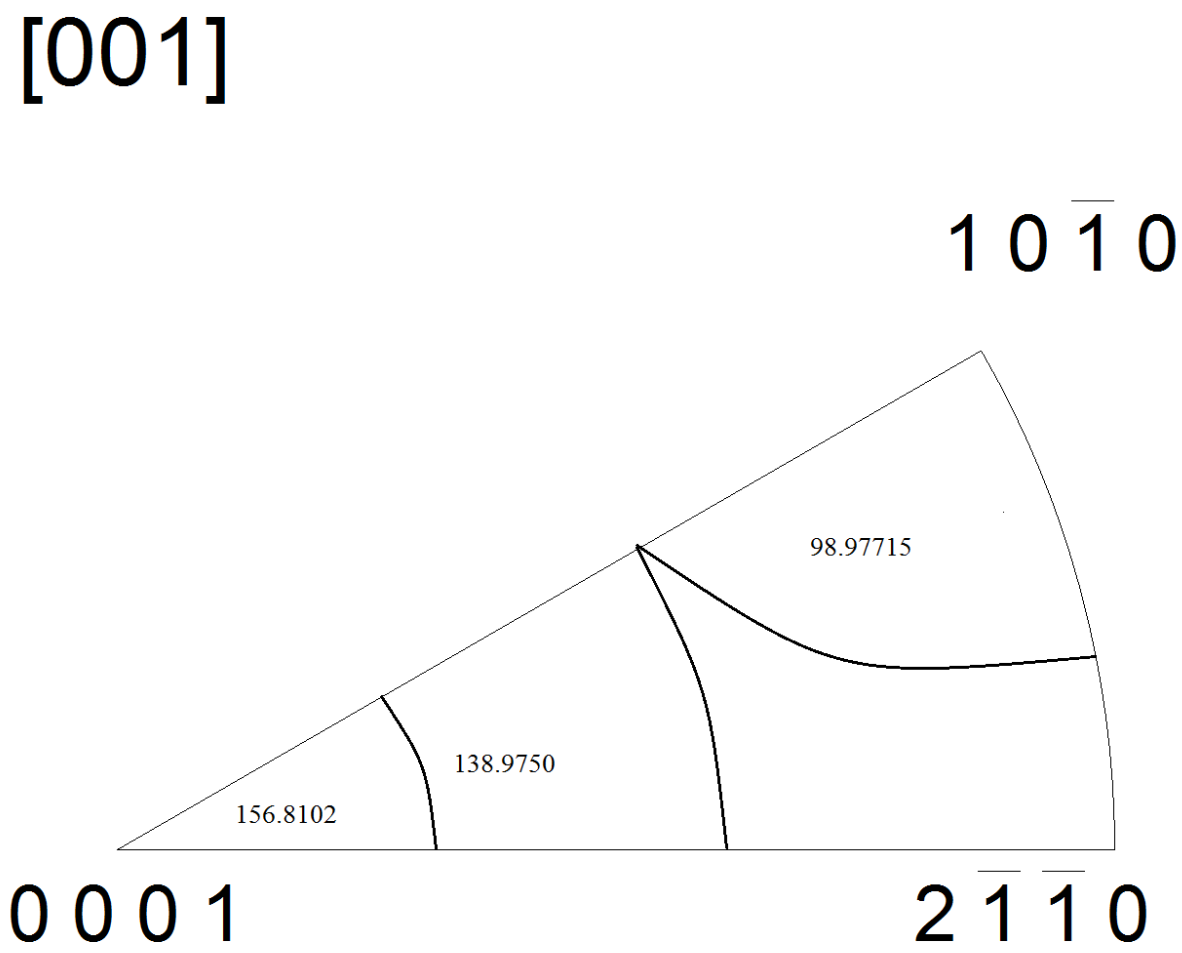

Figure 4.10 Inverse pole figure for elastic modulus

Now as we have seen that maximum number of grains have low angle grain boundary and these grains are oriented in the same direction. Now as seen from the inverse pole figure, the grains which have small grain boundary angle, small GAM and small GOS value fall in the basal region and this basal region have maximum number of grains. It suggest that plastic deformation mainly takes place in basal plane and its value decreases through the near basal plane to off basal plane. After this plastic deformation takes place by twinning. Now as it has observed that the grains which fall in the basal region have greater elastic modulus value.

5.0 CONCLUSIONS

From the above experiment we came to the following conclusions:-

1. Owing to the yielding upto ultimate tensile strength, the grains are mainly lies in the basal and near basal plane.
2. The GAM and GOS value are lowest in the basal region and highest in the near and off basal region.
3. Taylor factor increases as we move from the basal region through the near basal to off basal.
4. The grains in the basal region has the highest elastic modulus.
5. If the plastic deformation has taken place by basal plane, it has the highest hardness.
6. If the number of grains is high in basal region, good mechanical properties would be found.

6.0 FUTURE SCOPE OF THE WORK

In our present study, we have observed that mechanical properties of cp- titanium is dependent of the orientation of its grains i.e. on texture. But it structure and property co-relations are not fully understood. So there is bright future of its research where more techniques can come which will make it metal having desired properties.

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