

RECRYSTALLIZATION TEXTURE DEVELOPMENT IN CP-TITANIUM

A THESIS SUBMITTED IN PARTIAL FULLFILLMENT

OF THE REQUIREMENT FOR THE DEGREE OF

Bachelor of Technology

In

Metallurgical and Materials Engineering

By

SIDDHARTH PAL (109MM0120)

SANJAY KUMAR SAHOO (109MM0487)



DEPARTMENT OF METALLURGICAL AND MATERIALS ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

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Rourkela

Certificate

This is to certify that the thesis entitled “**Recrystallization texture development in CP-Titanium**” being submitted by **Sanjay Kumar Sahoo (109MM0487)**, **Siddharth Pal (109MM0120)** for the partial fulfillment of the requirements of Bachelor of Technology degree in Metallurgical and Materials engineering is a bona fide thesis work done by them under my supervision during the academic year 2012-2013, in the Department of Metallurgical and Materials Engineering, National Institute of Technology Rourkela, India.

The results presented in this thesis have not been submitted elsewhere for the award of any other degree or diploma.

Date:

(Prof. Santosh Kumar Sahoo)

Metallurgical and Materials Engineering

National Institute of Technology Rourkela

Rourkela, Odisha- 769008

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Place: NIT Rourkela
Date:

Sanjay Kumar Sahoo (109MM0487)
Siddharth Pal (109MM0120)

Abstract

Most of the structural components are subjected to annealing as a final forming operation for different applications. It is therefore very important to understand/know the texture development during annealing of a material. This will decide the mechanical property of the component. Annealing texture of cubic crystal system has been widely researched, but little work has been done for the hexagonal close-packing materials. In the present study recrystallization texture development in CP-titanium was investigated. CP-titanium plates were subjected to cold rolling of 90% reduction in thickness. The rolled samples were then subjected to isochronal annealing at 500⁰C, 600⁰C and 700⁰C for 30minutes to obtain the recrystallization temperature, determined by EBSD analysis. Final annealing was carried out at 600⁰C for different soaking time: 10sec, 20sec, 1min, 2min, 5min, 10min, 20min and 30min to establish the texture development during annealing. These annealed samples were subsequently characterized under XRD (X-ray Diffraction) for bulk texture measurement. The initial deformation texture i.e. (1 1 -2 4)<1 -1 0 0> got attenuated with time and development of new basal orientation i.e. (0 0 0 1)<1 -1 0 0> and non-basal orientations i.e. (2 1 -3 7)<1 -2- 1 0>, (3 1 -4 9)<2 -15 13 1> and (5 1 -6 15)<1 -5 4 0> were observed.

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1.0 Introduction

It has been quoted that the influence of texture on material properties is, in many cases, 20-50% of the property values [1]. Most of the researchers have involved in exploiting effect of texture on properties of materials. However, some researchers also worked on the mechanisms of texture developments [2,3]. It is well established that evolution of texture in a material occurs during plastic deformation or during annealing [4]. The former is known as deformation texture while the latter is recrystallization texture. The present study deals with recrystallization texture development in commercial purity (CP) titanium. Recrystallization texture development in cubic materials is well established [5,6,7], while in hexagonal materials it is not fully understood. Some researchers have reported the texture developments during annealing of hexagonal metals [2]. HCP metals and alloys exhibit a wider variety of recrystallization textures, which are strongly related to the initial deformation textures, annealing temperature and compositions. Some of HCP metals like zirconium and cadmium shows $\{11\bar{2}2\} \langle 10\bar{1}0 \rangle$ cold rolling texture, which on subsequent recrystallization annealing develops $\{10\bar{1}3\} \langle 11\bar{2}0 \rangle$ texture predominantly [8].

1.1 Objectives

In the present study the following objectives were aimed at:

- Prediction of recrystallization texture developments during '*precise*' annealing of CP titanium. Such annealing was done at recrystallization temperature for *a very small to large* soaking time.
- Quantification of recrystallization texture developments during annealing of CP titanium.

2.0 Literature Survey

2.1 Introduction

The micro-structure of a polycrystalline material, as we know, helps in determining its properties [9]. The grains within a polycrystalline material have the same crystal structure but different crystallographic orientation. It is due to these different crystallographic orientations exhibited by different grains, within the material, that the material is considered to be isotropic. However, in most of the materials, it is observed that the grains are not randomly oriented and is said to possess a definite crystallographic texture. Besides a crystallographic texture, materials can also show a morphological texture, e.g. when all grains are elongated in a particular direction (as in a rolled product) or when the material contains precipitates that are oriented in one or more preferential directions. The figure 2.1 shows schematic representation of a material without texture, with texture and with morphological texture. Crystallographic texture can be generated in a material by deformation or by recrystallization & grain growth.

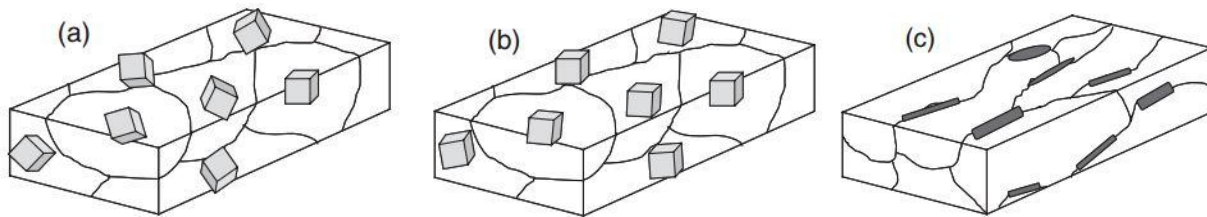


Figure 2.1 Illustration of (a) a material without texture, (b) a material with a crystallographic texture and (c) a material with a morphological texture. The small cubes represent the crystallographic orientations of the grains in (a) & (b). [9]

2.2 Texture Representation

Usually an external co-ordinate system is employed for expressing the orientation of a grain. For flat products like sheets or plates this frame of reference consists of rolling direction (RD), a normal direction (ND) and transverse direction (TD). The texture or the preferred orientation is commonly represented by $\{h\ k\ l\} [u\ v\ w]$. This represents the $\{h\ k\ l\}$ plane is parallel to the sheet plane and the direction $[u\ v\ w]$ is parallel to rolling direction (see figure 2.2a). In axisymmetric products (wires, extruded bars), the crystal orientation is described by one set of Miller indices

[$u\ v\ w$], indicating that this crystallographic direction is parallel with the sample axis (see figure 2.2b).

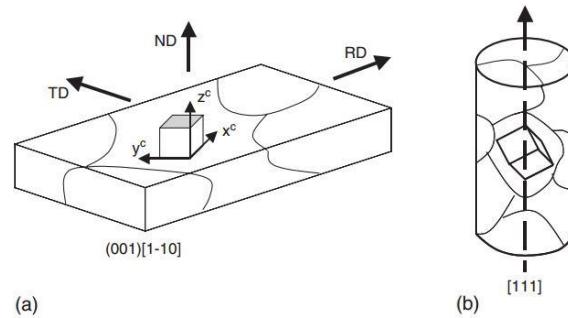


Figure 2.2 Representation of crystallographic orientations in, (a) sheet and (b) wire, expressed with Miller indices. [10.]

2.2.1 Pole Figure

The pole figure is the most frequently used method of representing conventional textures. A pole figure is a 2-dimensional representation of a particular set of equivalent crystal orientations, following the principle of stereographic projection [9-11]. The sample is placed on the center of the stereographic sphere, as shown in figure 2.3a, and for a particular pole figure construction, the plane normal is projected on the equatorial plane of the sphere. Figure 2.3 shows the (100) pole figure in a cubic material. Poly-crystalline materials having no definite texture have all these projection points scattered over the equatorial plane – figure 2.3c. When the material has a crystallographic texture, the projections will cluster together – figure 2.3d. But In most cases, it is not useful to show individual points but contour lines of equal pole density distribution – figure 2.3e.

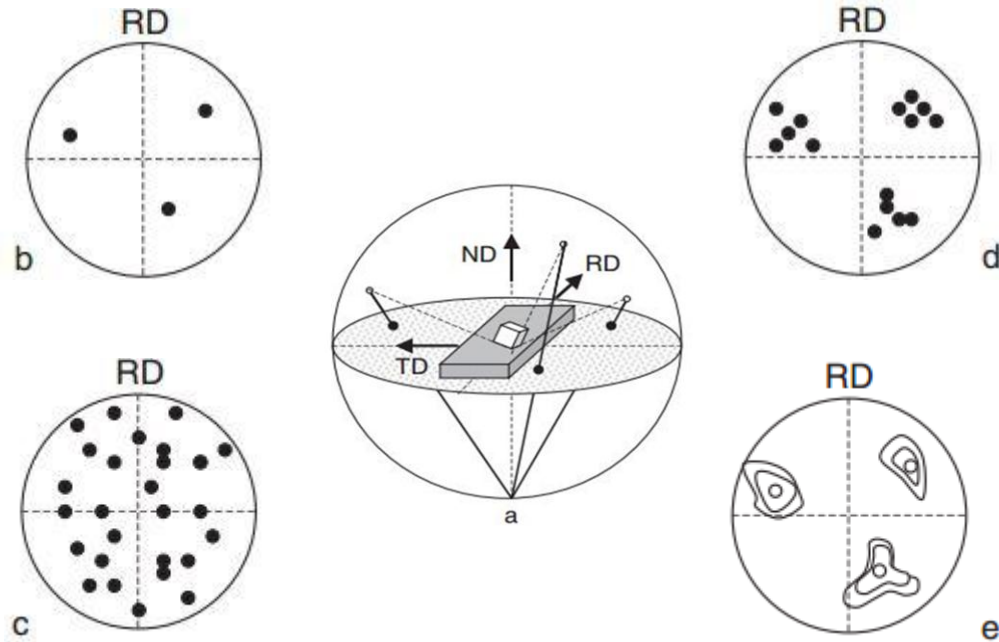


Figure 2.3 Construction of a (100) pole figure in a cubic material. (a) Stereographic projection of the (100) poles; (b) projection of the (100) poles of one grain on the equatorial plane; (c) projection of the (100) poles of a poly-crystal; (d) projection of the (100) poles of a textured poly-crystal; (e) contour map of the (100) pole density distribution. [11]

2.2.2 Orientation Distribution Functions (ODF)

There are certain limitations in pole figure representation of texture – in pole figure representation, it is necessary to analyze all intensities separately, and during projection it is always possible to miss certain orientations [12,13]. However, ODF describes complete texture information in a sample. An ODF is mathematically expressed as:

$$\frac{dV}{V} = f(g)dg$$

Where, g is the orientation of a grain and f is the volume fraction of grains in all intervals $g \pm \Delta g$. The ODF of a sample without texture is a constant. If the sample has a texture, the ODF has maxima and minima [14-16].

2.2.2.1 Euler Angles

Euler angles are used to define the orientation g of a grain, in order to give a graphical representation of an ODF. Two different co-ordinate systems are defined: the first is connected to the sample (sample axes system X_i) and the second to the crystal of a grain (crystal axes

system X_i^c) – see figure 2.4. The sample system is related to the shape of the sample. For example, for a rolled sheet, the axis X_1 is taken in the RD, X_2 in the transverse and X_3 in the ND of the sheet. The orientation of the crystal axes system can now be expressed in the reference frame of the sample axes system by three rotations (figure 2.5). Though there are several conventions of performing these rotations, most widely used system is the system of Bunge.

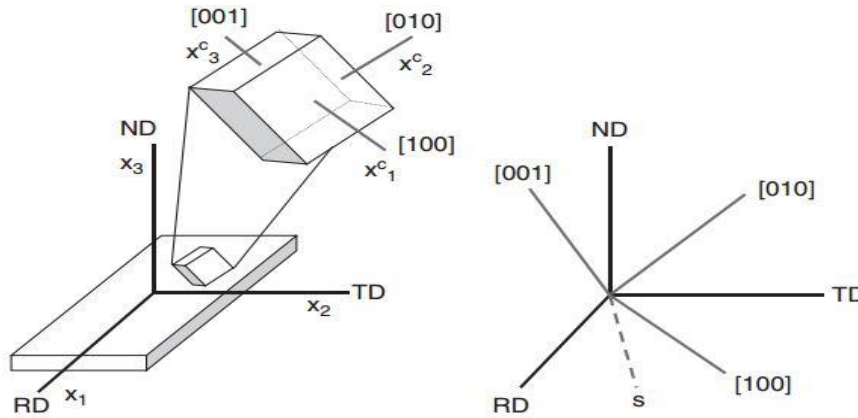


Figure 2.4 Orientation of the crystal axis system (X_i^c) and the sample axis system {RD, TD, ND}; s is the intersection of the planes (RD–TD) and ([100]–[010]). [17]

In Bunge system, first a ϕ_1 rotation is performed around normal direction (ND) which brings RD in the position s , with s the intersection of the planes (RD–TD) and ([1 0 0] – [0 1 0]). Now the RD' and TD' are the new positions of RD and TD due to the rotation. Then a rotation around RD' by Φ brings ND together with [0 0 1]. Because of this rotation position of TD' changes to TD''. Finally a rotation of ϕ_2 around ND axis or about [0 0 1] is given. Due to this rotation RD' falls on [1 0 0] and TD'' falls on [0 1 0]. The three angles (ϕ_1, Φ, ϕ_2) used for rotation about the different axes are called the Euler angles.

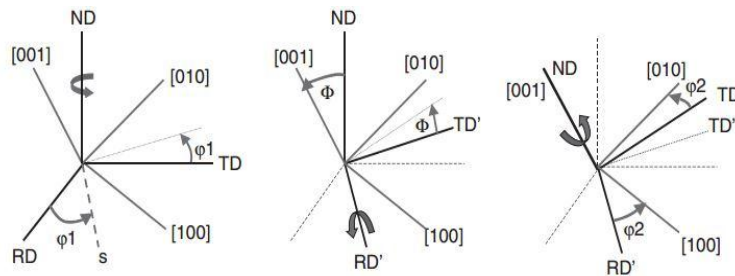


Figure 2.5 Definition of Euler angles, ϕ_1, ϕ and ϕ_2 , in Bunge convention.

2.2.2.2 Euler Space

This is obtained by plotting the three Euler angles in Cartesian co-ordinates (figure 2.6). The angles ϕ_1 and ϕ_2 can be varied between 0° and 360° but the angle ϕ is varied between 0° and 180° . In this representation, individual orientations will be found at several locations.

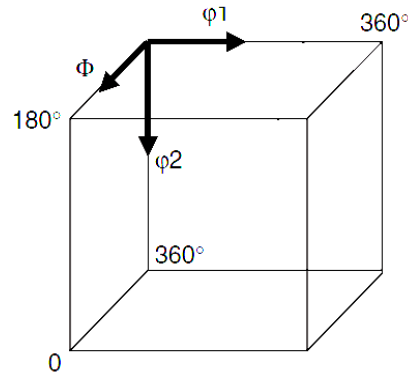


Figure 2.6 Representation of Euler space with Euler angles.

2.3 Recrystallization Texture

During plastic deformation of metals, the slip imposed crystal rotation of each grain gives rise to the development of deformation textures. When this deformed material is annealed, the initial cold deformed texture changes during recrystallization and subsequent grain growth. There are at least three different types of recrystallization texture known to be produced by recrystallization: First one is a largely retained rolling texture and secondly strongly randomized texture and last one is a completely new recrystallization texture [18-19]. This texture is responsible for the anisotropy in the mechanical properties of material and will in many cases determine the properties of the end product.

The development of recrystallization textures has been debated for the past 50 years. There are two competing recrystallization theories, which give idea about recrystallization texture:

- a. oriented nucleation theory (ON)
- b. oriented growth theory (OG)

2.3.1 Oriented Nucleation Theory

It is assumed that in a deformed matrix some grains or zones have more potent nuclei than others. This suggestion is based on the observation that certain sites are preferred sites for nucleation of recrystallized grains, for example: persistent cube bands, zone around non

shearable second phase, shear bands, etc. It is observed that in most of these preferred sites a prominence of recrystallized grains with a particular orientation can be found e.g. cube $\{100\}$ $\langle 001 \rangle$ oriented grains in persistent cube bands in Al, S $\{123\}$ $\langle 63-4 \rangle$ grains in shear bands in Al alloy etc. From such observation it has been concluded that not all potential nuclei are really activated but some kind of preferred nucleation exist in the deformed matrix. That is called oriented nucleation [16].

2.3.2 Oriented Growth Theory

As per oriented growth theory, all potential nuclei are activated without any preference. But those nuclei which have a fast growing grain boundary in nature compared to their neighbour will develop much faster and will determine the recrystallization texture. It is based on the observation that components of recrystallization texture can often be achieved from deformation texture by some particular rotation around some simple crystallographic orientation. Some examples are 30° rotation around a common $[0001]$ axis or 90° around $[10\bar{1}0]$ axis in hexagonal metals. 30° - 40° rotation around a common $[111]$ axis in FCC metals.

2.3.3 Parameters influencing recrystallization texture

- cold deformation
- starting texture and microstructure
- purity of metals
- amount of grain growth overlapping with recrystallization
- alloying elements

2.4 Recrystallization Texture in Rolled hexagonal Metals

Recrystallization texture of hexagonal metals has not been studied extensively. Early studies on recrystallization texture of hexagonal metals reported that the former rolling textures were often retained. However after high temperature annealing, there occurs distinct change in cold deformed texture. It is observed that recrystallization and rolling texture are related through a $30^\circ \langle 0001 \rangle$ orientation relationship that for hexagonal crystal symmetry. In particular $\{0001\}$ fiber texture is commonly found in Mg alloys. At high strain and high annealing temperature in both Zr and Ti alloy sheets, a preferred orientation $\{10\bar{1}3\} \langle 12\bar{1}0 \rangle$ has been described. This

Orientation has a $30^\circ\langle 0001 \rangle$ orientation relationship to the rolling texture orientation $\{12\bar{1}4\}\langle 10\bar{1}0 \rangle$.

2.5 Recrystallization Texture in Titanium Alloys

Some reported literature on recrystallization texture in titanium alloys is summarized in this section. Cold rolled (80% reduction) titanium sheets were given annealing heat treatment at temperature 500°C , 600°C and 700°C for 20mins, 30mins and 100mins respectively [1,18]. The figure 2.7a to d shows the evolution of texture from deformed state to advanced grain growth state. Interestingly, the texture has not changed substantially at the end primary recrystallization. The maximum of orientation, the ODF is still located around $\{\phi_1=0^\circ, \phi_2=40^\circ, \phi_3=0^\circ\}$. The so-called recrystallization-texture, characterized by an ODF maximum centered around $\{\phi_1=0^\circ, \phi_2=35^\circ, \phi_3=30^\circ\}$ appears progressively during grain growth, as visible on figures-2.7 c and d. The grain growth process is therefore, responsible for the well-known texture change of titanium (often described as a rotation by 30° about the c-axis). The primary recrystallization process, which fully regenerates the microstructure has nearly no effect on texture. The orientations for which the volume fraction has increased (positive values of the ODF difference function) are represented with red (or dark grey) level lines and the decreasing ones with green (or light grey) level lines. All over the recrystallization process, texture component corresponds to a broad peak centered around $\{\phi_1=0^\circ, \phi_2=35^\circ, \phi_3=30^\circ\}$, which will define the increasing recrystallization texture after extensive annealing.

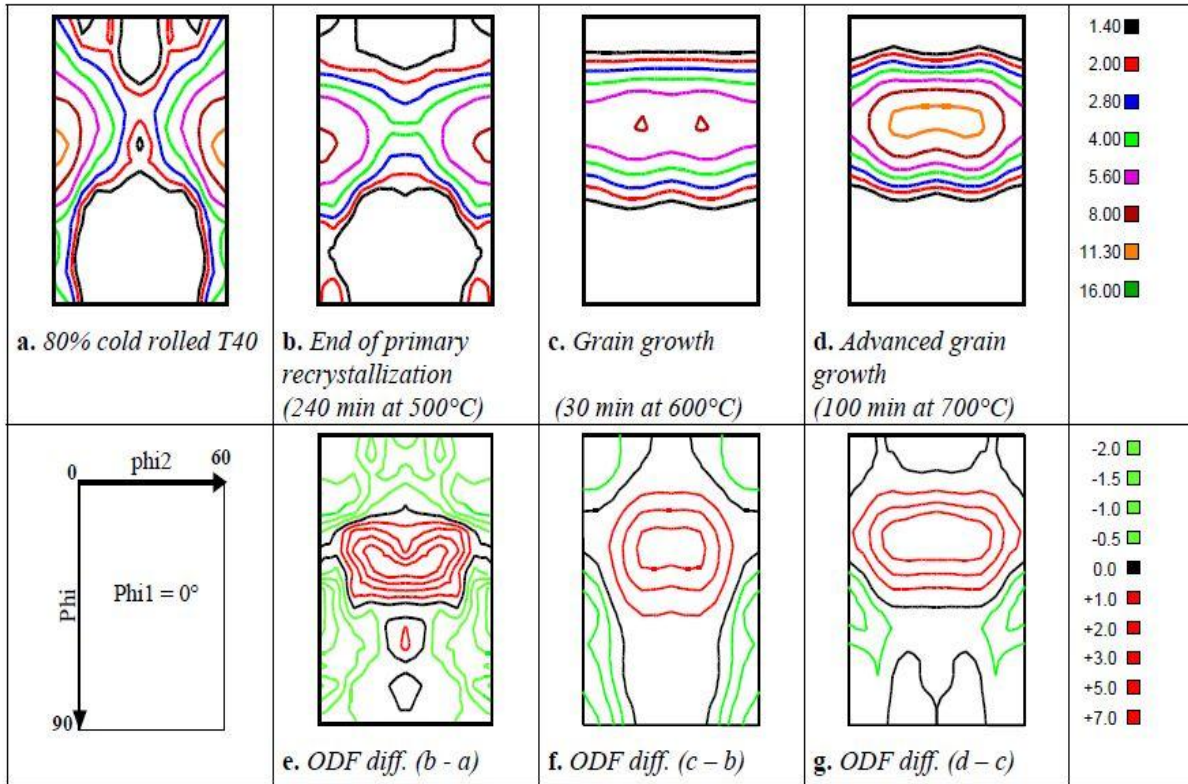


Figure 2.7 $\phi_1 = 0^\circ$ sections of the ODF (a-d) or of the ODF difference (e-g) corresponding to different states of recrystallization. [1]

The following three observations are made concerning the different stages of recrystallization:

- i) the orientations at Φ below 20° or above 50° strongly decrease during primary recrystallization.
- ii) the same type of orientations continue to disappear during subsequent moderate grain growth.
- iii) for an extensive compared to a moderate grain growth, the disappearing orientations are located around $\{\phi_1=0^\circ, \phi = 50^\circ \text{ to } 65^\circ\}$, the one at $\phi_1=\phi_2= 0^\circ$ and ϕ below 20° or above 65° do not exist any longer.

The orientations which totally disappear first (ϕ below 20° or above 65°) are highly misoriented with growing “stable” recrystallization component in the vicinity of $\{\phi_1=0^\circ, \phi = 35^\circ, \phi_2=30^\circ\}$. Deformation simulations have shown that these misorientations may result from twinning, and a detailed analysis of the grain size based partial textures has shown that the corresponding grains

belong to the smallest size range. Because of their smaller size and higher misoriented boundaries the grains belonging to these orientations clearly have strong disadvantage.

Another study on crystallographic texture in CP-titanium during annealing was reported [19-22]. First CP-titanium was given hot rolling from its cast slab, then annealing at 550°C for 30 min, followed by cold rolling (60% reduction in thickness) and annealing at 700°C for 30 min and 60 min [23,24]. The measured pole figures {0002} with five different processes, *i.e.*, the initial hot-rolled plate, annealing after hot rolling (550°C, 30 min), cold rolling, annealing after cold rolling (700°C, 30 min), and annealing after cold rolling (700°C, 60 min) shows that a texture is developed with basal poles tilted 40° towards transverse direction. Although, the texture strength of the initial hot-rolled plate is relatively high initially, it decreases sharply after annealing, but it increases at the processes of cold rolling and the second annealing [25-26]. The analysis of ODF sectional views with five different process shows that the texture of the initial hot-rolled plate can be classified into three kinds, namely, the (-1 0 1 3)[5-2-30] and (-2021)[10-15], the basal plane texture (0001)[2-1-10], and the stronger prism texture (11-20)[0001]. With regard to the texture processed by annealing after hot rolling, it not only inherits the texture of the initial hot-rolled plate (-2021)[10-15], but also forms new textures (-1013)[1-210] and (11-25)[1-123]. After cold rolling with a reduction of 60%, the typical cold-rolled texture (1 1 -2 5)[1-100], which is the common texture type for the pure titanium plate, forms with a higher strength. In the case of recrystallization annealing after cold-rolling, the titanium plate not only gives birth to the recrystallization texture (1 0 -1 3)[1 -2 1 0], but also basically inherits the cold-rolled texture(11-25)[1 -100]. As the annealing time increases, and the texture (10-13)[1-210] preferentially forms by consuming the cold-rolled texture (11-25)[1 -100] during the recrystallization process [27].

3.0 Experimental Details

3.1. Material and Sample Preparation

Commercial pure (CP) titanium (Grade-2) of chemical composition shown in table 3.1, was collected in the form of plates. Such CP titanium plates were subjected to cold rolling of 90% reduction in thickness in a laboratory rolling mill. The cold rolling was carried out at IISc Bangalore. The samples of 10mm length X 10 mm width were prepared from the cold rolled titanium plates. The samples were then subjected to isochronal annealing for 30 min at 500°C, 600°C and 700°C to obtain the recrystallization temperature. Then recrystallization was done at 600°C for 10sec, 20sec, 60sec, 2min, 5min, 10min, 20min, and 30min. The recrystallized samples were electro-polished using an electrolyte of methanol and perchloric acid (90:10) at 21V.

Table 3.1 Chemical composition (in wt. %) of CP titanium used in the present study.

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

3.2 Characterization Techniques

3.2.1.1 X-Ray Diffraction (XRD)

XRD was carried out at IIT Bombay in an analytical XRD system. Five different pole figures (0 0 2), (0 0 1), (0 1 2), (0 1 3) and (1 1 4) were measured and subsequent ODF was estimated using an academic software LABOTEX .

3.2.1.2 Data Analysis

First from the XRD data 2theta vs. Intensity graphs were generated by using the software *XpertHighscore* and stacked in increasing order of recrystallization time upward in figure 4.2. It showed the attenuation of some peaks while formations of others with increase in anneal time. Then using the software LABOTEX, ODF images are generated for each annealed sample, then for maximum intensity contours $\{\Phi_1, \Phi, \Phi_2\}$ Euler space $(h k l) \langle u v w \rangle$, volume fraction and

intensity were noted in a tabular form. Maximum intensity vs. annealing time graph is plotted. For identification of new evolving texture a table was made in which recrystallization annealing time, corresponding ODF figure, dominating orientations in ODF and intensity were entered and analyzed for a conclusion.

3.2.2 Electron Backscattered Diffraction (EBSD)

EBSD was carried out in a SEM (scanning electron microscope) at IIT Bombay. EBSD of isochronal annealing samples were measured *only*. Other recrystallized samples were not measured because the statistics of texture measurement is poor compared to XRD texture measurement.

4.0 Results and Discussion

4.1 Recrystallization Temperature

Figure 4.1 shows the microstructure of cold rolled CP-titanium annealed at 500 °C, 600 °C and 700 °C for 30mins. It may be noted this isochronal annealing treatment was done to find out the recrystallization temperature of the CP-titanium used in the present study. The figure clearly shows the sample was fully recrystallized at 600 °C annealing temperature (figure 4.1b). 500 °C annealed sample showed under recrystallized and 700 °C showed substantial grain growth.

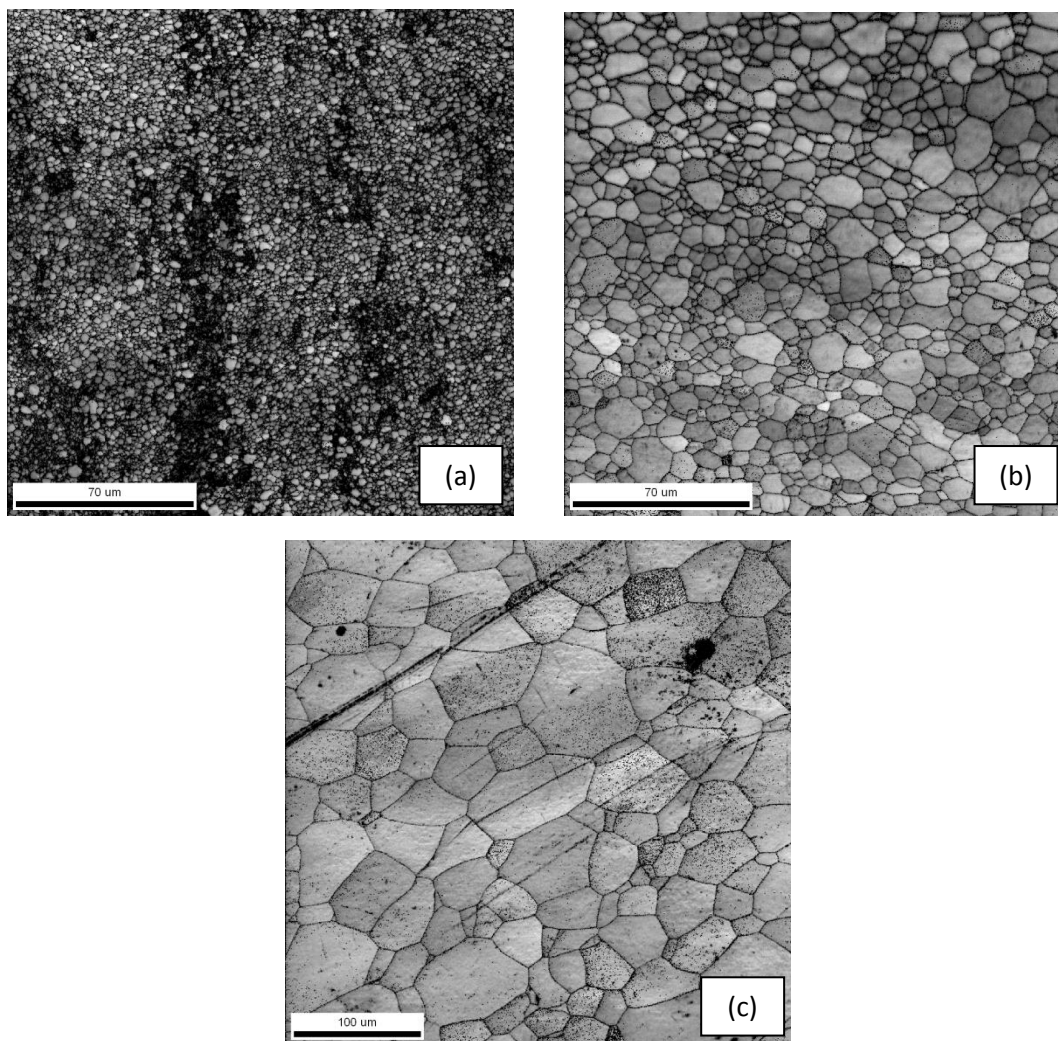


Figure 4.1 Microstructure of CP-titanium under isochronal annealed for 30mins – (a) 500 °C, (b) 600 °C and (c) 700 °C. This was obtained by EBSD analysis.

4.2 Texture Determination

Figure 4.2 shows the XRD peak profile of different annealing samples. The XRD peak profile qualitatively shows the texture development in a material [28]. The figure showed that a decrease in basal (0002) pole ($2\theta=38^\circ$) intensity and increase in (10-12) ($2\theta=53^\circ$) and (10-13) ($2\theta=71^\circ$) pole. Hence this qualitatively showed texture developments in the samples under progressive annealing treatments.

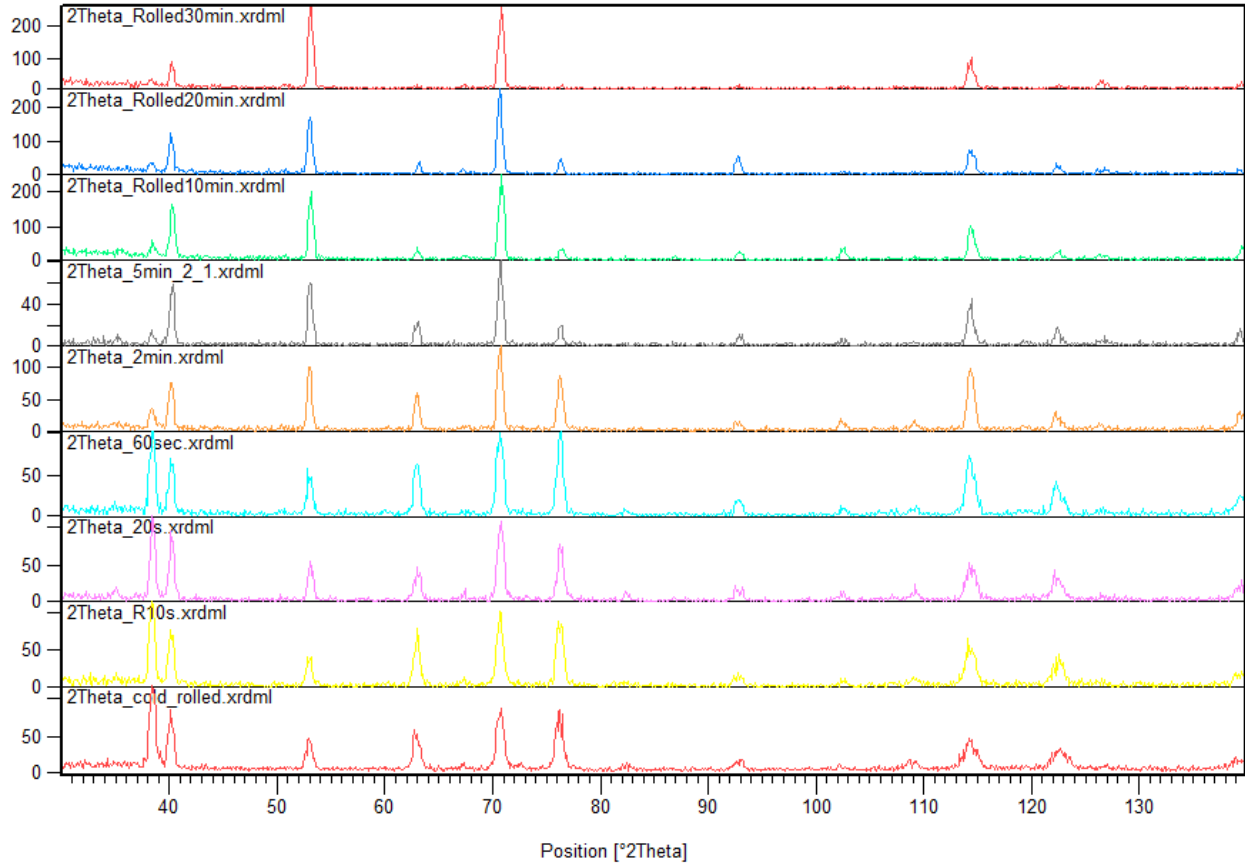
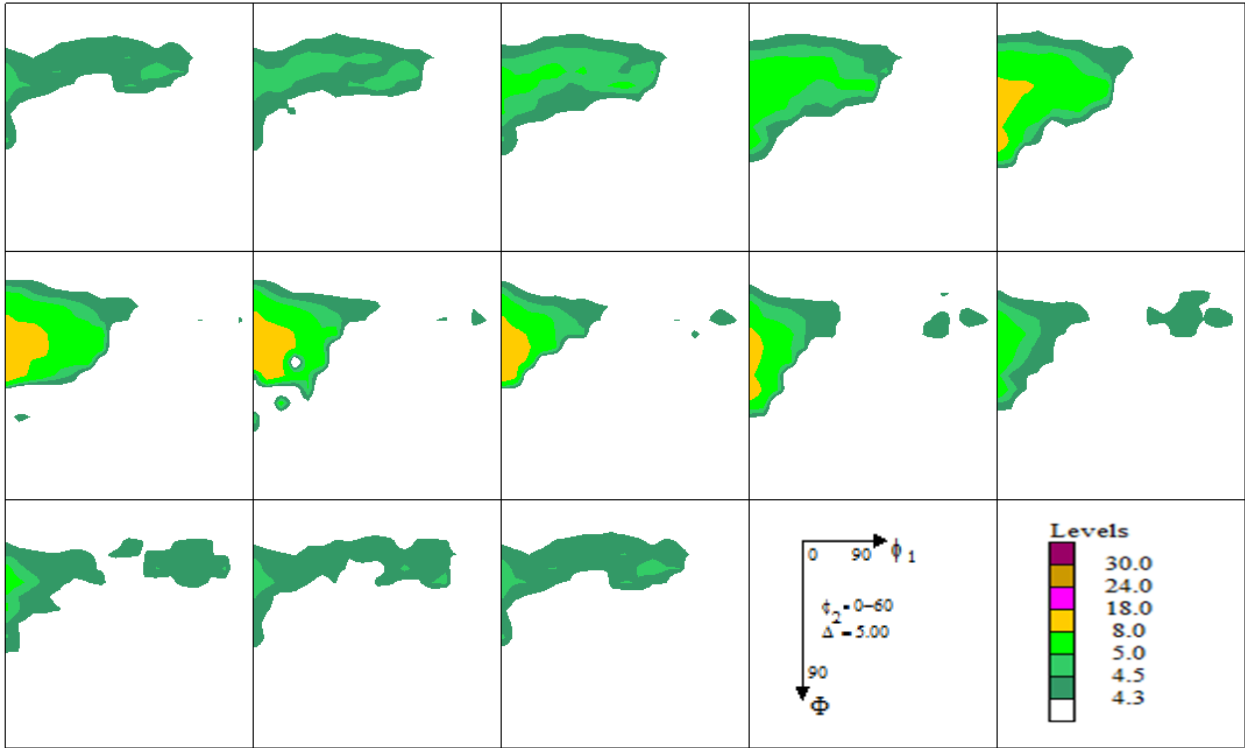
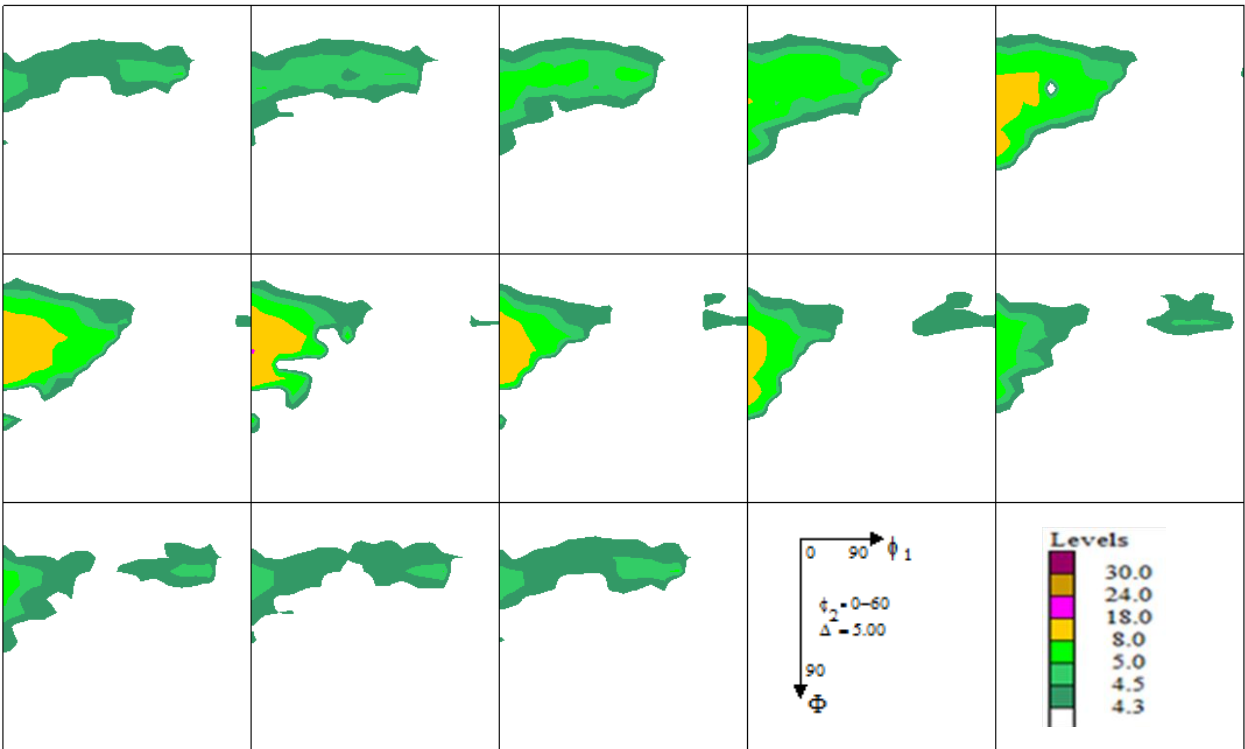


Figure 4.2 XRD peak profile of different annealed samples.

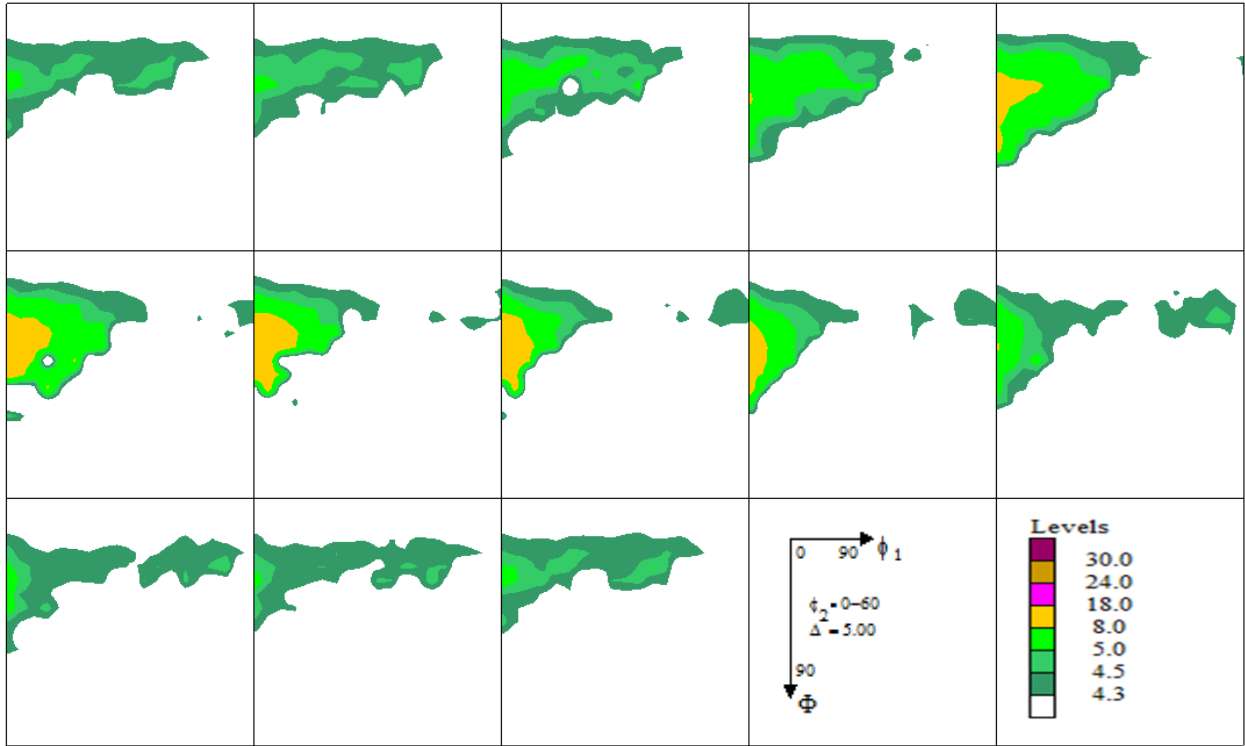
Figure 4.3 shows the ODF plots of cold rolled and annealed samples at constant ϕ_2 section. The major texture developments were tabulated in Table 4.1. The maximum ODF intensity at different annealing time is shown in figure 4.4. The result clear showed the initial deformation texture i.e. $(1\ 1\ -2\ 4)\langle 1\ -1\ 0\ 0\rangle$ has vanished beyond 5mins of annealing time. Similarly, the basal orientation i.e. $(0\ 0\ 0\ 1)\langle 1\ -1\ 0\ 0\rangle$ and non-basal orientations i.e. $(2\ 1\ -3\ 7)\langle 1\ -2\ -1\ 0\rangle$, $(3\ 1\ -4\ 9)\langle 2\ -15\ 13\ 1\rangle$ and $(5\ 1\ -6\ 15)\langle 1\ -5\ 4\ 0\rangle$ were developed at increasing annealing time.



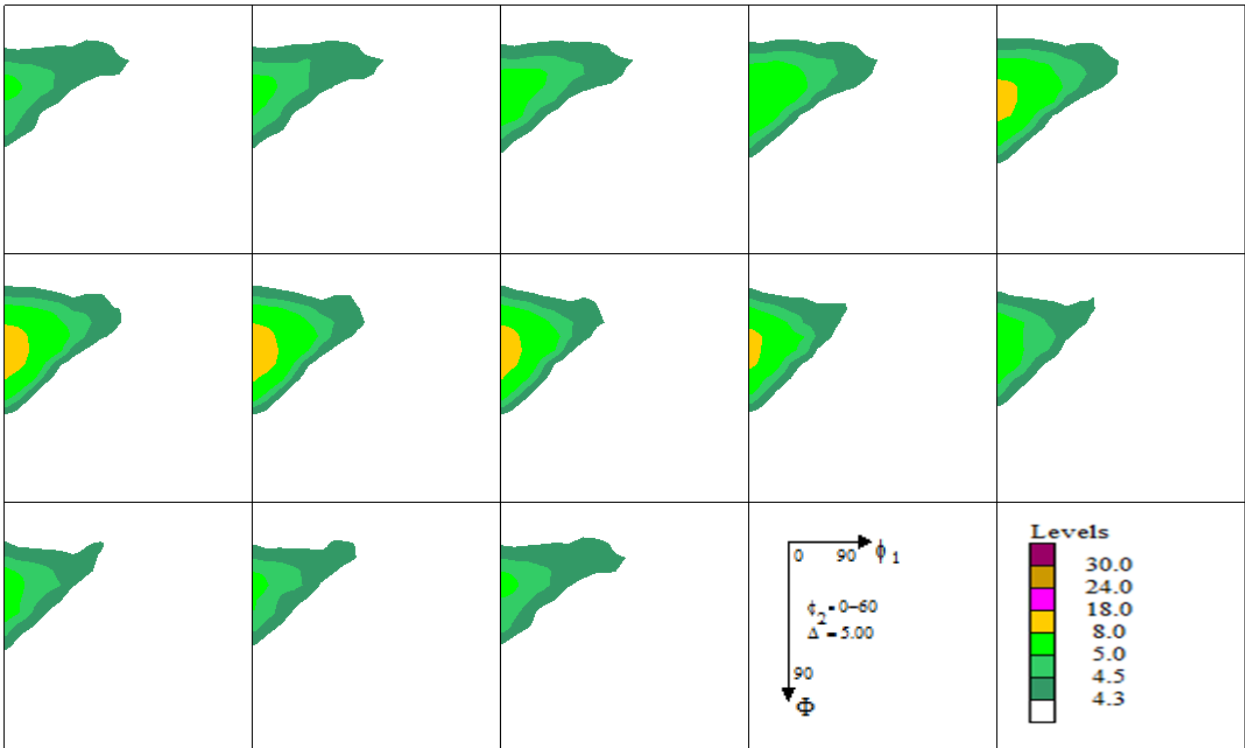
(a)



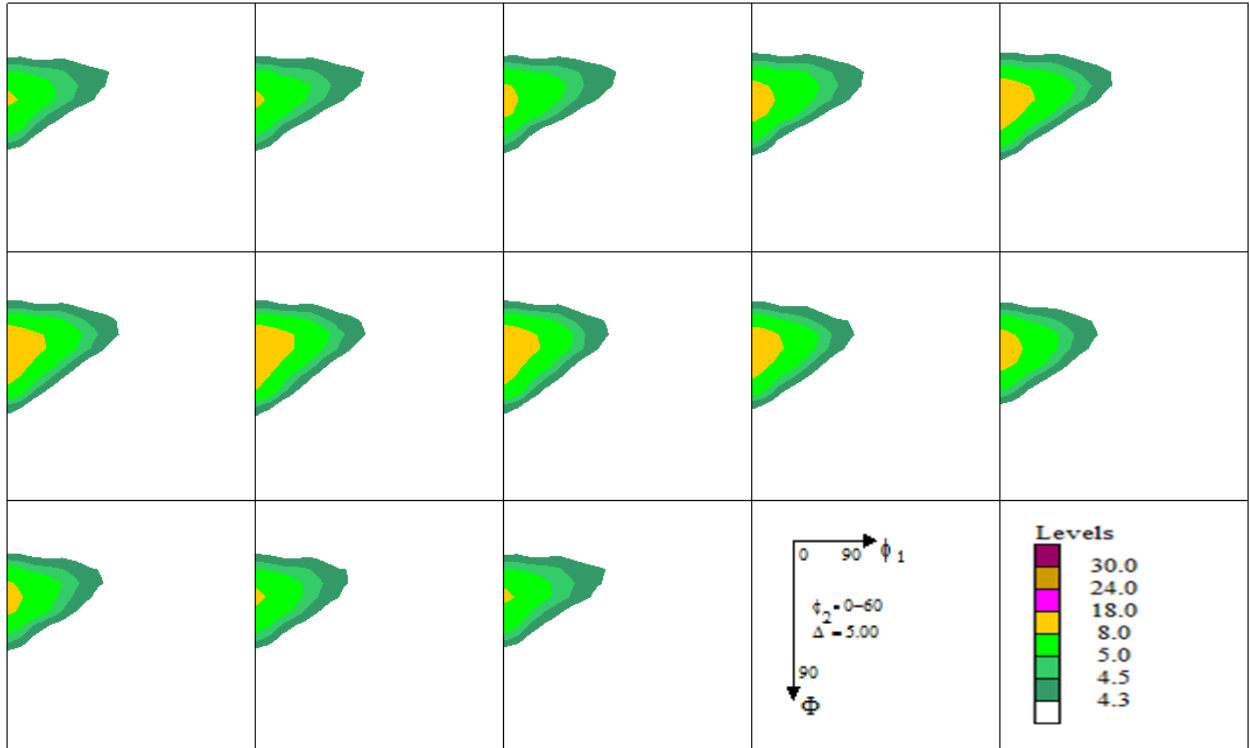
(b)



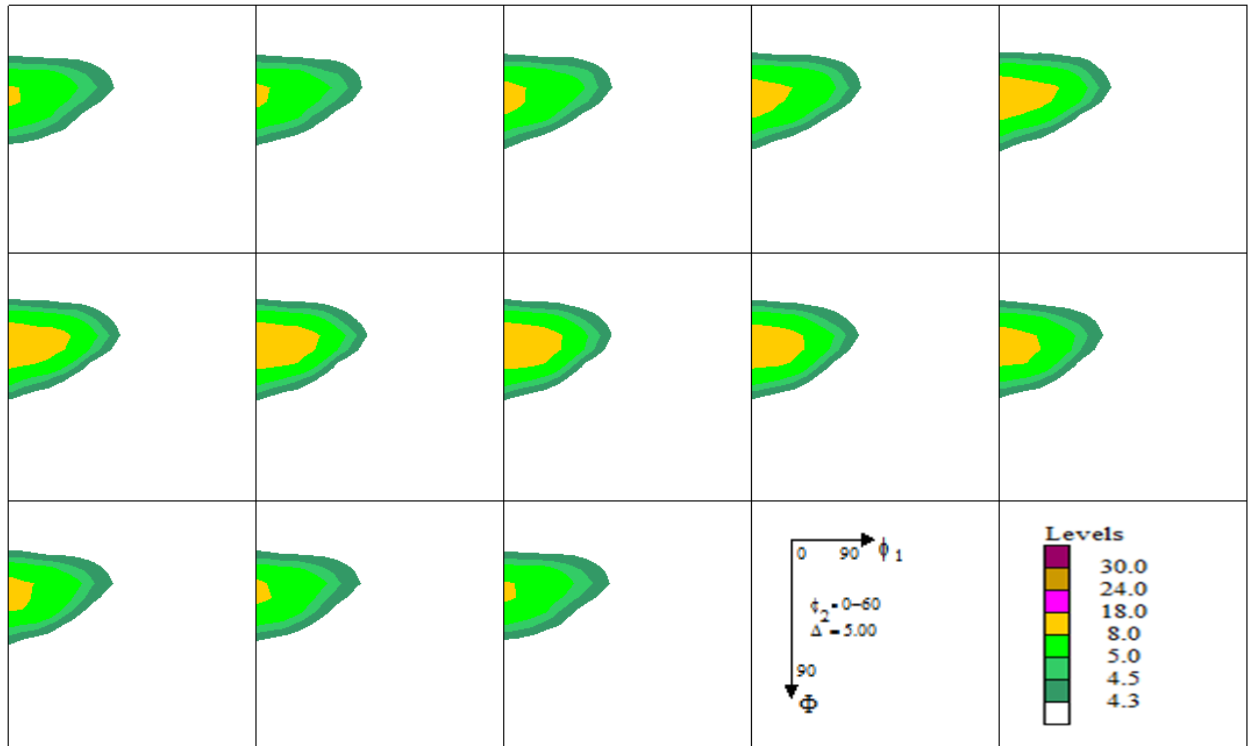
(c)



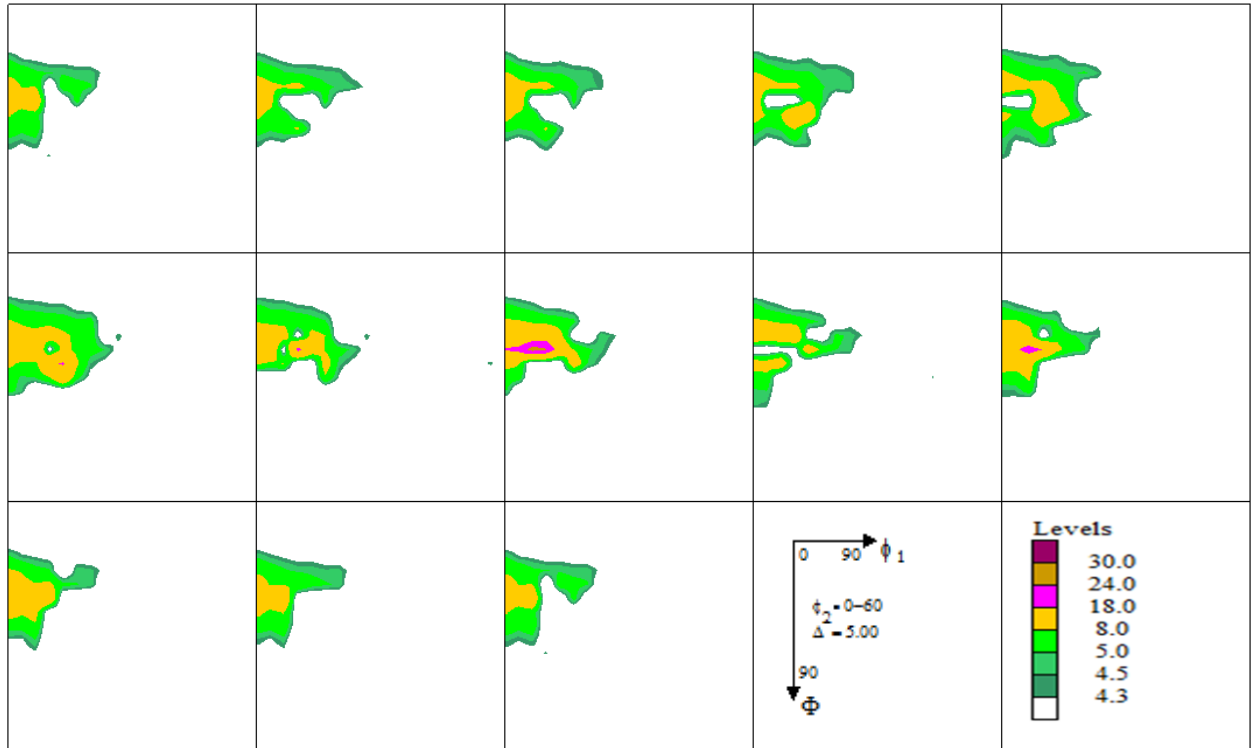
(d)



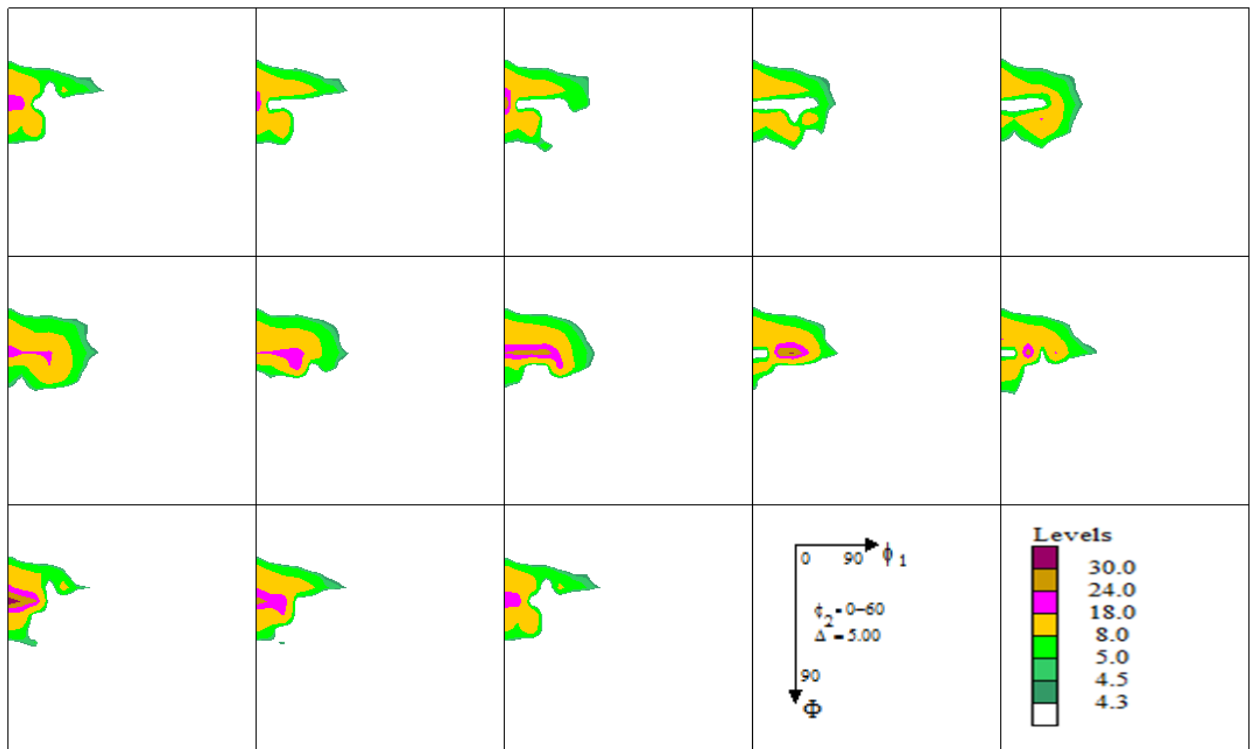
(e)



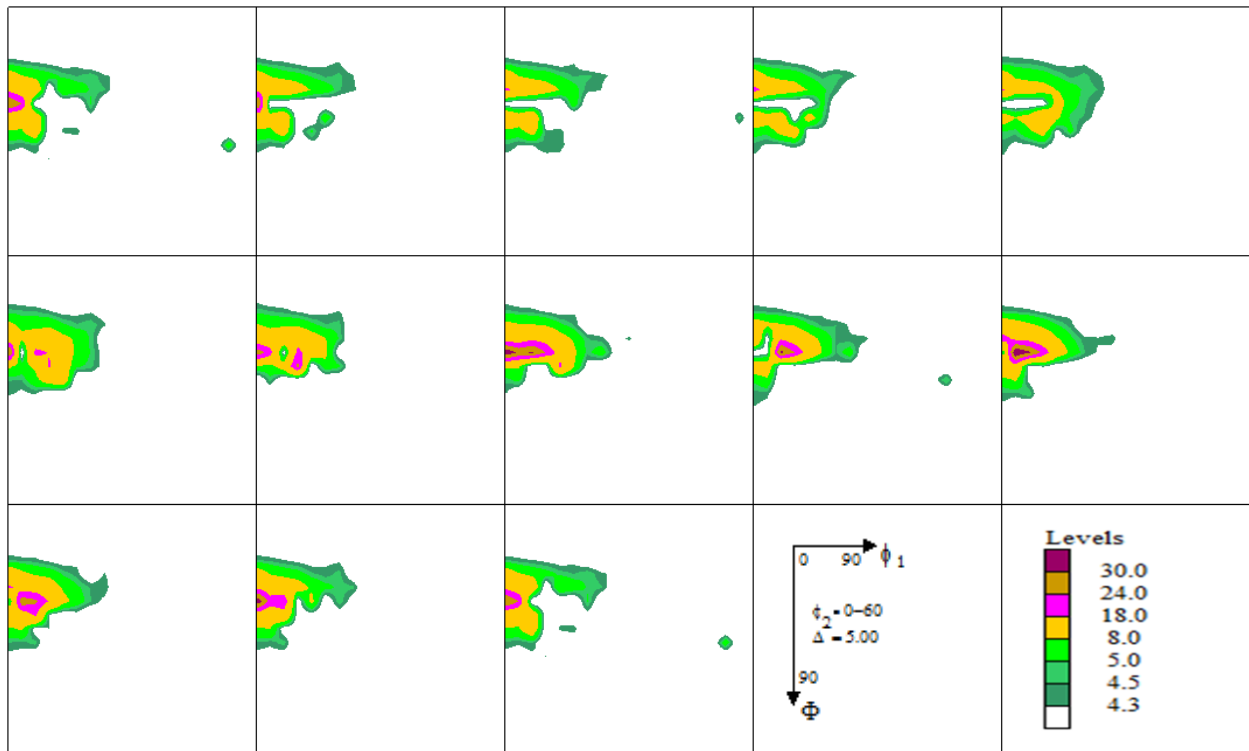
(f)



(g)



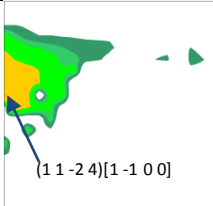
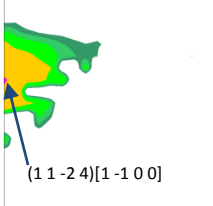
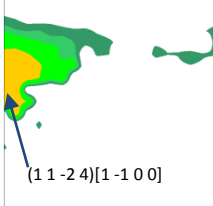
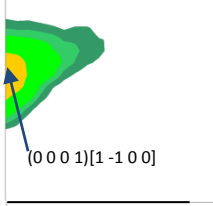
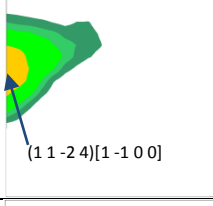
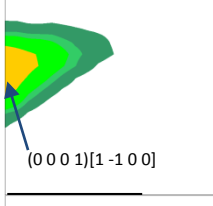
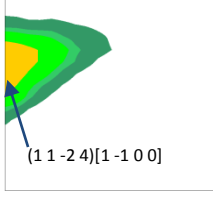
(h)

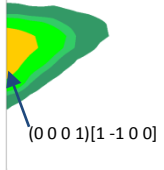
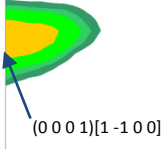
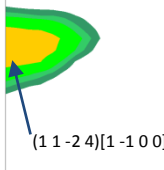
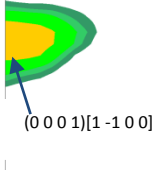
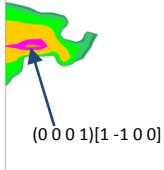
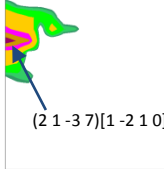
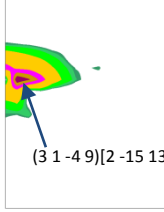


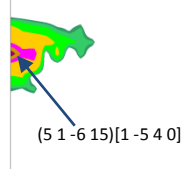
(i)

Figure 4.3 ODF plots for cold rolled and annealed samples at constant ϕ_2 section: (a) Cold rolled, (b) 10sec, (c) 20sec, (d) 60sec, (e) 2min, (f) 5min, (g) 10min, (h) 20min, and (i) 30min. The contour levels are at 30, 24, 18, 8, 5, 4.5 and 4.3 times random.

Table 4.1 Specific orientations developed during annealing of CP-titanium.

SAMPLE	ODF figure	ORIENTATION		INTENSITY F(g)
		ϕ_1, φ, ϕ_2	$(hkl)[uvw]$	
Cold rolled		0 35 30	$(11-24)[1-100]$	14.60
Annealed 10 sec		0 35 30	$(11-24)[1-100]$	17.70
Annealed 20 sec		0 35 30	$(11-24)[1-100]$	14.10
Annealed 60 sec		0 30 25	$(0001)[1-100]$	10.60
		0 35 30	$(11-24)[1-100]$	10.60
Annealed 2 min		0 35 25	$(0001)[1-100]$	11.80
		0 35 30	$(11-24)[1-100]$	11.80

Annealed 2 min	 (0001)[1-100]	0 35 35	(0001)[1-100]	11.80
Annealed 5 min	 (0001)[1-100]	0 35 25	(0001)[1-100]	11.90
	 (11-24)[1-100]	0 35 30	(11-24)[1-100]	11.90
	 (0001)[1-100]	0 35 35	(0001)[1-100]	11.90
Annealed 10 min	 (0001)[1-100]	10 35 35	(0001)[1-100]	23.90
Annealed 20 min	 (21-37)[1-210]	0 35 50	(21-37)[1-210]	30.40
Annealed 30 min	 (31-49)[2-15131]	0 35 45	(31-49)[2-15131]	31.50

Annealed 30 min		0 35 55	(5 1 -6 15) [1 -5 4 0]	31.50
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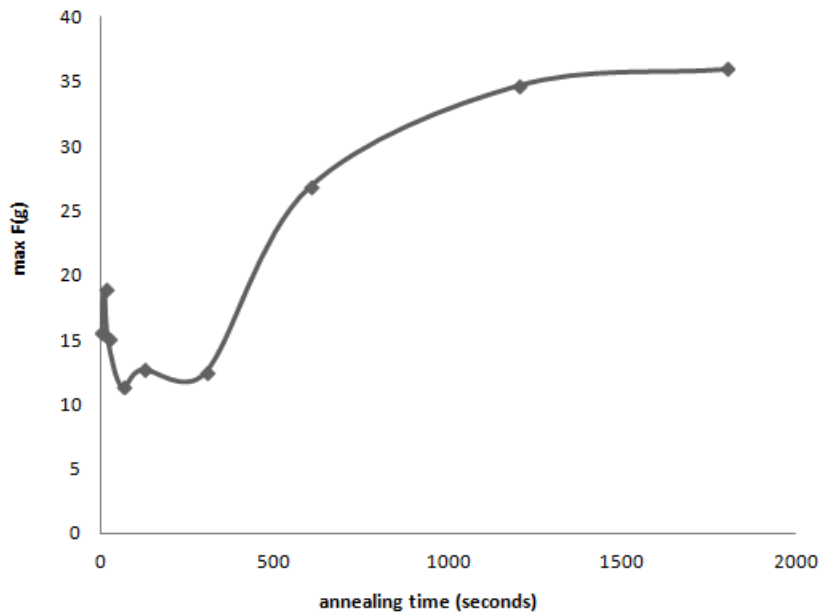


Figure 4.4 Maximum ODF intensity as a function of annealing time.

This plot shows that the initial cold deformation texture gradually decreases which is indicated by the lowering of max $f(g)$ during the initial annealing stages. With increase in annealing time, new recrystallization texture develops which is indicated by the increase in the value of maximum intensity ($f(g)$) after the anneal time increases beyond 5 minutes.

Conclusions:

1. Cold rolled (90% reduction) CP-Titanium sheets undergo complete recrystallization at temperature around 600°C as clearly evident from EBSD microstructure images.
2. ODF results clearly showed that the initial deformation texture i.e. $(1\ 1\ -2\ 4)\langle 1\ -1\ 0\ 0\rangle$ has vanished beyond 5mins of annealing time. Similarly, the basal orientation i.e. $(0\ 0\ 0\ 1)\langle 1\ -1\ 0\ 0\rangle$ and non-basal orientations i.e. $(2\ 1\ -3\ 7)\langle 1\ -2\ -1\ 0\rangle$, $(3\ 1\ -4\ 9)\langle 2\ -15\ 13\ 1\rangle$ and $(5\ 1\ -6\ 15)\langle 1\ -5\ 4\ 0\rangle$ were developed at increasing annealing time.

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