Shear Mechanisms During Cryogenic Treatment of Ti6Al4V

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ABSTRACT

Cryogenic treatment has been widely accepted as a method to improve wear properties of tool steel. However, very limited knowledge can be found in the literature regarding its mechanism and application to other metals. This paper describes shear effects during cryogenic treatment on Ti6Al4V alloy compared with cold rolling process. Cryogenic treatment was conducted by heating the alloy up to 950°C followed with cooling process using liquid nitrogen spraying. Optical and scanning electron microscope observation show that elongated primary α phase and shear band width below 4 µm were observed in both cryogenic and cold rolled sample. However both elongated α phase structure and shear band width are finer in the cold-rolled sample which is an indication that shear effect is higher in this sample. In addition, as observed by X-ray diffraction peak calculation using Mud-Master computer program, movement of atom during cryogenic treatment is more intense in the direction parallel with the basal plane, which is the slip plane for hexagonal α phase of titanium alloy. From the above observations it can be concluded that cryogenic treatment on Ti6Al4V has a strong relation with the mechanism of plastic deformation in general.

Keywords

Cryogenic treatment, shear mechanism, plastic deformation, titanium alloy

1. INTRODUCTION

The relative amounts of phases in the microstructure can be adjusted by varying heating temperature, deformation process, and cooling rate [1]. Heat-treatment alone or combination between heat-treatment and deformation process, which is known as thermo-mechanical treatment, can be a useful method to improve properties of metal by microstructure improvement. Cryogenic treatment is one of the important methods that can be used to improve the properties of metal. Cryogenic treatment is commonly used after heat treatment as a panacea for incomplete transformation of austenite in steel, but there may be reason to consider cryogenic treatment not as a fix for improperly processed parts but as a standard step in the heat treatment process [2]. The ability of cryogenic treatment to complete martensitic transformation is considered due to the characteristics of this structure that becomes more supersaturated with decreasing temperature [3].

Although this method has been widely used in tool steel application, especially to improve wear characteristic of this alloy [4], very limited literature can be found on the detail study of this method, especially on the implementation of this method to other metals. Therefore study on the cryogenic treatment of Ti6Al4V has been conducted in the present research. Ti6Al4V is a well-known titanium alloy that has been widely used in air-craft industry due to its high strength to weight ratio and the ability of this metal to be deformed superplastically [5]. Another inspiration for the present research is from the phenomena of transformation induces plasticity (TRIP) steel, where plastic deformation can be used to transform retained austenite to martensite structure of water quenched high-carbon steel [6]. The similar mechanism between plastic deformation and cryogenic treatment, where shear process occurs during both process [7, 8], is considered to play important role for the martensitic transformation to take place after both processes. In the present paper, comparison study between cryogenic treatment and plastic deformation on Ti6Al4V is discussed based on the optical and scanning electron microscope observation and structure parameter calculation of XRD peak using Mud-Master computer program.

2. EXPERIMENTAL WORKS

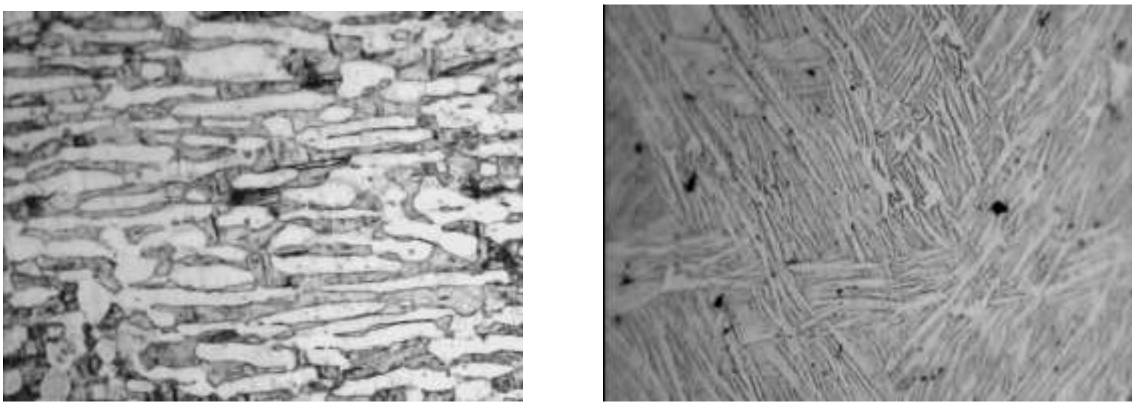
Initial Ti6Al4V alloy was prepared through heat-treatment at dual phase temperature (950^{o}C) and cooling in the air medium. Cryogenic treatment was conducted through heating process at dual phase temperature (950^{o}C) followed with cooling process by liquid nitrogen spraying. Cold rolling process was conducted on 10 mm thickness of Ti6Al4V alloy with 50% reduction in thickness.

Study on the microstructure of cryogenic and cold-rolled sample was conducted by optical and scanning electron microscope observation. In order to study crystal structure of both processes X-ray diffraction study was conducted after the process. This characterization was conducted with analytical step size of 0.02^o two-theta.

Crystal structure parameter was calculated using Mud-Master computer program based on the calculation of diffraction peak from X-ray diffraction data. Calculation of X-ray diffraction peak is possible to be done since X-ray diffraction peak can be treated as Fourier series, therefore Fourier analysis can be done as the start point for crystal structure parameter measurement by Mud-Master method. Calculation of X-ray diffraction peak using Mud-Master program involving several step, such as choosing the XRD peak to be analyzed, removing background, removing $K\alpha 2$ component of radiation, performing Fourier analysis of interference function maximum and correction Fourier coefficient for symmetrical strain. This method has been previously success in the measurement of crystal parameter of clay material including strain and crystal size measurement [9]. The theory for the operation of Mud Master program is described by Drits *et al.* [10]. One point to be noted here is crystal structure parameter obtained in the present research is not the absolute value for the alloy structure and only suitable as estimation behavior of material substructure under different treatment.

3. RESULTS AND DISCUSSION

Figure 1 shows microstructure of cold rolled sample, as representation of plastic deformation sample, and cryogenic treated titanium alloy that has been solution treated in the air before the process. Both of the processes produce an elongated primary α structure (light particle), however much finer structure is found in the cold rolled sample. This condition suggests us that both of the processes give a deformation effect on the alloy and this effect is greater in the cold rolled sample than cryogenic treated sample. Deformation load during cold rolling process is considered to induce shear stress inside the alloy, whereas the military movement of highly disciplined and coordinated movement of atom during cryogenic treatment results in the similar effect on the alloy.

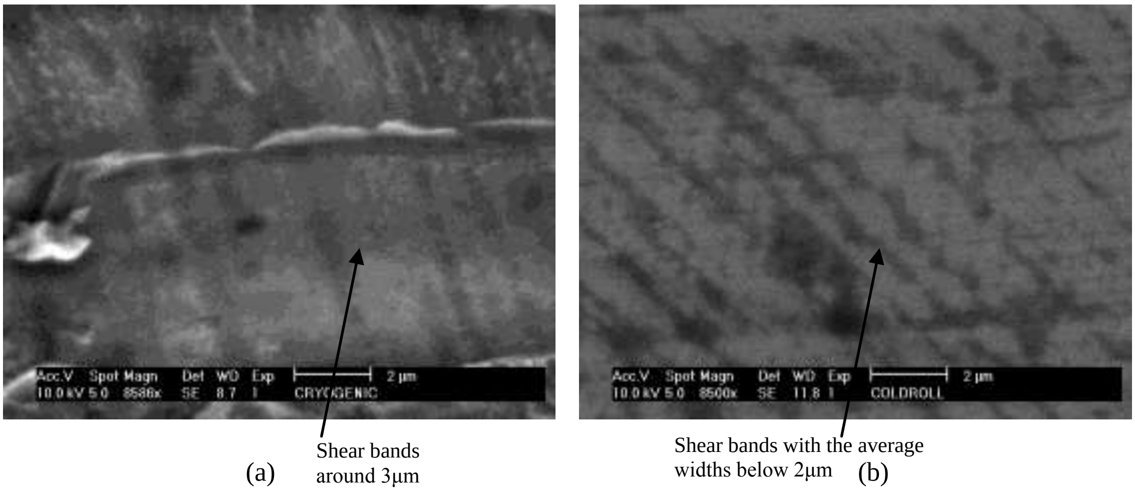


(a) (b)

Figure 1: Microstructure of cold rolled sample (a) and cryogenic treated sample (b)

Another interesting feature is shown in the Figure 2 that shear bands was observed in both cryogenic treated and cold rolled samples. The appearance of shear bands is associated with the onset of plasticity [11] and thermal softening in the material [12]. On the other hand, the width of shear bands is influenced by the degree of plastic deformation [13] and also defects in the material [14]. Repetition of similar degree of deformation was also found to decrease the width of shear band, such as repeating similar parameter process of equal channel angular pressure (ECAP) up to four passes significantly decrease the width of shear band [13]. Decreasing shear band width is important in superplastic deformation process since plastic deformation becomes more intense inside the shear band [14]. Therefore plastic deformation will be easier if the width of shear band is small during the process. From the previous research [13], successful superplastic deformation process was found for the sample with the shear band width around 4-5μm.

From Figure 2 (a) and (b) it can be seen that lower width of shear bands found in the cold-rolled sample than the cryogenic treated sample. This is an indication that higher degree of plastic deformation is produced in the cold rolled sample compared to cryogenic treated sample. In addition shear bands width that is resulted from both processes has the comparable value with the successful condition for superplastic deformation (below 4 μm) as suggested in the previous research (14). This condition suggest us that both cryogenic treatment and cold rolling process on the air cooled titanium alloy, produce properties of the alloy that is suitable to be treated for superplastic deformation process.



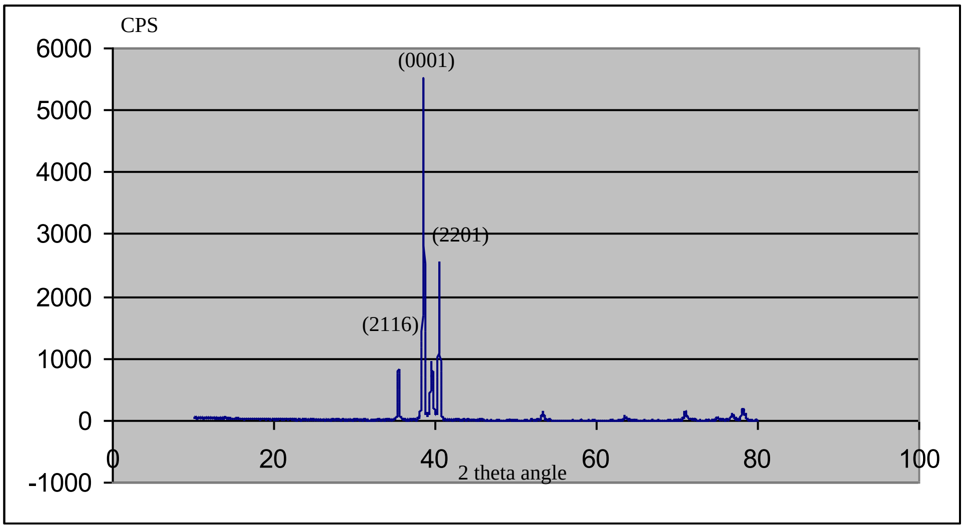
(a) (b)

Figure 2: Shear band after cryogenic treatment (a) and cold rolling process (b) of initial air cooled sample

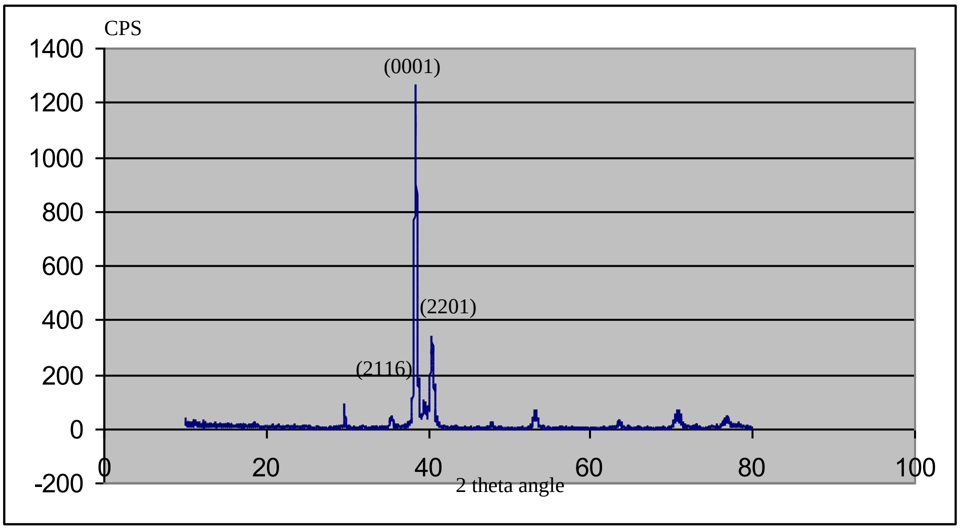
On the other hand, shear mechanism by cold rolling process conducted in this research does not contribute to the martensitic transformation. It is due to the initial condition alloy before the process in the air cooled condition does not have β phase structure. The similarity of the X-ray diffraction pattern shown in the Figure 3 (a) and (b) between initial air cooled and cold rolled samples confirm this fact. There are not appearance important peaks for martensite phase such as (1120) plane after cold rolling process. However decreasing intensity was found in this sample, which is considered due to straining effect after the process. This condition is also confirmed from strain measurement conducted by Mud-Master computer program as is shown in the Table 1, 2 and 3 that certain level of strain was found after cold rolling process.

A different situation was found for cryogenic treatment sample that shear mechanism during this process contribute to the martensitic transformation. Figure 3 (c) shows the appearance of several important peaks for martensite phase, such as (1120) plane at 2θ angle of 63.2. This plane is formed from the movement of atomic plane of (111) in the β phase [15]. Shear mechanism during cryogenic treatment also induce crystallographic strain on the primary α phase plane as can be seen in the Table 2 and 3, that certain level of strain are found in the (2201) plane and (2116) plane. However strain does not occur on (0001) plane which is the slip plane for hexagonal structure (Table 1). This observation agrees with the slip theory for titanium alloy that since the slip vectors are parallel to the basal plane (0001), if stress applied in the direction of perpendicular to this plane, there will be no critical resolved shear stress acting in this plane [15]. The difference on the strain value is also influenced by the habit plane and orientation relationship plane of the previous β phase structure [1].

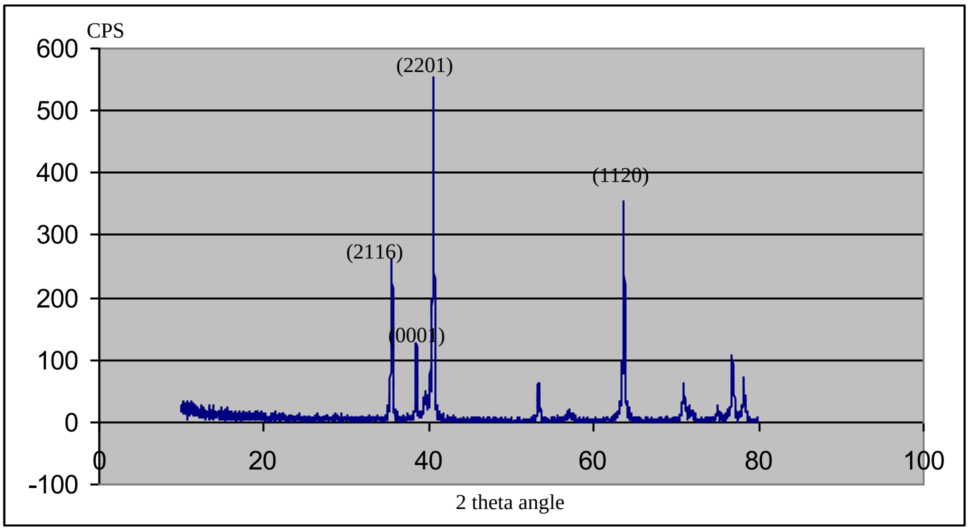
In addition as can be seen in the Table 1, certain degree of strain is found in the cold rolled sample for (0001) plane. Higher degree of deformation and shear stress as also confirmed from the micrograph in the Figure 3 is considered to responsible for this condition. It can also be understood that no stress condition on (0001) plane for cryogenic treated sample is due to movement limitation of atom during this process that only possible in a small number of atomic distances during this fast cooling process [1].



(a)



(b)



(c)

Figure 3: X-ray diffractograph of initial sample (a) cold rolled sample (b) and cryogenically treated sample (c)

Table 1: Stress and crystallographic parameter at plane (0001)) for cold rolled and cryogenic treated sample

Parameter	Air cooled	Cryogenic treated	Cold rolled sample
POSITION INT. FN. (two-theta)	38.55	38.47	38.31
d-SPACING INT. FN. (Å)	2.335	2.340	2.349
ROOT MEAN SQUARE STRAIN		0.000	0.138

In order to give better understanding for the shear mechanism and atomic movement after cryogenic treatment, Figure 4 illustrate the condition of crystallographic plane in the hexagonal α before and after the process. From Figure 4 (b) it can be seen that in the basal plane (0001) increasing *d* spacing is found after cryogenic treatment as measured by Mud-Master computer program. However movement of atom in the direction parallel with the basal plane is considered to be higher since there is also diffraction angle decrement after the process (Table 2). It is only possible if atomic movement in the *C*-axis direction is accompanied with a higher atomic movement in the direction of parallel with basal plane. Higher movement of atom in the direction parallel with the basal plane can be understood since it is the slip plane of the hexagonal crystallographic of this alloy [15]. With the assumption that atomic movement in the *C*-axis direction is symmetrical that movement of atom in the basal plane and the top plane is the same at 0.0025 À, movement of atom in the direction parallel with basal plane can be measured as 0.0143 À.

Table 2: Stress and crystallographic parameter at plane (2201) for cold rolled and cryogenic treated sample

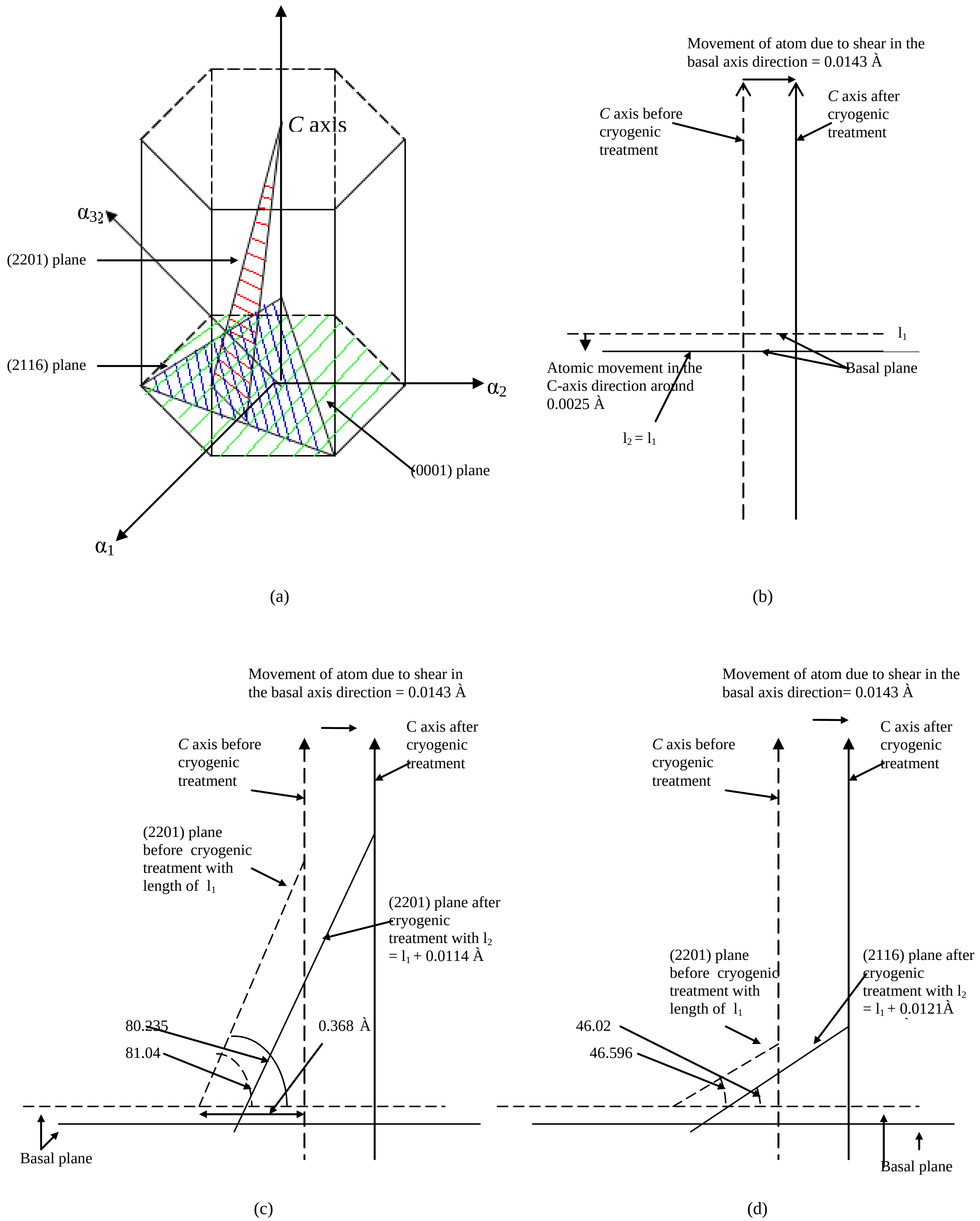
Parameter	Air cooled	Cryogenic treated	Cold rolled sample
POSITION INT. FN. (two-theta)	40.49	40.55	40.45
d-SPACING INT. FN. (Å)	2.228	2.225	2.230
ROOT MEAN SQUARE STRAIN	-	0.107	0.165

Table 3: Stress and crystallographic parameter at plane (2116) for cold rolled and cryogenic treated sample

Parameter	Air cooled	Cryogenic treated	Cold rolled sample
POSITION INT. FN. (two-theta)	35.45	35.51	35.55
d-SPACING INT. FN. (Å)	2.532	2.528	2.525
ROOT MEAN SQUARE STRAIN	-	0.110	0.235

On the other hand, as can be seen in the Figure 4 (c), movement of atom in the direction parallel with the basal plane and in the *C*-axis direction result in the lower angle of (2201) plane that also induce straining effect on this plane as also measured by Mud master computer program (Table 2). Lower *d* spacing (Table 2) is also found on this plane as a consequence of this atomic movement. Similar condition is expected to occur on the (2216) plane that the atomic movement in these directions induces lower angle of (2201) plane that also results in the straining effect on this plane (Figure 4 (d)). Lower *d* spacing is also found in this plane as a consequence of this atomic movement.

In addition if we take into account the perspective length of (2201) plane in the *C*-axis direction of cryogenic treated as 2.34 À, by calculation tangential value for 80.235 (figure 3.4 (c)), we get the perspective length of (2201) plane in the basal plane direction as 0.403À. Since the perspective length of (2201) plane in the *C*-axis and basal plane direction for initial air cooled sample is 2.335 À and 0.368 À respectively, strain in each direction can be calculated as 0.005 À and 0.035À respectively. Taking into account the elastic modulus value of titanium alloy for *C* axis direction is 145 GPa [15] and we consider the process is still in the area of linier relation of stress-strain, therefore we get shear stress value in this direction as 72.5 Pa. In the similar way, shear stress in the basal plane of this alloy can be obtained by multiplying elastic modulus in this plane, 99.5GPa [15] with its strain value, which is 0.035À, and we get the shear stress value for this direction as 348.25 Pa. This calculation agrees with the previous hypothesis [1] that lower shear stress will be observed in the *C*-axis direction than in the basal plane direction which is the slip plane of this hexagonal crystal structure. This observation also agrees with another hypothesis [16] that the resolved shear stress, which drives dislocation in a crystal, is strongly orientation dependent.



(a) (b)

(c) (d)

Figure 4: Crystallographic plane of titanium alloy (a) and two dimension movement of the plane after the alloy treated by cryogenic treatment for (0001) plan (b), (2201) plane (c) and (2116) plane (d)

4. CONCLUSION

Shear mechanism takes place during cryogenic treatment of titanium alloy as can be observed from optical and scanning electron microscope observation. However shear effect in the cryogenic treated sample is lower than in the cold-rolled sample. The appearance of shear band width below 4μm in both cryogenic treated and cold-rolled sample is an indication that the treated alloy has been appropriately treated for the application of superplastic deformation process. Based on the X-ray diffraction characterization and calculation of X-ray diffraction peak by Mud master computer program, displacement of plane during cryogenic treatment has been successfully proposed and confirmed the previous theory and hypothesis on the atomic movement during martensitic transformation. Movement of atom in a short distance and well arrangement is found after cryogenic treatment, which is the characteristic of military movement, typical for martensitic transformation. In addition, the movement of atom is more intense in the direction parallel with the basal plane which is the slip plane for hexagonal α phase of titanium alloy. This observation and also the appearance of shear band width after cryogenic treatment are indication that this process has a typical mechanism for plastic deformation in general.

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