

Effect of Microstructural Parameters on
Mechanical Properties and Fracture in
 $\alpha + \beta$ Titanium Alloy

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Effect of Microstructural Parameters on Mechanical Properties and Fracture in $\alpha + \beta$ Titanium Alloy

$\alpha + \beta$ 型チタン合金の機械的性質と破壊に及ぼす
微視組織の影響



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CHAPTER 1

BACKGROUNDS AND PURPOSE	1
<i>1.1. General properties of titanium and titanium alloys</i>	<i>1</i>
<i>1.2. Classification of titanium alloys.....</i>	<i>4</i>
<i>1.3. Microstructure and mechanical properties of $\alpha + \beta$ titanium alloys</i>	<i>7</i>
1.3.1. Equiaxed and bi-modal microstructures	8
1.3.2. Lamellar microstructures	9
<i>1.4. Bi-lamellar microstructure, and motivation and objective of the present study</i>	<i>11</i>
<i>1.5. Outlines of dissertation.....</i>	<i>16</i>

CHAPTER 2

THERMOMECHANICAL PROCESSING FOR COLONY SIZE REFINEMENT OF LAMELLAR AND BI-LAMELLAR MICROSTRUCTURES	20
<i>2.1. Introduction</i>	<i>20</i>
<i>2.2. Experiment procedure</i>	<i>22</i>
2.2.1. Fabrication of lamellar microstructures with various colony sizes	22
2.2.2. Fabrication of bi-lamellar microstructures with various colony sizes	27
2.2.3. Microstructure characterization	28
<i>2.3. Results and Discussion</i>	<i>30</i>
2.3.1. Colony size refinement in lamellar microstructures through simple deformation process.....	30
2.3.2. Colony size refinement in lamellar microstructures through multiple deformation process.....	34
2.3.3. Crystallographic features in lamellar microstructure.....	36

2.3.4. Colony size refinement in bi-lamellar microstructures.....	40
2.3.5. Crystallographic features in bi-lamellar microstructure	42
2.4. Conclusions	46
References	49

CHAPTER 3

MECHANICAL PROPERTIES OF LAMELLAR AND BI-LAMELLAR MICROSTRUCTURES WITH DIFFERENT COLONY SIZES51

3.1. Introduction	51
3.2. Experiment procedure	52
3.2.1. Materials	52
3.2.2. Nano indentation hardness test	53
3.2.3. Tensile test.....	55
3.3. Results and discussion	56
3.3.1. Nano-hardness of each phase in lamellar and bi-lamellar microstructures	56
3.3.2. Tensile properties in lamellar microstructures with different colony sizes.....	59
3.3.3. Tensile properties in bi-lamellar microstructures with different colony sizes	63
3.4. Conclusion	68
References	71

CHAPTER 4 72

CHARACTERISTICS OF CRACK FORMATION IN LAMELLAR AND BI-LAMELLAR MICROSTRUCTURES WITH VARIOUS COLONY SIZES ... 72

4.1. Introduction	72
4.2. Experiment procedure	73

4.2.1. Materials	73
4.2.2. Micro-crack observation	73
4.2.3. Main-crack characterization.....	75
4.2.4. Micro-DIC technique in microstructure.....	76
4.3. Results and discussion	77
4.3.1. Characterization of micro-crack formation in lamellar microstructure	77
4.3.2. Characterization of main-crack formation in lamellar microstructure.....	80
4.3.3. Relationship between crack propagation and deformation behavior in lamellar microstructure	84
4.3.4. Characterization of micro-crack formation in bi-lamellar microstructure.....	87
4.3.5. Characterization of main-crack formation in bi-lamellar microstructure	90
4.3.6. Relationship between crack propagation and deformation behavior in bi-lamellar microstructure	93
4.3.7. Effect of colony size on mechanical properties and crack propagation in lamellar and bi-lamellar microstructures	97
4.4. Conclusion	99
References	103
 CHAPTER 5	
FRACTURE PROPERTIES IN VARIOUS MICROSTRUCTURES WITH DIFFERENT GRAIN SIZES OR COLONY SIZES	104
5.1. Introduction	104
5.2. Experiment procedure	105
5.2.1. Fabrication of equiaxed microstructures with various grain sizes.....	105
5.2.2. Measurement of fracture properties in various microstructures	107
5.2.2.1 Three-point bending test	107

5.2.2.2 <i>Calculation of J-integral</i>	107
5.2.2.3 <i>Crack length measurement for R-curve</i>	109
5.3. Results and discussion	111
5.3.1. Load-displacement curve obtained by three-point bending tests in various microstructures	111
5.3.2. Effect of microstructural parameters on fracture toughness, J-integral, in various microstructures	113
5.3.3. Effect of microstructural parameters on crack growth resistance in various microstructures (R-curve)	116
5.4. Conclusion	119
 CHAPTER 6	
SUMMARY AND CONCLUSION	123
ACKNOWLEDGEMENT	130

CHAPTER 1

BACKGROUNDS AND PURPOSE

1.1. General properties of titanium and titanium alloys

Titanium is a transition metal having electrical and magnetic properties of atomic number 22 located on group 4B and period 4 in the periodic table. Physical properties of high purity-titanium are summarized in **Table 1.1** [1]. Melting temperature of titanium ($\approx 1668\text{ }^{\circ}\text{C}$) is higher than that of iron and steels ($\approx 1536\text{ }^{\circ}\text{C}$), but the thermal expansion coefficient and thermal conductivity is lower than that of iron and steels. Titanium and titanium alloys are known as relatively high-cost metallic materials due to not only the difficulties of a smelting process but also the necessity to adopt advanced technologies for manufacturing. Since titanium and titanium alloys possess high reactivity to interstitial elements (i.e., oxygen, nitrogen, carbon and hydrogen), manufacturing high-quality titanium and titanium alloys needs advanced technologies. Nevertheless, the demands of titanium and titanium alloys have gradually increased because of their superb mechanical properties. High strength, low density, and excellent corrosion resistance of titanium and titanium alloys are attractive for using in a variety of applications such as aircraft (high specific strength, **Figure 1.1**), aero-engines (high strength, low density, and good creep resistance up to approximately $550\text{ }^{\circ}\text{C}$), biomedical devices (corrosion resistance and biocompatibility), and components in chemical processing equipment (corrosion resistance) [2]. Characteristics of titanium and titanium alloys considered the most important in industrial fields are summarized as follows [3, 4]:

- 1) The density of pure titanium is 4.51 g/cm^3 , which is approximately 60 % lighter than that of steels, and 1.6 times heavier than that of aluminum alloy (2.71 g/cm^3). Since specific strength of titanium alloys at low and medium temperatures (up to $\sim 600 \text{ }^\circ\text{C}$) is relatively high, it is favorable to use in aerospace and vehicles industries.
- 2) Thermal expansion coefficient is $2/3$ lower than carbon-steel and $1/2$ lower than stainless steel. Since the thermal expansion coefficient is relatively lower, it is favorable to design structural components used in high temperature environment with considering the dimensional stability.
- 3) Elastic modulus ($\approx 110 \text{ GPa}$) and shear modulus ($\approx 44 \text{ GPa}$) is significantly lower than most metallic materials. Since the titanium has an HCP structure at room temperature, the elastic modulus varies ranging from 100 GPa to 145 GPa depending on deformation texture. It is advantageous to use as an implant material to replace human bones due to the low elastic modulus.
- 4) Titanium is unsurpassed in corrosion resistance among metallurgical materials due to the dense oxidation layer which formed on the surface of the titanium. The dense oxidation layer not only has a large corrosion inhibitory effect into the material but also instantly re-formed in the air even if it is destroyed. Especially, titanium is strongly advantageous in utilizing marine constructions, chemical processing equipment in extreme environment and biomedical industry due to its strong corrosion to chlorine ion.

Table 1.1 Physical properties of high-purity titanium (> 99.9 %) [1].

Structure prototype	Mg
Space symbol	P6 ₃ /mmc (194)
Atomic weight [g/mol]	47.867
Density [g/cm ³]	α phase: 4.507, β phase: 4.35
β transus temperature [°C]	882 $\pm$ 2
Melting temperature [°C]	1.668 $\pm$ 10
Boiling temperature [°C]	3,260 (estimated)
Lattice parameters [nm], c/a ratio	a = 0.295, c = 0.468, c/a = 1.587
Thermal expansion coefficient [10 ⁻⁸ /K]	836 (at 20 °C), 10.1 (at 1,000 °C)
Thermal conductivity [W/mK]	14.99
Specific heat capacity [J/kgK]	523
Elastic modulus [GPa]	115
Shear modulus [GPa]	44
Poisson's ratio	0.33

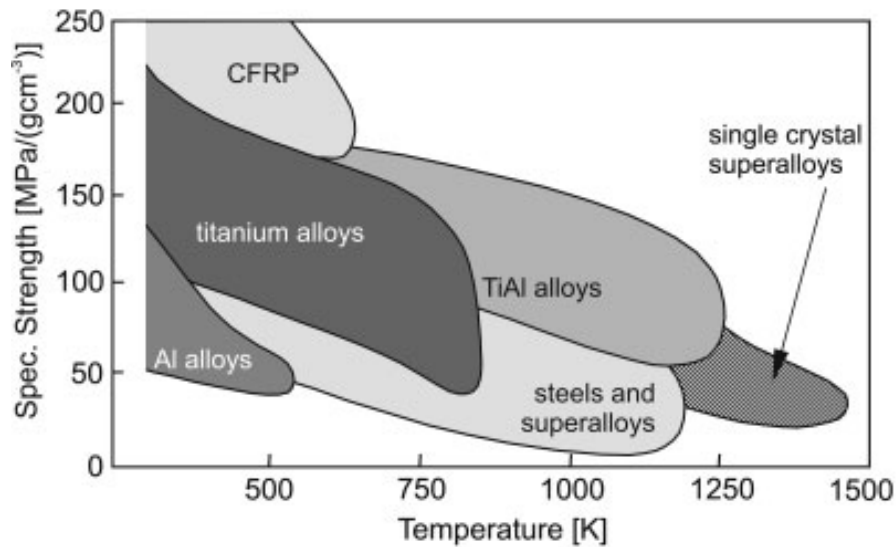


Figure 1.1 Specific strength of various structural materials at different temperatures [1].

1.2. Classification of titanium alloys

Pure titanium has two allotropes; hexagonal closed-pack (HCP) α phase below 882 °C and body-centered cubic (BCC) β phase above 882 °C (i.e., β transus temperature). The atomic unit cells of the HCP α phase and BCC β phase are schematically given in **Figure 1.2(a)** and **(b)**. Highlighted planes in **Figure 1.2** indicate the most atomic dense planes which act as slip planes in plastic deformation.

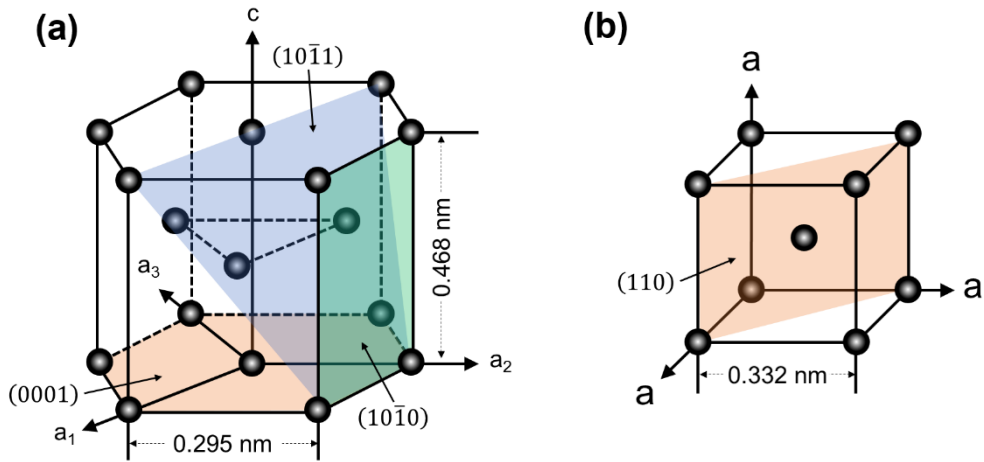


Figure 1.2 Crystal structures of (a) hexagonal closed-pack (HCP) α phase and (b) body-centered cubic (BCC) β phase [1].

The existence of two different crystal structures and corresponding allotropic transformation temperature (i.e., β transus temperature) is the most important characteristics of titanium and titanium alloys to determine their mechanical properties. The β transus temperature of titanium alloy becomes higher or lower according to the alloying elements and its composition. For instance, the β transus temperature increases when aluminum (Al) element is contained in the titanium alloys. The elements like aluminum which make the β transus

temperature increased are called an α -stabilizer. Representative α -stabilizers are Al, O, N, Ga, and Ge. The α -stabilizers not only increase the β transus temperature but also expand a two-phase $\alpha + \beta$ phase region. On the other hand, the β transus temperature decreases when molybdenum (Mo) element is included in titanium alloys. The elements like Mo which make the β transus temperature decreased are called β -stabilizer, and they are V, Nb, Ta, Cr, Mn, Fe, Co, Ni and Cu. The amount of α -stabilizers and β -stabilizers (alloying elements) is an important parameter in titanium alloys to determine not only the volume fraction of α phase and β phase but also the classification of titanium alloys. Titanium alloys can be classified into three different categories; α alloys, $\alpha + \beta$ alloys, and β alloys, as schematically shown in **Figure 1.3**.

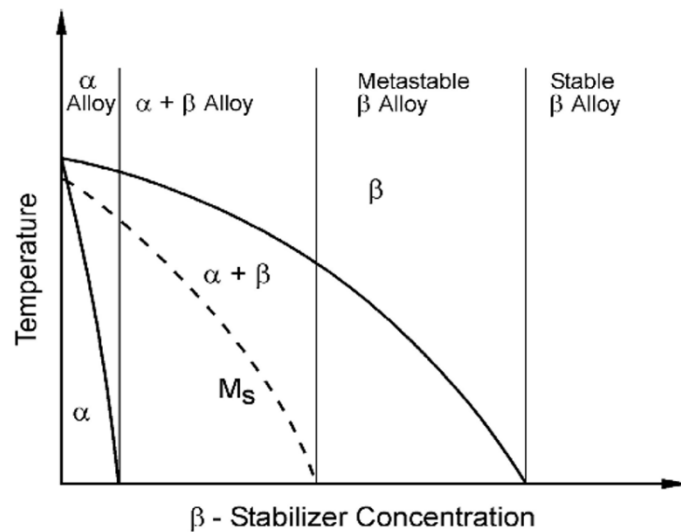


Figure 1.3 Schematic phase diagram of titanium alloys and their classification as a function of the beta stabilizer concentration [2].

According to the schematic diagram, the group of α titanium alloys are exclusively alloyed with α -stabilizers without so much β -stabilizers. Concerning commercial purity (CP) titanium,

there are four different grades with respect to their oxygen content from 0.18% (grade 1) to 0.40% (grade 4). Interstitial alloying element, oxygen has an effect on increasing strength with losing ductility. Among the four grades of CP titanium, grade 1 CP titanium possesses the lowest strength level but outstanding cold formability. Grade 4 CP titanium has the highest strength up to approximately 740 MPa, but complex parts made of grade 4 CP titanium should be manufactured at high temperatures over 300 °C due to its poor formability at low temperature. On the other hand, a representative α titanium alloy containing α -stabilizers is a Ti-5Al-2.5Sn alloy. Ti-5Al-2.5Sn alloy has much higher tensile strength (780 ~ 820 MPa) than the CP titanium grade 4. Two kinds of α titanium alloys, Ti-0.2Pd and Ti-0.3Mo-0.8Ni, are advantageous in corrosion resistance. Such α titanium alloys possess superior properties in particular fields but they cannot be expected to have strengthening effects through heat-treatments due to low alloying compositions.

The most widely used alloy group, $\alpha + \beta$ titanium alloys, have volume fractions of β phase ranging from about 5 to 40 % due to the coexistence of α -, β -stabilizers and neutral elements (the elements which do not effect on the β transus temperature). Mechanical properties of $\alpha + \beta$ titanium alloys strongly depend on the volume fraction of two phases. Among the group of $\alpha + \beta$ titanium alloys, alloys containing relatively small amounts of α -stabilizers (less than approximately 10 % of β phase volume fraction) are called near- α titanium alloys and they are mainly used at high service temperatures. Ti-6Al-4Zr-2Mo and Ti-8Al-1Mo-1V alloys are the representative near- α alloys. The $\alpha + \beta$ titanium alloys have over than 10 % of α phase volume fraction at room temperature, and the most used $\alpha + \beta$ titanium alloy is Ti-6Al-4V alloy. Including the near- α titanium alloys, more than 60 % of practically used titanium alloys are the

$\alpha + \beta$ titanium alloys. Among the $\alpha + \beta$ titanium alloys, the Ti-6Al-4V alloy occupies over 50 % in usage due to its good mechanical properties (balanced strength and ductility, advantageous in forging and rolling fabrications and ability of aging strengthening, etc.). Typical yield strength and tensile strength of Ti-6Al-4V alloy are reported to be 823.2 MPa and 891.8 MPa, respectively. It is possible to enhance the yield strength over 980 MPa through aging treatment. Not only the Ti-6Al-4V alloy but also Ti-6Al-6V-2Sn, Ti-6Al-2Sn-4Zr-6Mo and Ti-10V-2Fe-3Al alloys are used when the relatively higher strength is necessary at room temperature.

β titanium alloys that can show metastable BCC single phase at room temperature have excellent cold workability due to their outstanding tensile ductility up to 90 %. Even when the alloy amount of β -stabilizers further increases to the level where β phase no longer transforms to martensite upon rapid quenching, the alloys are still in the two-phase field at room temperature. It should be noted that these alloys can still reveal an equilibrium α volume fraction of more than 50 %. Finally, single-phase β alloys appear at the end of the alloying scale in conventional titanium alloys. By conducting aging treatment, yield strength and tensile strength of the single-phase β alloys can reach to 1470 MPa and 1372 MPa, respectively. For this reason, they are expected to be applied in various industries in future.

1.3. Microstructure and mechanical properties of $\alpha + \beta$ titanium alloys

Thermomechanical processing (TMP) that means a combination of plastic deformation process (i.e., compression, forging, rolling, etc.) and thermal processes (i.e., heat-treatment and cooling at various rates) is a useful way to control microstructural features of metallic materials. Indeed, microstructural features in $\alpha + \beta$ titanium alloys strongly depend on the condition of

the TMP [5-8]. Volume fraction and shape (morphology) of α and β phases vary depending on TMP conditions. According to the temperature and cooling rate in TMP, three types of typical microstructures can be obtained in $\alpha + \beta$ titanium alloys: i.e., equiaxed, bi-modal or lamellar microstructures. Each microstructure represents different mechanical response.

1.3.1. Equiaxed and bi-modal microstructures

When TMP including hot-deformation is conducted in $\alpha + \beta$ phase region, equiaxed microstructures (like **Figure 1.4(a)**) composed of mainly equiaxed α grains can be obtained after slow cooling. On the other hand, bi-modal microstructures (like **Figure 1.4(b)**) composed of equiaxed primary α and fine plates of secondary α can be obtained by rapid cooling after TMP in $\alpha + \beta$ phase region. The grain size and aspect ratio of the equiaxed α in the equiaxed and bi-modal microstructures depend on deformation temperature, applied strain, and strain rate [5, 9-12]. The equiaxed microstructure is well known to possess balanced strength and ductility. Furthermore, the equiaxed microstructure provides excellent resistance against fatigue initiation, but poor resistance for propagation [1]. On the other hand, the bi-modal microstructure exhibits better strength than that of the equiaxed microstructure, superb creep property and well-balanced fatigue properties [1].

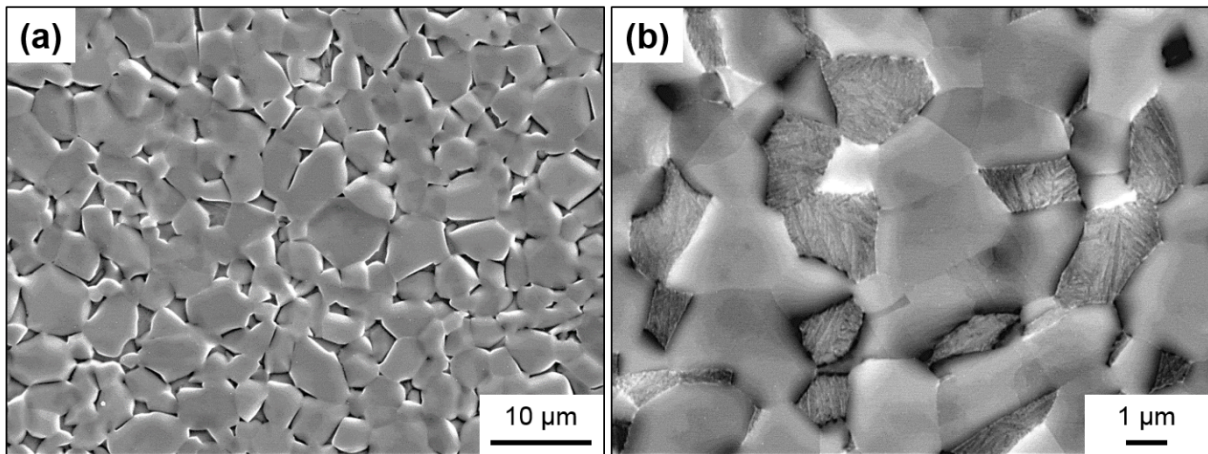


Figure 1.4 SEM micrographs showing typical (a) equiaxed and (b) bi-modal microstructures obtained through high temperature compression test at 900 °C followed by slow cooling and water quenching, respectively.

1.3.2. Lamellar microstructures

When TMP is carried out above β transus temperature, lamellar microstructure is obtained after relatively slow cooling, as shown in **Figure 1.5**. The lamellar microstructure exhibits coarse α plates (hereafter called α -lamellae). The thickness of α -lamellae is reported to be determined by cooling rate after heat treatment [2, 7]. With increasing the cooling rate, the thickness of α -lamellae decreases. Martensite microstructure composed of fine plates of martensite (α') with various orientations is obtained after rapid cooling condition from β single phase temperature through diffusionless (martensitic) transformation. The lamellar microstructure is composed of the colonies each of which is an agglomerate of α -lamellae having identical crystallographic orientation. A previous research has indicated that the lamellar microstructure exhibits lower strength, lower ductility, but better fatigue resistance for crack propagation compared with the equiaxed microstructure [13]. Early investigations in this

area have shown that the lamellar microstructure has better fracture resistance than $\alpha + \beta$ titanium alloys with the equiaxed or bi-modal microstructures [14-19].

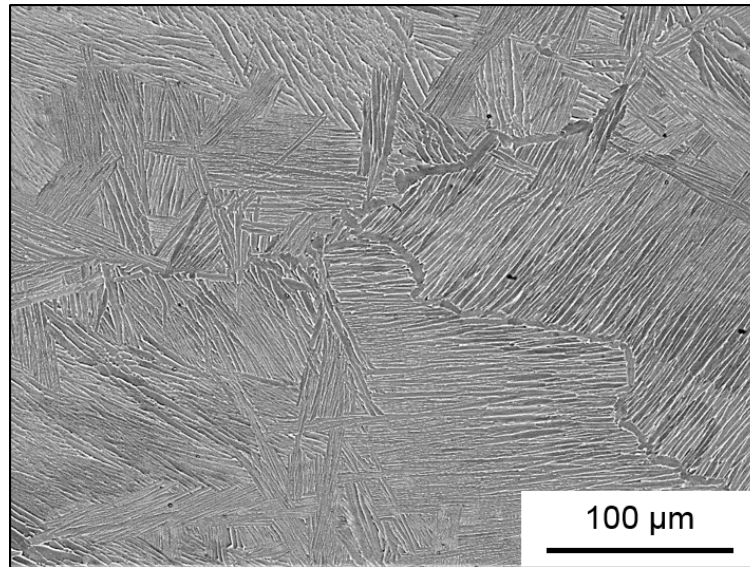


Figure 1.5 SEM micrograph of a typical lamellar microstructure.

It is well known that microstructural parameters in the lamellar microstructure, such as prior- β grain size, colony size and thickness of α -lamellae, have strong effects on mechanical properties and the fatigue crack growth resistance of the material [20-22]. Additional studies proved that fully lamellar microstructures, particularly those having coarse lamellae, improved fatigue strength due to effective crack closure [23, 24]. Eylon et al. [14-16] concentrated to study on characterizing fatigue behaviors in lamellar microstructures. They found that fatigue cracks propagated along shear bands formed at crack tip. Moreover, they insisted that refinement of colony size led to a reduction of the growth rate of fatigue cracks due to the interruption of crack propagation at colony boundaries. Lütjering [7] studied the effect of colony size on various mechanical properties of lamellar microstructures, including yield

strength, ductility, fracture toughness, creep strength, fatigue crack nucleation and propagation. It was concluded that reduced slip length in smaller colony size played a key role to change the mechanical properties. Shi et al. [18, 19] investigated the influence of colony size on the growth rate of fatigue cracks in a Ti-6Al-2Zr-2Sn-3Mo-1Cr-2Nb alloy as well as the influence of α -plate thickness on the fracture toughness in a Ti-17 alloy. The growth rate of fatigue cracks was found to decrease with decreasing the colony size. They thought that the propagation of main crack in lamellar microstructures with smaller colony size was more easily deflected than that in lamellar microstructures with larger colony size. Moreover, they asserted that α -plate thickness also contributed to the propagation path of the main crack, influencing on the fracture toughness. It was found that the microstructure with thicker α -plate showed more rugged crack propagation path and higher fracture toughness.

1.4. Bi-lamellar microstructure, and motivation and objective of the present study

Based on the background introduced in the previous sections, several microstructural parameters (i.e., colony size, thickness of α -lamellae and prior- β grain size) for improving mechanical properties of Ti-6Al-4V alloy with lamellar microstructure have been clarified in several decades. Not only changing microstructural parameters in the lamellar microstructure but also changing the lamellar microstructure into the bi-lamellar microstructure (**Figure 1.6**) is another method to improve mechanical properties of $\alpha + \beta$ titanium alloys [22]. The bi-lamellar microstructure which is a complex microstructure composed of initial α -lamellae (also called primary α -lamellae) and transformed- β regions including fine and variously oriented secondary α -lamellae transformed from β regions formed between primary α -lamellae in heat

treatment can be obtained by intercritical annealing at $\alpha + \beta$ two-phase temperatures followed by relatively fast cooling. The secondary α -lamellae are fine α -plates having various crystallographic orientations and elongated directions, and a small amount of retained- β exists between the secondary α -lamellae in the transformed β regions. The thickness of the secondary α -lamellae is known to increase with decreasing the cooling rate [7].

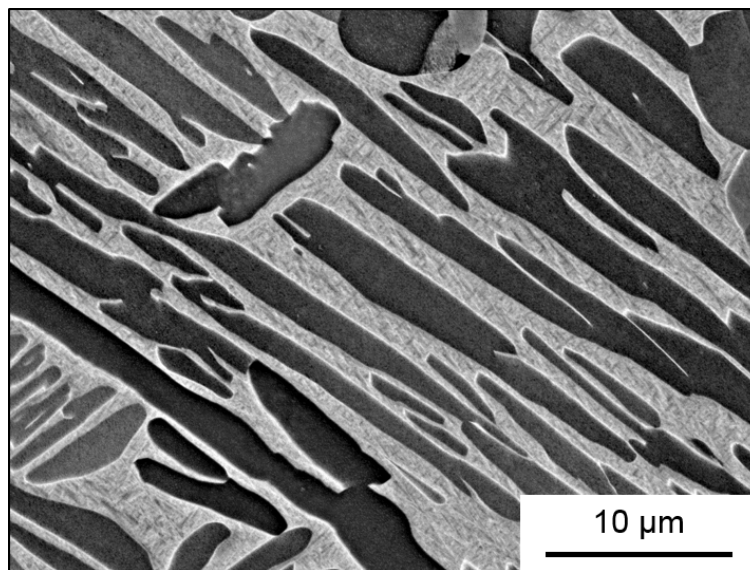


Figure 1.6 SEM micrograph showing a typical bi-lamellar microstructure. The black regions are primary α lamellae, while the grey regions including fine α plates are transformed β regions.

It is believed that the strength of the bi-lamellar microstructure is much higher than that of the lamellar microstructures, because the fine secondary α -lamellae with different orientations inhibit slip transfer across the transformed- β regions and therefore effectively reduce the slip length [7, 22]. **Figure 1.7** shows mechanical properties of the bi-lamellar microstructures as a function of the cooling rates after annealing at 880 °C and those of the lamellar microstructure

obtained under the slowest cooling condition [2]. It was found that the yield strength and the high cycle fatigue (HCF) strength (the resistance to crack nucleation) in the bi-lamellar microstructures gradually increased with increasing the cooling rate. The tensile elongation of the bi-lamellar microstructure was obviously smaller than that of the lamellar microstructure. On the other hand, the resistance to micro-crack propagation (ΔK) in the bi-lamellar microstructures was much higher than that in the lamellar microstructure but the resistance to macro-crack propagation in the bi-lamellar and the lamellar microstructures was about the same.

Although numerous mechanical properties in the various microstructures of $\alpha + \beta$ titanium alloys have been studied and compared, most of the studies on microstructural influence on mechanical properties have focused on equiaxed, bi-modal and lamellar microstructures. The studies on bi-lamellar microstructures are still limited very much by now, even though the bi-lamellar microstructure possesses remarkable mechanical properties. Since the bi-lamellar microstructure has a potential to significantly improve mechanical properties of $\alpha + \beta$ titanium alloys through the microstructure control, it is necessary to investigate the effect of microstructural parameters in the bi-lamellar microstructure on the mechanical properties. Hence, the objectives of the present study are established as follows:

- 1) To establish TMP for obtaining lamellar and bi-lamellar microstructures with various colony sizes. Microstructural features of lamellar and bi-lamellar microstructures are discussed in terms of morphology and crystallographic features of both α phase and β phase.

- 2) To verify the effect of colony size on mechanical properties in lamellar and bi-lamellar microstructures. Nano-hardness values of microstructural components in the lamellar microstructures and the bi-lamellar microstructures are compared. In addition, tensile properties of the lamellar and bi-lamellar microstructures with different colony sizes are discussed.

- 3) To clarify the reason for the different mechanical responses with respect to the colony size in the lamellar and bi-lamellar microstructures. Micro-crack and main-crack formation during tensile deformation in the lamellar and bi-lamellar microstructures are carefully characterized, considering the effect of the colony size. Furthermore, the obtained mechanical properties (especially fracture behaviors and elongation) of the lamellar and bi-lamellar microstructures are correlated to the observed micro-crack and main-crack characteristics in both microstructures.

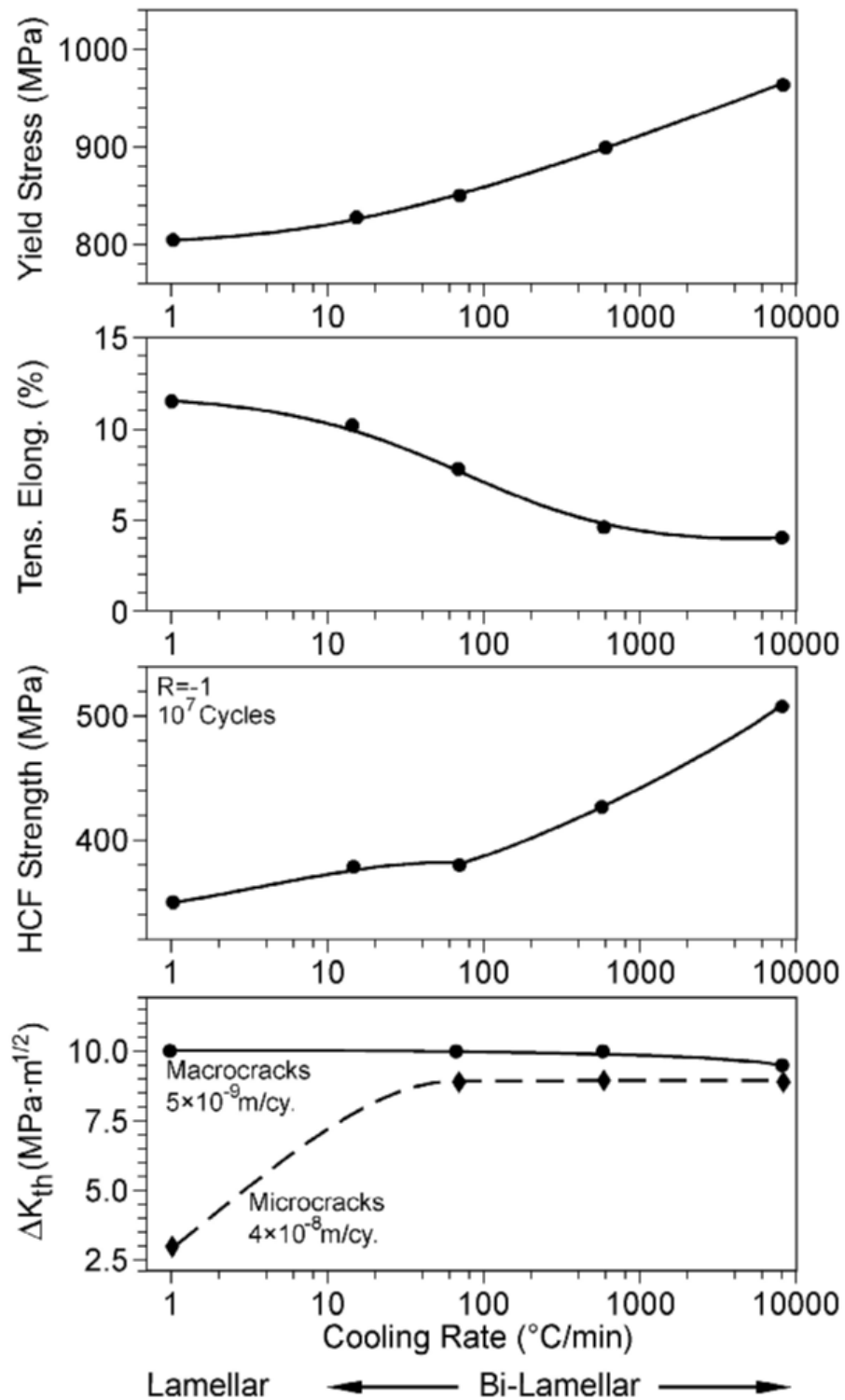


Figure 1.7 Mechanical properties of bi-lamellar microstructures as a function of cooling rate from an annealing temperature of 880 °C ($\alpha + \beta$ phase region) in Ti-6Al-4V alloy. The slowest cooling rate (1 °C / min) indicate lamellar microstructure [2].

1.5. Outlines of dissertation

The present dissertation includes six chapters as described below:

In **Chapter 1: Background and Purpose**, the classification of titanium alloys and characteristics of microstructures in $\alpha + \beta$ titanium alloy and its mechanical properties are described. And then the aim of the present study is stated.

In **Chapter 2: Thermomechanical Processing for Colony Size Refinement of Lamellar and Bi-lamellar Microstructures**, the results of colony size refinement in the lamellar and bi-lamellar microstructures of Ti-6Al-4V alloy obtained through hot compression test under different TMP conditions are explained. Microstructural features (morphologies and crystallographic features) in the lamellar and bi-lamellar microstructures are characterized by scanning electron microscopy (SEM) and electron back-scattering diffraction (EBSD) method in SEM.

In **Chapter 3: Mechanical Properties of Lamellar and Bi-lamellar Microstructures with Different Colony Sizes**, the mechanical properties of the obtained lamellar and bi-lamellar microstructures with various colony sizes are evaluated. Uniaxial micro-tensile tests are carried out at room temperature to discuss the effect of colony size on tensile properties in the lamellar and bi-lamellar microstructures. The hardness (local strength) of each phase in the lamellar and bi-lamellar microstructures is investigated by nano indentation test as well.

In **Chapter 4: Characteristics of Crack Formation in Lamellar and Bi-lamellar Microstructures with Various Colony Sizes**, the micro-crack formation in tensile fractured specimens with the lamellar and bi-lamellar microstructures is quantitatively investigated using

SEM. The characteristics of main-crack formation in the lamellar and bi-lamellar microstructures are categorized in terms of possible crack formation sites. In addition, the crystallographic orientation dependence of crack propagation path against colony was investigated using EBSD. The reason for the obtained results in Chapter 2, i.e., the different mechanical responses depending on the colony size in the lamellar and bi-lamellar microstructures, are discussed based on the obtained results for the crack formation in the lamellar and bi-lamellar microstructures.

In Chapter 5: Fracture Properties in Various Microstructures with Different Grain Sizes or Colony Sizes, the fracture toughness and crack propagation resistance in the equiaxed and bi-modal microstructures with different α grain sizes and the lamellar and bi-modal microstructures with different colony sizes are investigated through three-point bending tests combined to digital image correlation (DIC) technique for characterizing local strain developments.

Finally, the important results and findings obtained in this dissertation are summarized in **Chapter 6: Summary and Conclusions**.

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CHAPTER 2

THERMOMECHANICAL PROCESSING FOR COLONY SIZE REFINEMENT OF LAMELLAR AND BI-LAMELLAR MICROSTRUCTURES

2.1. Introduction

Thermomechanical processing (TMP) means a combination of plastic deformation process (i.e., compression, forging, rolling, etc.) and thermal processes (i.e., heat-treatment and cooling at various rates). TMP is an effective method to control microstructural features of metallic materials. Microstructural features of $\alpha + \beta$ titanium alloy can be classified into equiaxed, bi-modal or lamellar microstructure depending on the condition of TMP [1-5].

Since phase transformation between hexagonal closed pack (HCP) α phase to body-centered cubic (BCC) β phase in titanium alloys takes place above β transus temperature (T_β), the microstructural features are varied depending on the temperature region where the TMP is performed. Lamellar microstructure in the titanium alloy is easily obtained through annealing or deformation above T_β followed by relatively slow cooling [5, 6]. During subsequent cooling, the colonies each of which is an agglomerate of α -plates (hereafter called α -lamellae) having equivalent crystallographic orientation are formed inside β grains. The continuous α layers having an equivalent crystallographic orientation with adjacent colony precipitate at β grain boundaries prior to the formation of colonies, which are called grain boundary- α .

The crystallographic orientation of the colonies formed within an identical β grain follows the Burgers orientation relationship (BOR) having $\{0002\}_\alpha \parallel \{110\}_\beta$ and $\langle 11\bar{2}0 \rangle_\alpha \parallel \langle 111 \rangle_\beta$ which is the crystallographic orientation relationship in the $\alpha \leftrightarrow \beta$ phase transformation [7-9].

The BOR can be confirmed through retained- β in-between α lamellae within the colonies. It is believed that in subsequent plastic deformation, the effective slip length in lamellar microstructure is the colony size as the lamellar boundary within the identical colony is a low-angle boundary and possesses little resistance to dislocation glide. Therefore, smaller colony size is favorable for higher strength.

In order to avoid large colony size or large β grain size, a high-temperature deformation is usually conducted by forging or rolling in the lower β phase region or in the $\alpha + \beta$ phase region. In case of the deformation in the $\alpha + \beta$ phase region, a subsequent annealing treatment is performed at 30 - 50 °C above the T_β to obtain relatively fine recrystallized β grains. The cooling rate after the annealing treatment or deformation in the β phase region is the most important parameter to obtain desired microstructural features, such as the colony size or the thickness of α -lamellae in the lamellar microstructure. G. Lütjering [5] has proved that the colony size and the thickness of α -lamellae increase with decreasing the cooling rate. Additionally, when the cooling rate is fast enough (i.e., water quenching), a martensite microstructure composed of acicular shapes of α phase is obtained through diffusionless transformation [2].

It has been proved by many researchers that the microstructural parameters of lamellar microstructure, like colony size, β grain size, and thickness of α -lamellae, are important factors to influence mechanical properties of the lamellar microstructure [5, 10-18]. In order to obtain better mechanical properties than the lamellar microstructure, a new microstructure named the bi-lamellar microstructure which is obtained by precipitating fine secondary α -lamellae within thickened β phase between primary α -lamellae has been suggested by G. Lütjering. The

secondary α -lamellae can precipitate during fast cooling after inter-critical annealing in $\alpha + \beta$ phase regions. Like the thickness of α -lamellae in the lamellar microstructure, the thickness of the secondary α -lamellae in the bi-lamellar microstructure is varied depending on the cooling rate after inter-critical annealing. It has been reported that the presence of fine secondary α -lamellae having different elongated directions and different crystallographic orientations effectively reduce the slip length within an identical colony in plastic deformation, resulting in the enhanced strength [5, 19].

Since the lamellar and bi-lamellar microstructures are obtained in the relatively higher temperature, it is necessary to establish the proper TMP for effectively controlling microstructural parameters such as colony size and thickness of primary/secondary α -lamellae. Hence, the present study aims to establish the TMP for obtaining various colony sizes in the lamellar and the bi-lamellar microstructures in Ti-6Al-4V alloy. Additionally, the crystallographic features of the obtained lamellar and bi-lamellar microstructures are studied to compare the microstructural characteristics in both microstructures.

2.2. Experiment procedure

2.2.1. Fabrication of lamellar microstructures with various colony sizes

An as-cast Ti-6Al-4V billet is used for this study. The detailed chemical composition is given in **Table 2.1**. Schematic illustration of the as-cast billet is shown in **Figure 2.1(a)** and **(b)**. Two different zones with different microstructures were confirmed due to the difference in cooling rate between the inner part (i.e., slow cooled zone) and the outer part (i.e., rapidly cooled zone). The initial microstructure in the slowly cooled zone was used as the lamellar

structure composed of colonies and thick α -lamellae shown in **Figure 2.2**. Average β grain size, colony size, and thickness of α -lamellae were measured to be 600 μm , 300 μm , and 3 μm , respectively. Microstructure in the rapidly cooled zone will be described in the later part. Cylindrical specimens with 8 mm in diameter and 12 mm in height were cut from the as-cast billet, as shown in **Figure 2.1(c)**. In order to obtain lamellar microstructures with various colony sizes, TMP was carried out using hot compression in a thermomechanical simulator (Thermecmasto-Z). The deformation process for obtaining lamellar microstructures with different colony sizes is schematically depicted in **Figure 2.3**.

Figure 2.3(a) indicates the deformation process in β phase region. The specimens were heated to 1050 $^{\circ}\text{C}$ and held for 0.3 ks to homogenize temperature in the specimen. Subsequently, the compression test was performed at a strain rate of 10^{-2} s^{-1} . In order to obtain various colony sizes, the compressive strain was controlled ranging from 0 to 1.2 in equivalent strain (ϵ). After the hot-compression, the specimens were slowly cooled to 200 $^{\circ}\text{C}$ at a cooling rate of 10 $^{\circ}\text{C min}^{-1}$ to obtain colonies composed thick α -lamellae. This process is named as the ‘simple deformation process’.

Table 2.1 Chemical compositions of the as-cast Ti-6Al-4V alloy.

Elements	Ti	Al	V	Fe	O
Contents (wt. %)	Bal.	6.29	4.35	0.225	0.155

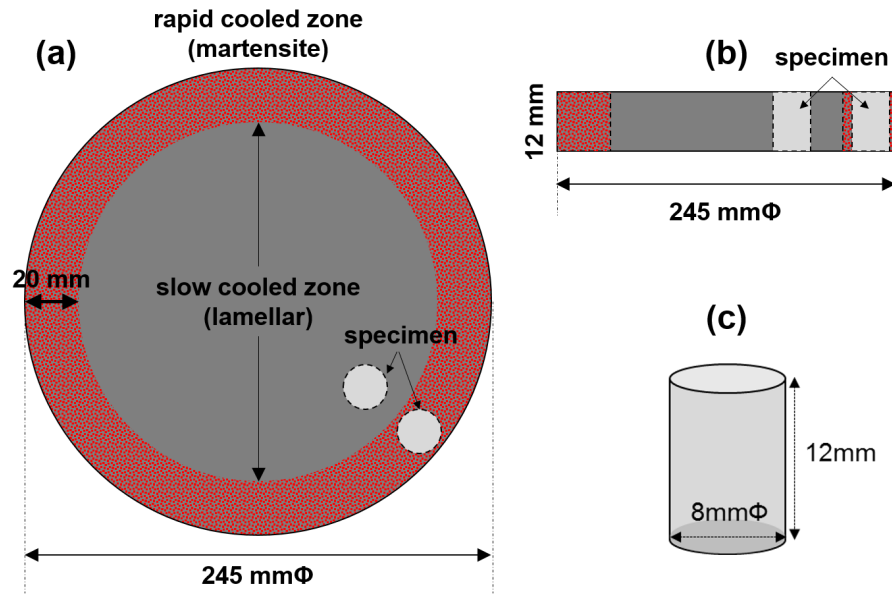


Figure 2.1 Schematic illustration of the as-cast Ti-6Al-4V billet in (a) plan view and (b) side view. Light grey circle in (a) and light grey rectangular in (b) correspond to the slowly cooled regions having the coarse lamellar microstructure used for (c) cylindrical specimens for TMP processes.

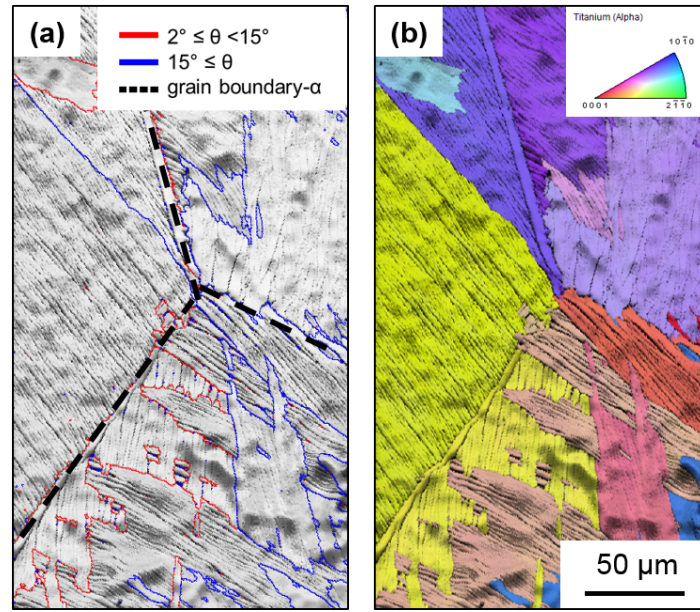


Figure 2.2 Lamellar microstructure in the slowly cooled zone of as-cast billet, represented by (a) EBSD image quality (IQ) map and (b) EBSD orientation color map.

In the simple deformation process, it was found that the colony size refinement in the lamellar microstructure was limited even though the compressive strain increased. Detailed results of colony size refinement are given in the later section 2.3.1. Hence, an additional compression test was performed in $\alpha + \beta$ phase region to realize smaller β grain sizes before the hot-compression in the β phase region equivalent to the simple deformation process, as shown in **Figure 2.3(b)**. The initial microstructure in the rapidly cooled zone was used for the process shown in **Figure 2.3(b)**. The microstructure in the rapidly cooled zone was a martensite microstructure comprised of acicular shape of α phase shown in **Figure 2.4**. The specimens were heated to 700 °C ($\alpha + \beta$ phase region) and held for 0.6 ks. Subsequently, the specimens were uniaxially compressed to 0.6 of compressive strain at a strain rate, 10^{-3} s^{-1} . After the compression in the $\alpha + \beta$ phase region, the specimens were rapidly heated to 1050 °C and held

shortly for 60 s. And then, the specimens were compressed again to another 0.6 of compressive strain at 1050 °C (i.e., a total compressive strain of 1.2). In the same manner as the simple deformation process in **Figure 2.3(a)**, the specimens were subsequently cooled to 200 °C at a cooling rate of 10 °C min⁻¹. This process was named as the ‘multiple deformation process’.

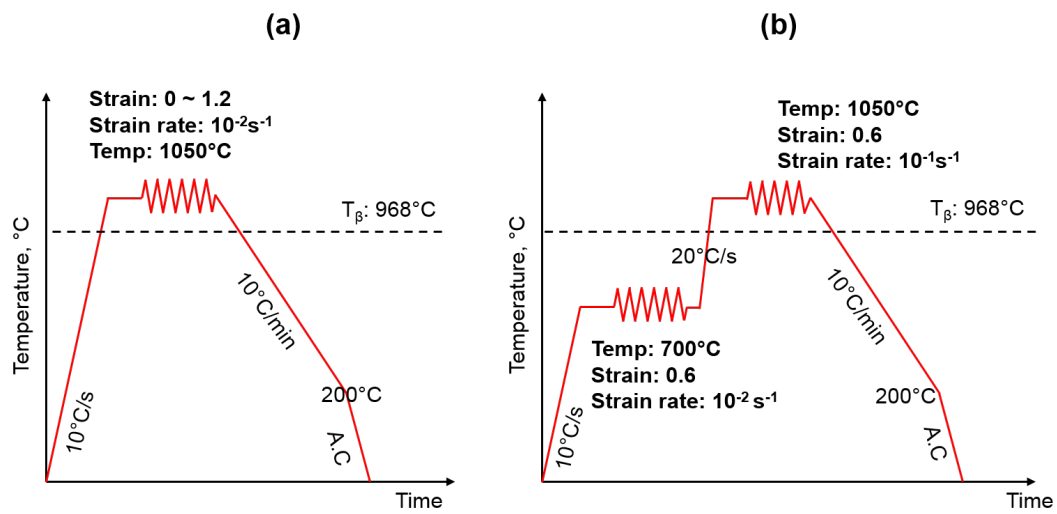


Figure 2.3 Schematic illustrations of (a) the simple deformation process and (b) the multiple deformation process to obtain various colony sizes in lamellar microstructures of Ti-6Al-4V alloy.

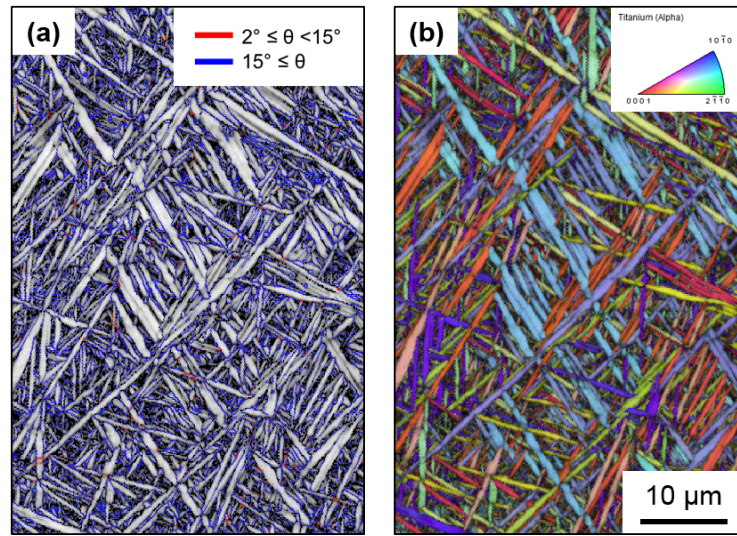


Figure 2.4 Martensite microstructures in the rapidly cooled zone of the as-cast billet, represented by (a) EBSD image quality (IQ) map and (b) EBSD orientation color map. The microstructure was used as the initial microstructure for the multiple deformation process.

2.2.2. Fabrication of bi-lamellar microstructures with various colony sizes

In order to obtain bi-lamellar microstructures with various colony sizes, the specimens having the lamellar microstructures with various colony sizes obtained were inter-critically annealed at 920 °C corresponding to an $\alpha + \beta$ phase region for 0.6 ks, followed by water quenching. The additional annealing process was named as the inter-critical annealing. The inter-critical annealing process is represented schematically in **Figure 2.5**.

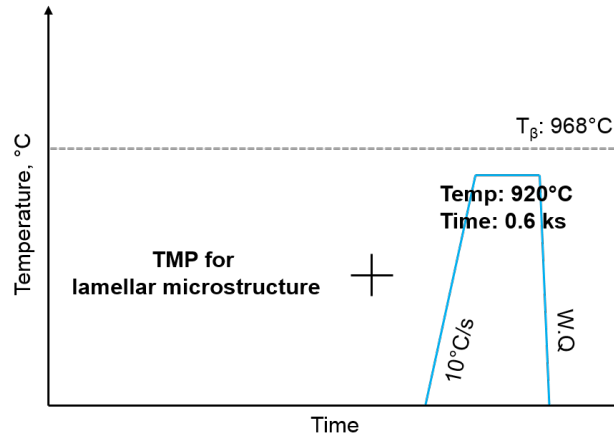


Figure 2.5 Schematic illustration of the inter-critical annealing process at 920 °C for 0.6 ks followed by water quenching to obtain bi-lamellar microstructures.

2.2.3. Microstructure characterization

Microstructural features of the lamellar and bi-lamellar microstructures were characterized by a field-emission type scanning electron microscope (FE-SEM, JEOL-6800F) and electron back-scattering diffraction (EBSD, JEOL-7100F). For the SEM and EBSD observations, the specimens processed by the TMP described above were sectioned parallel to the compression axis using wire electron discharge machine. The surface of each sectioned specimen was mechanically polished by emery paper (#120 – 4000) first, and then electronically polished in a solution of 10 % perchloric acid (HClO_3) and 90 % methanol (CH_3OH) at -30 °C.

In the high temperature compression tests, high friction at contact surfaces between the specimens and the compression anvils introduces heterogeneous deformation in the specimen. The heterogeneities introduced by high-friction compression are usually distinguished into three different deformation zones. In order to confirm the heterogeneities during the compression test, the finite element (FE) simulation was performed using commercial finite element analysis software (DEFORM-3D ver. 6.1) shown in **Figure 2.6(a)**. The FE simulation

was performed based on the physical property of the typical Ti-6Al-4V alloy and the standard of the specimen in **Figure 2.1(c)**. Friction coefficient (μ) was given as Coulomb's friction coefficient ($\mu = 0.1$). The condition of the compression test was established to be performed at 925 °C with a strain rate, 10^{-1} s^{-1} . Blue colored zone in the upper and lower part of the compressed specimen termed as the dead-metal zone (DMZ), where the metal is deformed little. On the other hand, the center region of the specimen termed as severe plastic deformation (SPD) zone, where the metal is deformed severely. **Figure 2.6(b)** presents the optical micrograph of the macro-etched hot-compressed specimen. The different microstructured zones, DMZ and SPD zone were clearly observed and their boundaries were indicated by the red-colored broken lines. Hence, the center region of each polished specimen was observed to avoid the DMZ in the compressed specimen. The microstructural features of the lamellar and bi-lamellar microstructures were exhibited by EBSD inverse pole figure (IPF) maps and image quality (IQ) maps analyzed by TSL OIM Analysis software (Orientation Imaging Microscopy v7, EDAX Inc.). The IPF map is a powerful mapping tool to visualize crystallographic orientations of materials in specific colors. However, it was difficult to distinguish the α -lamellae boundaries from the IPF maps due to the equivalent crystallographic orientation (i.e., the same color in IPF maps) of α -lamellae and thin retained- β within an identical colony. In order to overcome this problem, the IQ maps were combined with the IPF maps in the present study. The IQ map which indicates the difference of contrast reflecting the quality of Kikuchi patterns in EBSD measurement can distinguish the α -lamellae boundaries due to the weak signal of Kikuchi patterns at boundaries and on thin retained- β in the lamellar microstructures of Ti-6Al-4V alloy.

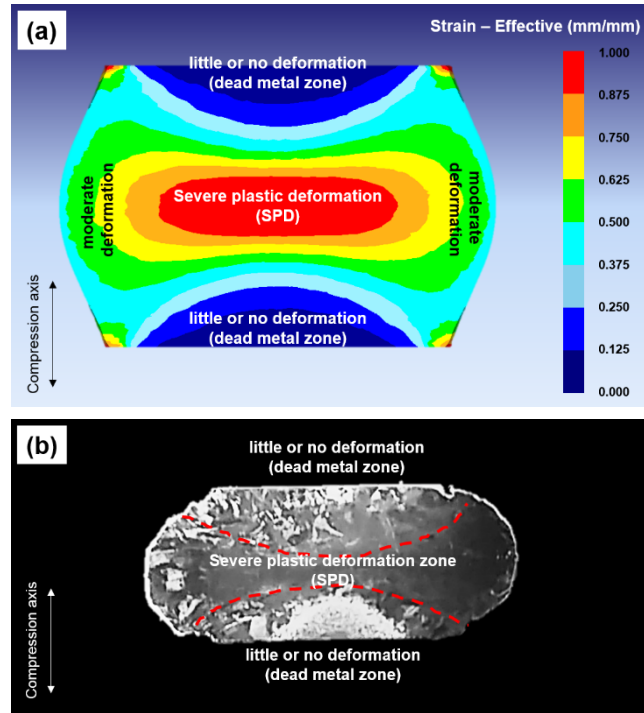


Figure 2.6 (a) Strain distribution in the hot compressed specimen obtained by a FE simulation and (b) an optical micrograph of the macro-etched hot-compressed specimen.

2.3. Results and Discussion

2.3.1. Colony size refinement in lamellar microstructures through simple deformation process

Figure 2.7 shows EBSD IPF maps and SEM images of lamellar microstructures deformed at different compressive strains ranging from 0 to 1.2 in the simple deformation process described in **Figure 2.2(a)**. The colors in the IPF maps indicate the crystallographic orientations of each point parallel to the compressive direction (CD) of the specimens, according to the key stereographic triangle shown in the figure. Every microstructure was composed of many colonies with different crystallographic orientations (different colors). Each colony was composed of α -lamellae having an equivalent crystallographic orientation. The β grains composed of several colonies were surrounded by grain boundary- α emphasized by

yellow lines in **Figure 2.7(f)**. The obtained lamellar microstructures were composed of thick α -lamellae and thin retained- β layer sandwiched by the α -lamellae as shown in **Figure 2.7(g)**. The thickness of α -lamellae was approximately 3 μm and that of retained- β was thinner than 1 μm . As shown in **Figure 2.7(a-e)**, the colony size of the specimen hot-compressed to higher strain was finer. In order to quantitatively compare the colony size in each compressive strain, the colony size was measured by length density of high angle grain boundaries (HAGB with misorientation angles (θ) higher than 15°) in EBSD boundary maps. The boundaries between different colonies were generally HAGBs. The length density of HAGBs (D_L) was calculated after dividing the total length of HAGBs by the measured area. It was found from the results summarized in **Figure 2.8** that the length density of HAGBs gradually increased with increasing the compressive strain because the number of colonies per unit area increased. The colony size (d_c) in the lamellar microstructures obtained in the present study were evaluated as, $d_c = 2/D_L$. Here, the quantitative microscopy shows that the three-dimensional average means interception length L [m] of grains (considered as the mean grain size) and boundary density per unit volume S_V [m^2/m^3], stands the relationship, $S_V = 2/L$, for any shapes of grains [20]. In the present case, D_L and d_c are considered equivalent to S_V and L , respectively, in the above mentioned relationship.

Change in the colony size is shown in **Figure. 2.8** as a function of the compressive strain. It was found that the colony size decreased from 234.4 μm to 128.7 μm when the compressive strain increased from 0 to 1.2. Lütjering [5] proposed that the cooling rate is an important parameter to determine not only the α -lamellae thickness but also the colony size. In this study, it was revealed that the compressive strain was another important parameter for determining

the colony size. The colony sizes of the specimens obtained by the simple deformation process by strains 0, 0.3, 0.6, 0.9 and 1.2, were 234.4 μm , 139.5 μm , 130.4 μm , 131.6 μm and 128.7 μm , respectively. Interestingly, the colony size was saturated to approximately 130 μm , when the compressive strain was over 0.3. This is considered to be due to the limited number of the crystallographic orientation of the grain boundary- α which determines the orientation of the adjacent colony. In case of the simple deformation process, recrystallization of β does not take place and the shape of unrecrystallized β grains are governed by the deformation condition such as the compressive strain and the temperature. It means that the formation site (i.e., β grain boundary) of the grain boundary- α is restrictive due to the coarse β grain size even after higher compressive strains, resulting in a small number of coarse colonies.

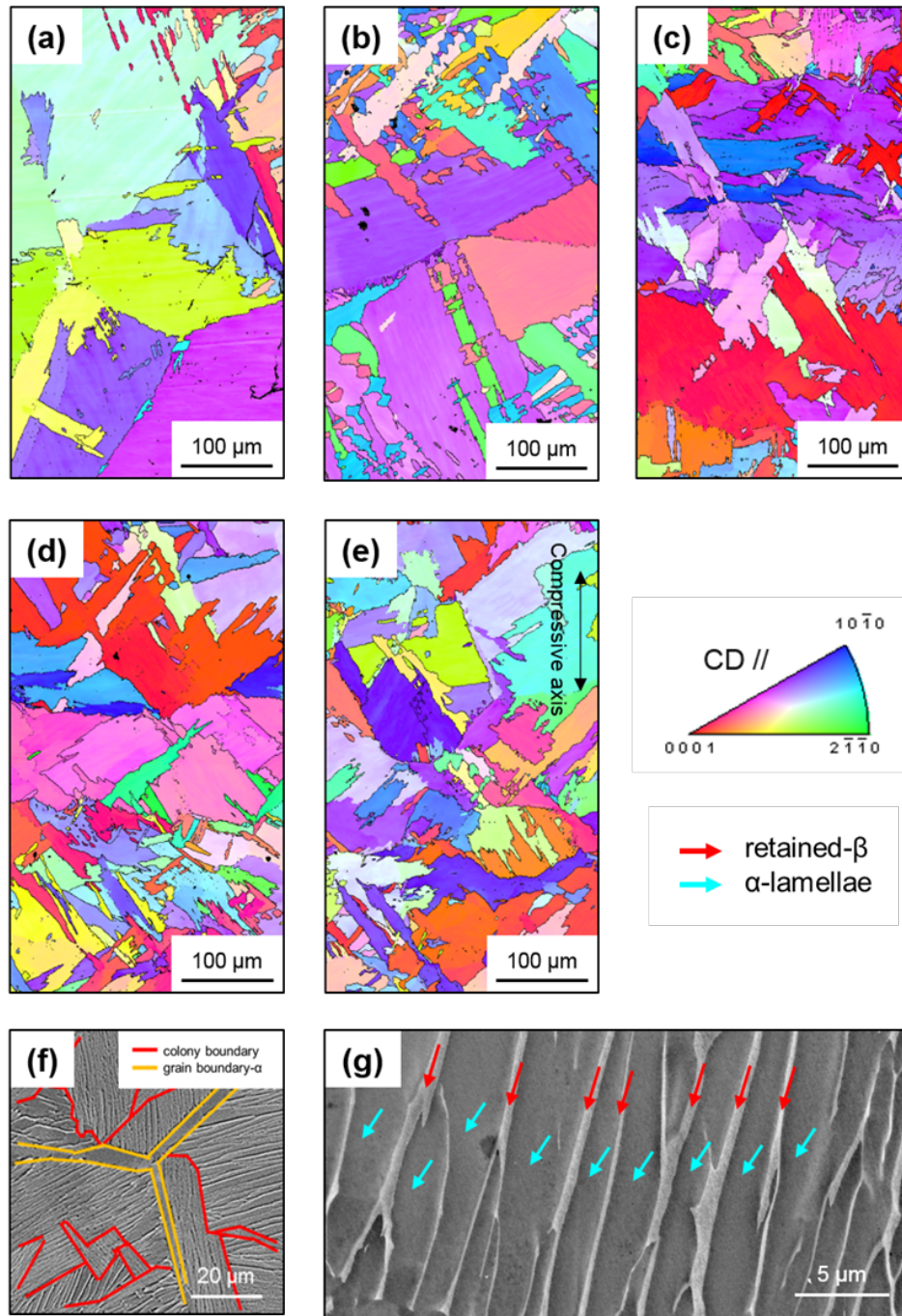


Figure 2.7 EBSD IPF maps of lamellar microstructures obtained through the simple deformation process with compressive strains of (a) 0, (b) 0.3, (c) 0.6, (d) 0.9 and (e) 1.2. (f) A SEM image corresponding to the specimen (a) indicating colony boundaries and grain boundary- α by red and yellow lines, respectively. (g) A SEM image of the specimen (a) in higher magnification to reveal α -lamellae and retained- β indicated by red arrows and blue arrow, respectively.

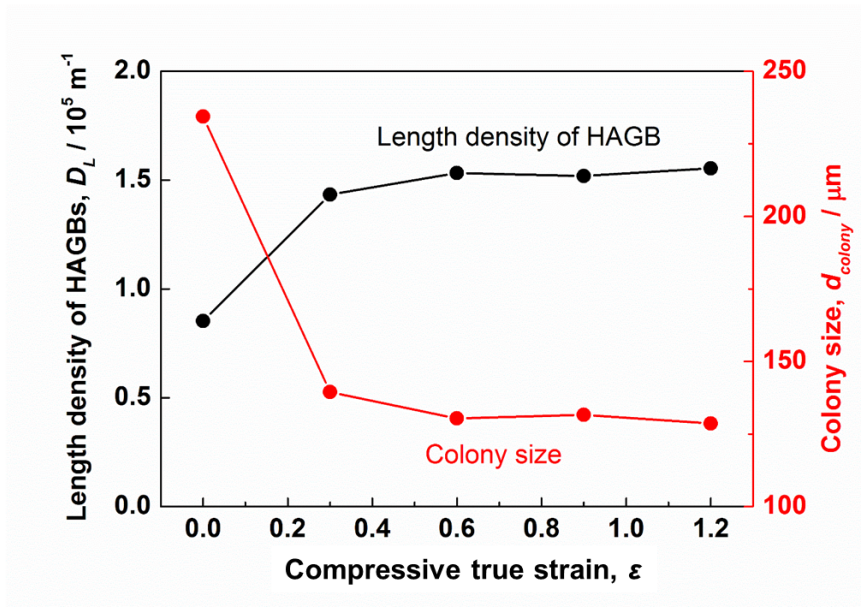


Figure 2.8 The length density of high angle grain boundaries (HAGBs) and the colony size in the lamellar microstructures obtained by the simple deformation process, plotted as a function of the compressive strain ranging from 0 to 1.2.

2.3.2. Colony size refinement in lamellar microstructures through multiple deformation process

In order to induce more crystallographic orientations in the grain boundary- α , the β grain size was refined through the multiple deformation process shown in **Figure 2.2(b)**. At the first deformation in the $\alpha + \beta$ phase region (700 °C), the acicular shape of fine α phases in the martensite microstructure was fragmented and transformed into fine and equiaxed α phases (hereafter called equiaxed- α) through a globularization behavior [21-25]. When the temperature elevated to the β phase region (1050 °C) after the deformation in the $\alpha + \beta$ region, the fine equiaxed- α transformed into fine β phase. **Figure 2.9** exhibits the SEM-BSE microstructure of the specimen manufactured through the simple deformation process with a strain of 0 and a microstructure of the specimen cooled just after the temperature reached

1050 °C after the deformation at 700 °C in the multiple deformation process, respectively. The white dotted lines indicate the prior- β grain boundaries. Since the grain boundary- α is formed along the prior- β grain boundaries at the beginning of the α phase nucleation [4], the grain boundary- α which was shown in **Figure 2.7(f)** was considered as the prior- β grain boundaries. It was found that the β grain size of the specimen manufactured by the multiple deformation process was obviously finer than that of the specimen manufactured by the simple deformation process. This is because the crystallographic orientation relationship between the parent phases and the produced phases in HCP based metals (i.e., Ti alloy or Zr alloy) follows the strict rule, i.e., the Burgers orientation relationship (BOR) [7-9]. In other words, the larger variation of crystallographic orientations of β phase (i.e., the smaller grain size of β) was obtained from the finer equiaxed- α . As shown in the EBSD IPF maps of **Figure 2.10**, a large number of colonies with different crystallographic orientations (i.e., finer colonies) were obtained in the specimen manufactured through the multiple deformation process. The average colony size in the lamellar microstructure manufactured through the multiple deformation process was refined down to 57.5 μm . This result indicates that the colony size in the lamellar microstructures of Ti-6Al-4V alloy depends on not only the strain in hot-deformation but also the grain size of β phase.

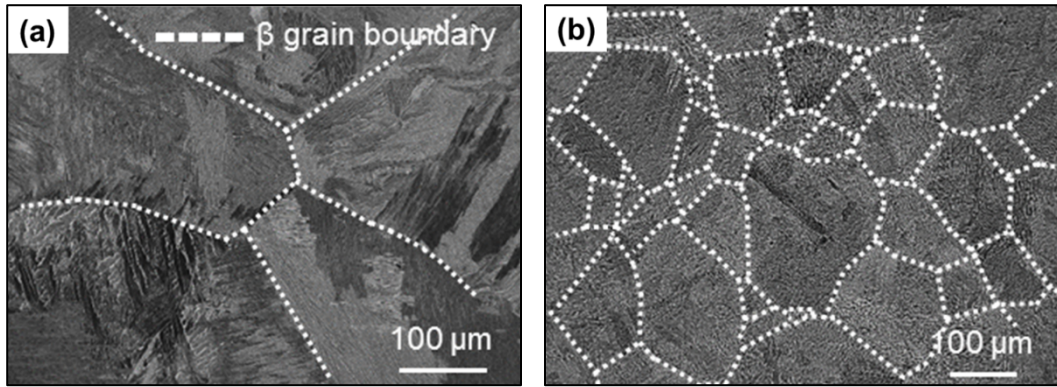


Figure 2.9 SEM-BSE images of the specimens manufactured through (a) the simple deformation process with compressive strain of 0 and through (b) the multiple deformation process using initial deformation at 700 °C and then short annealing at 1050 °C. White dotted line indicates β grain boundaries surrounded by grain boundary- α .

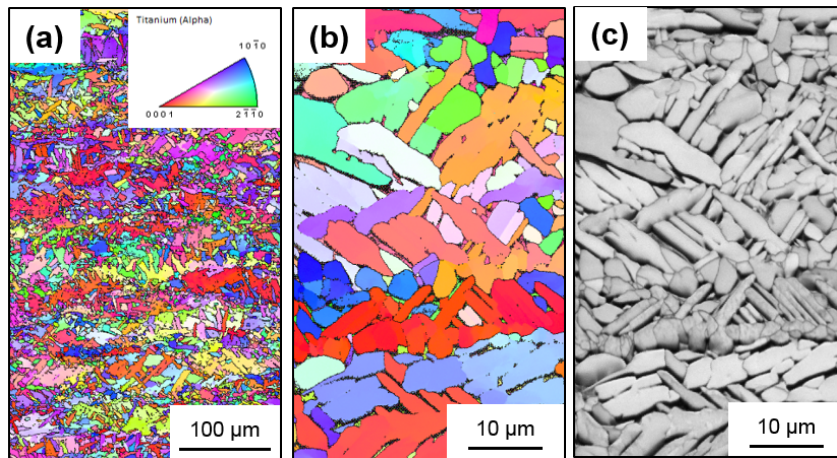


Figure 2.10 EBSD IPF maps in (a) lower magnification and (b) higher magnification of the specimen manufactured through the multiple deformation process. (c) EBSD IQ map corresponding to the observed area shown in (b).

2.3.3. Crystallographic features in lamellar microstructure

In this section, crystallographic features of the obtained lamellar microstructures were

investigated. The specimen having the smallest colony size was analyzed by EBSD for investigating the crystallographic orientation relationship between α -lamellae and retained- β in the lamellar microstructure. The crystallographic features of the colonies were mainly investigated because each colony was composed of α -lamellae with equivalent crystallographic orientation. **Figure 2.11(a)** and **(b)** are IPF maps of the α phase and the β phase, respectively, in the lamellar microstructure with the smallest colony size. Colonies with a large number of crystallographic orientations were observed in **Figure 2.11(a)**. On the other hand, only three different crystallographic orientations of retained- β were confirmed in **Figure 2.11(b)**. In order to investigate the crystallographic orientation relationship between the α -lamellae and the retained- β (i.e., BOR), (0002) and (11 $\bar{2}$ 0) pole figures (PFs) of α phase and the (110) and (111) pole figures of β phase were shown in **Figure 2.11(c)** and **(d)**. Each pole figure was reconstructed from the IPF maps of α and β phases obtained by EBSD analysis, which were shown in **Figure 2.11(a)** and **(b)**, respectively. It was confirmed that (0002) poles of α phase coincided with (110) poles of β phase, although the pole intensities were different. This result indicates that all the (0002) planes of α phase in colonies are parallel to the (110) planes of retained- β . According to the BOR, a [111] direction of β phase should be parallel to a [11 $\bar{2}$ 0] direction of α phase in addition to the plane parallel relationship confirmed above. In **Figure 2.11(a)** and **(b)**, all (111) poles of the β phase coincided with one of (11 $\bar{2}$ 0) poles of α phase. These results represent that the crystallographic orientation relationship between the retained- β and the α -lamellae in the lamellar microstructure maintains the BOR, $\{0002\}_{\alpha} \parallel \{110\}_{\beta}$ and $\langle 11\bar{2}0 \rangle_{\alpha} \parallel \langle 111 \rangle_{\beta}$. Considering the crystallographic symmetry of HCP α phase and BCC β phase, the BOR gives 12 possible variants expressed in **Table 2.2**. One plane parallel

combination has two different direction parallel combinations and the number of distinguishable $\{110\}$ planes of β is 6, so that the total number of variants is 12. The variant selection of colonies was investigated by extracting the microstructure evolved from one specific β grain (#1 β grain in **Figure 2.11(b)**), as shown in **Figure 2.12**.

Figure 2.12(a) represents the IPF map of α phase within the extracted β grain. A large number of colonies with different crystallographic orientations were observed within the initial β grain. The retained- β was confirmed having one crystallographic orientation shown in **Figure 2.12(b)**. In order to visualize the variants selection, the colonies were highlighted by specific colors, as shown in **Figure 2.13(a)**. Corresponding α poles were exhibited using the identical colors in (0002) and (11 $\bar{2}$ 0) PFs shown in **Figure 2.13(b)**. At the same time, (110) and (111) PFs of the β phase were shown in **Figure 2.13(c)**. The variants selection of the α phase were identified by identical colors. Two of three (11 $\bar{2}$ 0) poles of α phase coincided with one of (111) pole of β phase. This means that one (0002) pole (one orientation) of α phase pole resulted in two variants of β phase. All (0002) poles of α phase were analyzed in the same manner and totally 12 variants were verified within the #1 β grain. This result indicates that the colony composed of the α -lamellae in the lamellar microstructure can have a total of 12 crystallographic orientations (i.e., all the 12 possible variants) from one β grain.

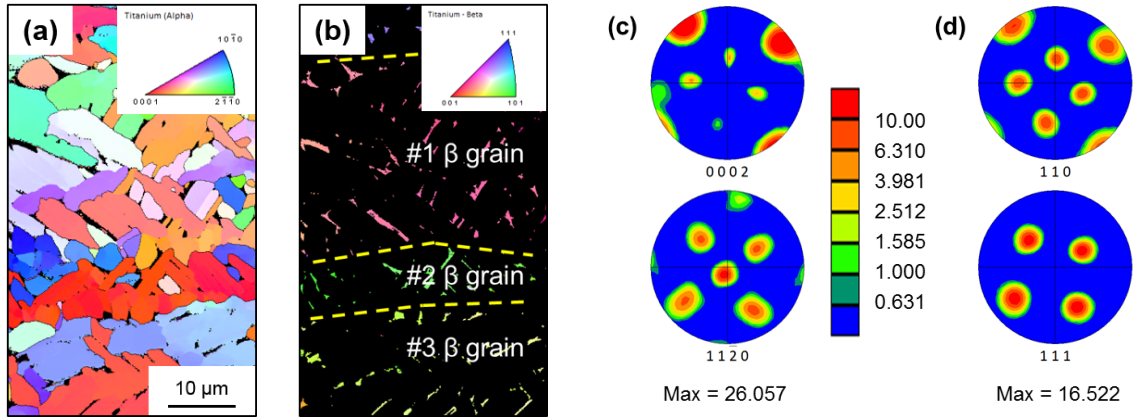


Fig. 2.11 EBSD IPF maps of (a) α phase and (b) β phase in the lamellar microstructure with the colony size of 57.5 μm . (c) (0002) and (11 $\bar{2}$ 0) pole figures (PFs) of α phase and (d) (110) and (111) PFs of β phase corresponding to (a) and (b), respectively.

Table 2.2 12 possible crystallographic variants of α phase transformed from single β phase maintaining the Burgers orientation relationship [8].

Variant	Plane parallel	Direction parallel
V1	$(110)_{\beta} \parallel (0002)_{\alpha}$	$[\bar{1}1\bar{1}]_{\beta} \parallel [11\bar{2}0]_{\alpha}$
V2		$[1\bar{1}\bar{1}]_{\beta} \parallel [11\bar{2}0]_{\alpha}$
V3	$(1\bar{1}0)_{\beta} \parallel (0002)_{\alpha}$	$[11\bar{1}]_{\beta} \parallel [11\bar{2}0]_{\alpha}$
V4		$[111]_{\beta} \parallel [11\bar{2}0]_{\alpha}$
V5	$(011)_{\beta} \parallel (0002)_{\alpha}$	$[11\bar{1}]_{\beta} \parallel [11\bar{2}0]_{\alpha}$
V6		$[\bar{1}1\bar{1}]_{\beta} \parallel [11\bar{2}0]_{\alpha}$
V7	$(01\bar{1})_{\beta} \parallel (0002)_{\alpha}$	$[\bar{1}11]_{\beta} \parallel [11\bar{2}0]_{\alpha}$
V8		$[111]_{\beta} \parallel [11\bar{2}0]_{\alpha}$
V9	$(101)_{\beta} \parallel (0002)_{\alpha}$	$[\bar{1}11]_{\beta} \parallel [11\bar{2}0]_{\alpha}$
V10		$[11\bar{1}]_{\beta} \parallel [11\bar{2}0]_{\alpha}$
V11	$(10\bar{1})_{\beta} \parallel (0002)_{\alpha}$	$[1\bar{1}1]_{\beta} \parallel [11\bar{2}0]_{\alpha}$
V12		$[111]_{\beta} \parallel [11\bar{2}0]_{\alpha}$

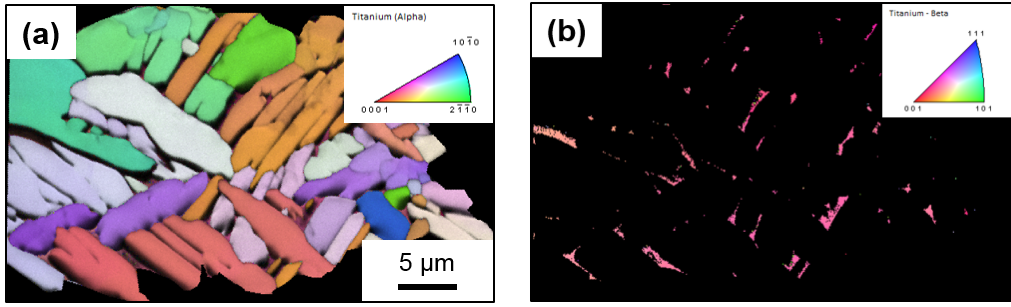


Figure 2.12 EBSD IPF maps of (a) α phase and (b) β phase within a single β grain extracted.

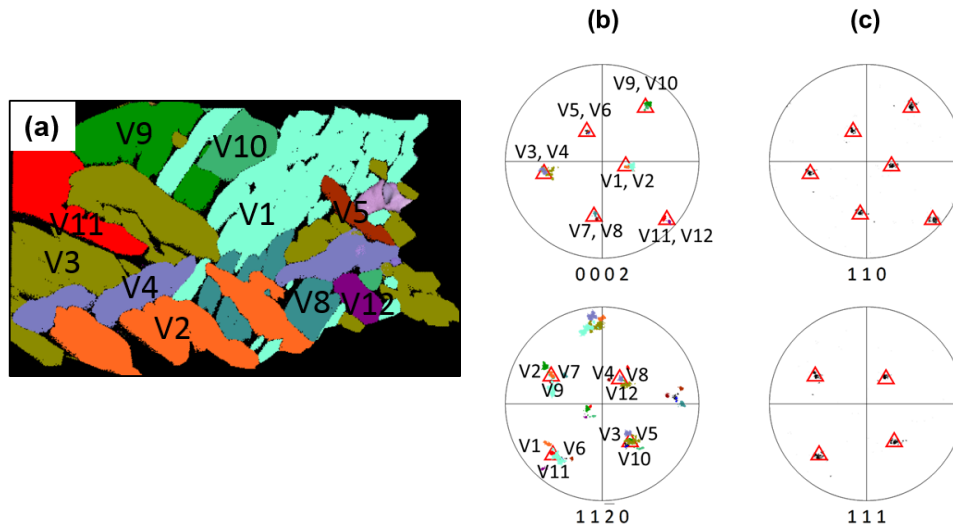


Figure 2.13 (a) EBSD highlighted map of colonies and corresponding pole figures (PFs) of (b) α phase and (c) β phase. Color of each pole corresponds to the crystallographic orientation of the colony highlighted in the same color in (a). Red triangle indicates the position of the poles of β phase.

2.3.4. Colony size refinement in bi-lamellar microstructures

In the previous sections, the colony size in the lamellar microstructures obtained through two different types of TMPs ranged from 234.4 μm to 57.5 μm . In this section, microstructural characteristics of the bi-lamellar microstructure and results of colony size refinement in the bi-lamellar microstructure are described.

Figure 2.14(a-c) represents EBSD IPF maps of bi-lamellar microstructures with various colony sizes. Even though the inter-critical annealing was carried out in the $\alpha + \beta$ phase region, the colony sizes were not changed. Hence, the colony sizes in the bi-lamellar microstructures shown in **Figure 2.14(a-c)** were considered to be equal to those in the initial lamellar microstructures obtained in section 2.3.1 and 2.3.2 (i.e., 234.4 μm , 128.7 μm and 57.5 μm). The former β phase regions between α -lamellae (hereafter called primary α -lamellae), which were indicated by red arrows in **Figure 2.14(d)** and **(e)**, were obviously thickened in comparison with the retained- β in the lamellar microstructures. During annealing at 920 °C, $\alpha + \beta$ phase region, the equilibrium fraction of β phase increased. It was confirmed that the thickness of the former β phase regions (hereafter called transformed- β) in the bi-lamellar microstructure was approximately 3 μm , corresponding to the equilibrium phase fraction at annealing temperature. With increasing the thickness of transformed- β , the thickness of primary α -lamellae was measured to be thinner than α -lamellae in the lamellar microstructure. As observed in **Figure 2.14(d-f)**, fine secondary- α lamellae with various crystallographic orientations precipitated inside transformed- β regions during subsequent cooling (water quenching in this study). The average thickness of the secondary α -lamellae was measured to be thinner than 0.2 μm . According to previous research [19], the thickness of secondary- α lamellae tends to increase with decreasing the cooling rate. It was reported that when the cooling rate exceeds 1000 °C/min, the transformation mode during cooling becomes martensite (α') in Ti-6Al-4V alloy [5]. Hence, it can be considered that the secondary α -lamellae observed in this study was martensite (α').

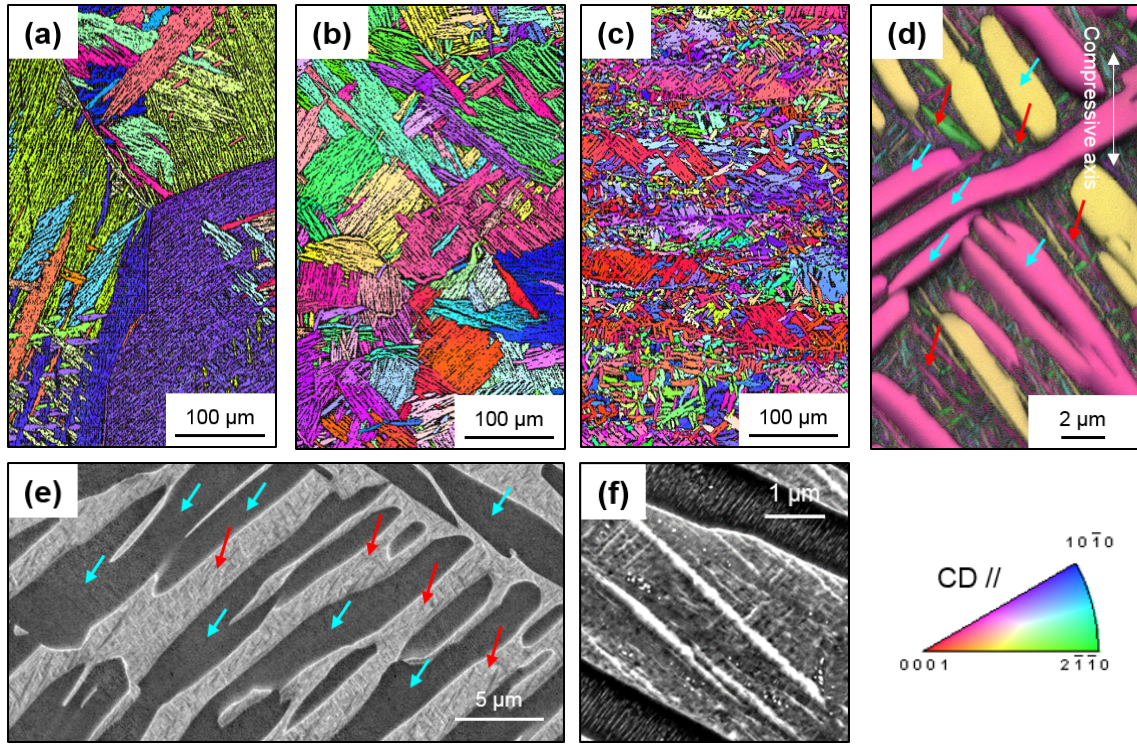


Figure 2.14 EBSD IPF maps and SEM images showing the evolution of bi-lamellar microstructures obtained through inter-critical annealing. The initial lamellar microstructures used for making the bi-lamellar microstructures were obtained through the simple deformation process at 1050 °C to a strain of (a, d, e, f) 0, and (b) 1.2, or obtained through (c) the multiple deformation process. Higher magnification of (d) EBSD IPF map and (e) SEM image represent primary α -lamellae and transformed- β regions indicated by blue arrows and red arrows, respectively. (f) SEM image in higher magnification clearly reveals fine secondary α -lamellae formed inside the transformed- β regions.

2.3.5. Crystallographic features in bi-lamellar microstructure

Crystallographic orientation relationship between the retained- β and the secondary α -lamellae inside transformed- β regions in the bi-lamellar microstructure was investigated in the same manner as that used for the lamellar microstructure. The bi-lamellar microstructure with 57.5 μm of colony size was observed by EBSD. EBSD IPF maps of α phase and β phase in the

observed region are shown in **Figure 2.15(a, b)** and **(c)**, respectively. The IPF maps of α phase with two different specimen axis, parallel to the compressive direction (**Figure 2.15(a)**) and perpendicular to the compressive direction (**Figure 2.15(b)**), were observed to clearly distinguish the crystallographic orientation of the secondary α -lamellae. The thick primary α -lamellae and the fine secondary α -lamellae inside the transformed- β regions were clearly observed, as shown in **Figure 2.15(a, b)**. As shown in the IPF map of the β phase (**Figure 2.15(c)**), the volume fraction of retained- β in the bi-lamellar microstructure was much smaller than that in the lamellar microstructure. Nevertheless, the information of crystallographic orientation of retained- β was successfully obtained, as shown in **Figure 2.15(e)**. In order to investigate the crystallographic orientation relationship between the secondary α -lamellae and the retained- β , PFs for the α and β phases were drawn using the EBSD analysis, as shown in **Figure 2.15(d)** and **(e)**, respectively, which corresponded to the α and β phases in the IPF maps (**Figure 2.15(a)** and **(c)**). (0002) poles of the α phase and (110) poles of the β phase obviously coincided to each other. At the same time, each (111) pole of the β phase coincided with one of (11 $\bar{2}$ 0) pole of the α phase. These results indicate that the crystallographic orientation relationship between the retained- β and the secondary α -lamellae in the bi-lamellar microstructure maintains to the BOR. Even though the fine secondary α -lamellae were obtained through water quenching, different from the thick α -lamellae in the lamellar microstructure obtained through slow cooling, the BOR was maintained between secondary α -lamellae and retained- β . Moreover, the various crystallographic orientations of the secondary α -lamellae were found inside the transformed- β region. Therefore, the variant selection of the secondary α -lamellae with respect to the retained- β was investigated based on the BOR.

In order to investigate the variant selection of the secondary α -lamellae, the secondary α -lamellae in **Figure 2.15(a)** were separated, as shown in **Figure 2.16(a)**. In the same manner as that used for the lamellar microstructure, crystallographic orientations of the secondary α -lamellae were highlighted by specific colors in **Figure 2.16(b)**. Corresponding crystallographic orientations of the secondary α -lamellae were exhibited using the same colors in (0002) and (11 $\bar{2}$ 0) PFs, as shown in **Figure 2.16(c)**. At the same time, (110) and (111) PFs of the retained β phase corresponding to the IPF map shown in **Figure 2.15(c)** were represented in **Figure 2.16(d)**. (0002) and (11 $\bar{2}$ 0) PFs of the 12 possible variants of the α phase transformed from the β phase were superimposed on the (110) and (111) PFs of β phase. This result indicates that the 12 different crystallographic orientations of the secondary α -lamellae are precipitated inside the transformed- β .

In the previous section 2.3.3, the crystallographic features of the lamellar microstructure were revealed that the 12 variants of colonies can be formed within one prior- β grain and the α -lamellae keeps a particular BOR with thin retained β within an identical colony. It is believed that the crystallographic relationship between the α -lamellae and retained- β leads to the little resistance for dislocation glides at the interface, and the dislocation easily penetrates the interface between α -lamellae and retained- β . On the other hand, the crystallographic features of the bi-lamellar microstructure within one colony were found to include not only the equivalent crystallographic orientation of the primary α -lamellae maintaining the BOR against the retained- β but also 12 variants of secondary α -lamellae based on the BOR against identical retained- β in-between the primary α -lamellae. It is expected that the 12 variants of secondary α -lamellae in the bi-lamellar microstructure may act as a barrier for the dislocation movements

across the interface between primary α -lamellae and transformed- β , resulting in the different mechanical response with that of the lamellar microstructure.

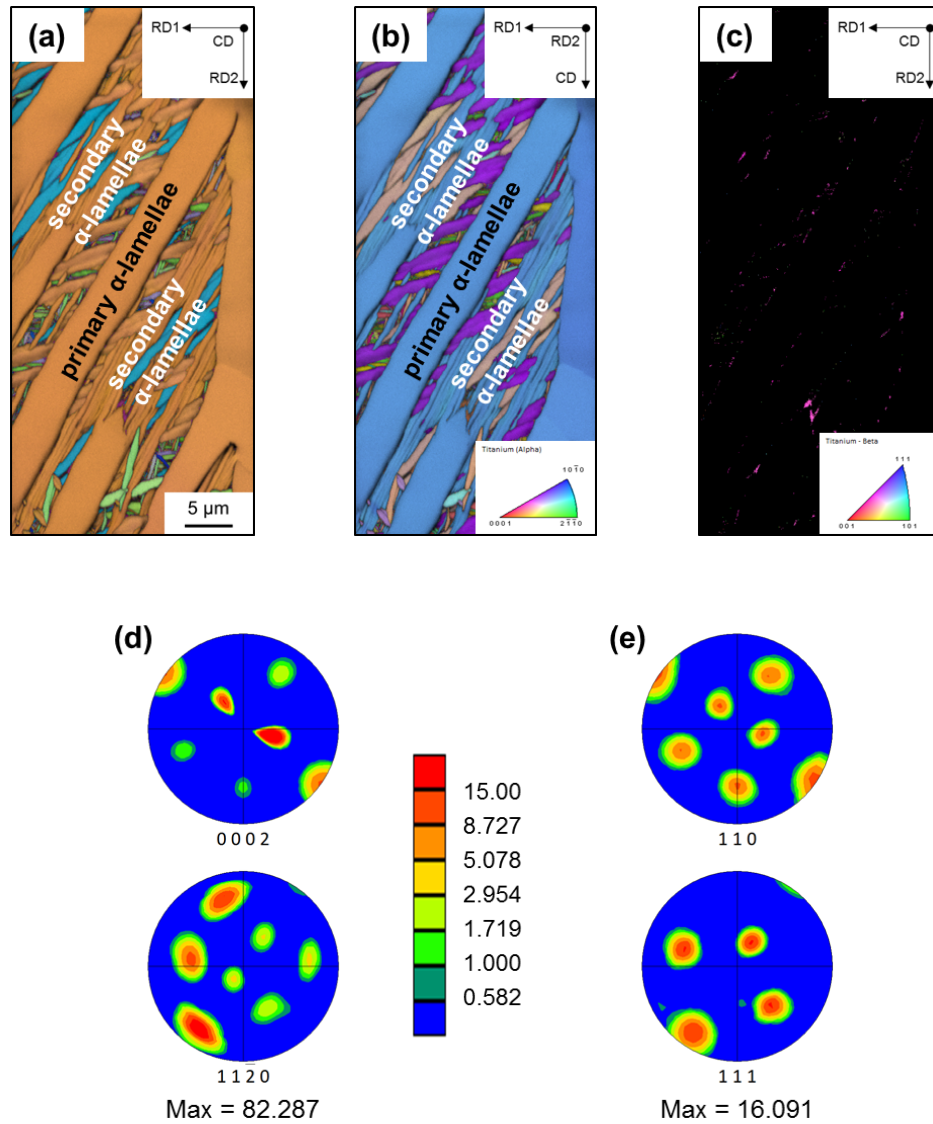


Figure 2.15 EBSD IPF maps of (a, b) α phase and (c) β phase in the bi-lamellar microstructure with the colony size of 57.5 μm . The colors in the IPF maps indicate crystallographic orientation (a) parallel to the compression direction (CD) in the specimen and (b) perpendicular to the CD. Pole figures (PFs) of (d) α phase and (e) β phase corresponding to (a) and (c), respectively.

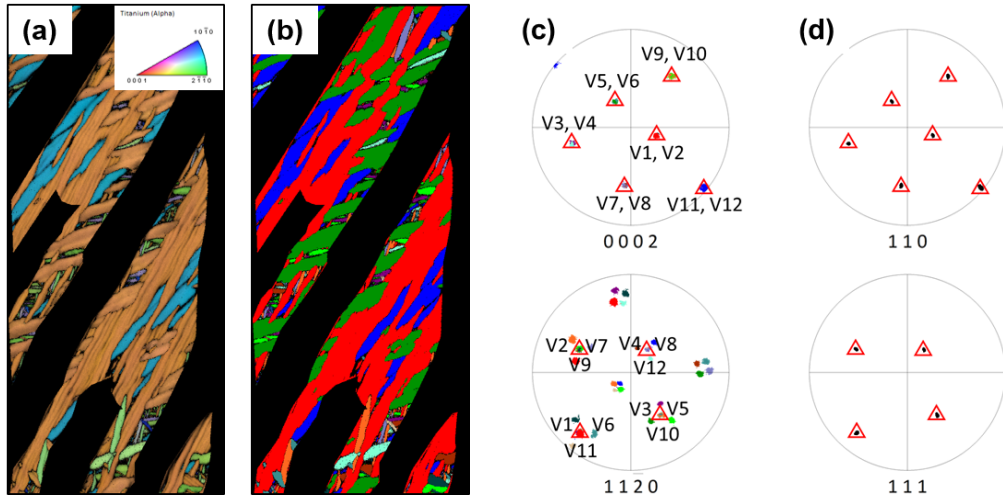


Figure 2.16 (a) EBSD IPF map of secondary α -lamellae, (b) EBSD highlighted map of secondary α -lamellae and corresponding pole figures (PFs) of (c) α phase and (d) β phase. Color of each pole corresponds to the crystallographic orientation of secondary α -lamellae highlighted in (b). Red triangle indicates the orientation of retained- β phase pole shown in Fig. 2.15 (c) and (e).

2.4. Conclusions

The different colony size in lamellar and bi-lamellar microstructures was obtained through different deformation conditions of the high temperature compression test. The microstructural features and the crystallographic features of the lamellar and the bi-lamellar microstructures were investigated using SEM/BSE and EBSD. The major results discussed in this chapter are summarized as follow:

1. Various colony sizes of lamellar microstructures were obtained through simple deformation process with various compressive strain at 1050 °C followed by slow cooling with $10^\circ\text{C min}^{-1}$ of cooling rate. The colony sizes were achieved ranging from 234.4 μm to 128.7 μm with increasing the compressive strain ranging from 0 to 1.2, but the colony size was saturated to

approximately 130 μm when the compressive strain was over 0.3.

2. The finer colony size than the colony size of lamellar microstructures manufactured by simple deformation process was obtained through multiple deformation process. The compression test was performed to 0.6 compressive strain at 700 °C and 1050 °C, respectively. It was found that β grain size of the multiple deformed specimen was much smaller than that in the simple deformed specimen. As a result, the colony size in the lamellar microstructure refined down to 57.5 μm .

3. Different colony sizes of bi-lamellar microstructures were obtained through inter-critical annealing at 920 °C followed by water quenching. Fine secondary α -lamellae was precipitated inside transformed- β region in-between primary α -lamellae. The different colony sizes of lamellar microstructures obtained through two different types of TMPs were used as initial microstructures, resultant in the various colony sizes in the bi-lamellar microstructures. The colony sizes in the bi-lamellar microstructures were considered to be equal to those in the initial lamellar microstructures, i.e., 234.4 μm , 128.7 μm and 57.5 μm .

4. The crystallographic orientation relationship between the retained- β and the α -lamellae in the lamellar microstructure was revealed to maintain the BOR having $\{0002\}_{\alpha} \parallel \{110\}_{\beta}$ and $\langle 11\bar{2}0 \rangle_{\alpha} \parallel \langle 111 \rangle_{\beta}$. The colony composed of the equivalent crystallographic orientation of α -lamellae was identified to have a total of 12 crystallographic orientations from one β grain. On the other hand, one colony in the bi-lamellar microstructure was composed of the additional 12

variants of secondary α -lamellae maintaining the BOR against identical retained- β in-between the primary α -lamellae.

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CHAPTER 3

MECHANICAL PROPERTIES OF LAMELLAR AND BI-LAMELLAR MICROSTRUCTURES WITH DIFFERENT COLONY SIZES

3.1. Introduction

The relationship between microstructural parameters and mechanical properties in lamellar microstructures of $\alpha + \beta$ dual phase titanium alloys has been intensively investigated for a long time. Grain refinement in metals is the most popular way to increase the strength of metals, according to Hall-Petch relationship [1, 2]. Even in lamellar microstructures of $\alpha + \beta$ dual phase titanium alloys, the Hall-Petch relationship can be applied as a function of the colony size instead of the grain size because colony boundaries can act as an obstacle for dislocation movement like grain boundaries. Eylon *et al.* [3-5] found that $\alpha + \beta$ dual phase titanium alloys with lamellar microstructures heat treated above the β transus temperature followed by relatively slow cooling possessed the best high cycle fatigue strength. Moreover, it was revealed that the refined colony size reduced growth rates of fatigue crack due to the crack propagation interruption at colony boundaries. Lütjering [6] investigated the effect of colony size on various mechanical properties in lamellar microstructures, such as yield strength, tensile ductility, fracture toughness, creep and fatigue crack nucleation. It was found that the smaller colony size in the lamellar microstructure was more beneficial for nearly all the mechanical properties. Especially in strengthening mechanism, the effective slip length in the lamellar microstructure is considered as the colony size, because lamellae within an identical colony have the same crystallographic orientation and keep a particular Burgers orientation relationship with thin retained β between α lamellae so that there is little resistance to

dislocation glides within the colony. Hence, it is believed that the smaller colony size in the lamellar microstructure is favorable for the strength.

On the other hand, Lütjering [6] reported that the strength in the bi-lamellar microstructure which composed of fine secondary α -lamellae within transformed β regions between primary α -lamellae has much higher strength than the lamellar microstructure. It was proved that fine secondary α -lamellae having different crystallographic orientations hindered slip transfer between primary α -lamellae and transformed β regions, and therefore effectively reduced the slip length rather than the colony size. However, the influence of microstructural parameters in the bi-lamellar microstructure (including the colony size) on mechanical properties has not been sufficiently studied yet due to the complex morphology of bi-lamellar microstructures. Hence, the present study aims to compare the mechanical properties of lamellar and bi-lamellar microstructures and to clarify the effect of the colony size on mechanical responses in lamellar and bi-lamellar microstructures.

3.2. Experiment procedure

3.2.1. Materials

The Ti-6Al-4V alloy specimens having lamellar microstructures with the colony size of 234.4 μm , 128.7 μm and 57.5 μm , which were obtained in Chapter 2, were used in this study to clarify the effect of colony size on tensile properties. The lamellar microstructures with the colony size of 234.4 μm and 128.7 μm were obtained from the simple deformation process (hot compression to a strain of 0 or 1.2 at 1050 $^{\circ}\text{C}$ followed by slow cooling at 10 $^{\circ}\text{C min}^{-1}$) shown in **Figure 2.3 (a)**. On the other hand, the lamellar microstructure with the colony size of 57.5

μm was obtained through the multiple deformation process (hot compression to a strain of 0.6 at 920 °C and subsequent hot compression to another 0.6 strain at 1050 °C (total strain of 1.2) followed by water quenching) shown in **Figure 2.3 (b)**. Corresponding bi-lamellar microstructures with the colony size of 234.4 μm , 128.7 μm and 57.5 μm were obtained through inter-critical annealing at 920 °C.

3.2.2. Nano indentation hardness test

Nano-hardness tests were carried out to compare the hardness of α -lamellae and retained- β in the lamellar microstructure, and also to compare the hardness of primary α -lamellae and transformed- β regions in the bi-lamellar microstructures. The specimen surfaces were prepared by chemical etching in a solution of 2 % hydrofluoric acid (HF), 2 % nitric acid (HNO_3) and 96 % distilled water at room temperature.

Specimens of lamellar and bi-lamellar microstructures with a colony size of 234.4 μm were examined by the nano-indentation (Ti-950 TriboIndenter, Hysitron *Inc.*) under a load of 500 μN and holding and unloading periods of 10s. The nano indentation test was performed on 20 $\times$ 20 grids with an interval of 0.3 μm using a Berkovich type tip as depicted in **Figure 3.1(a)**.

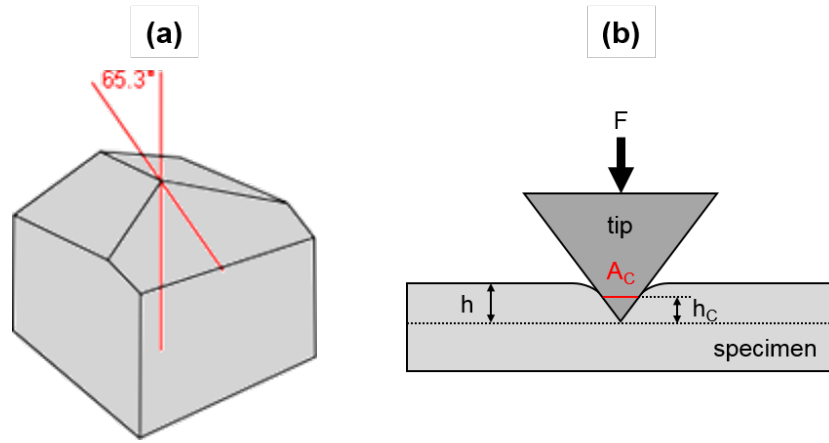


Figure 3.1 Schematic illustrations showing (a) Berkovich type nano-indentation tip and (b) nano-indentation test.

The hardness in the nano indentation is calculated by the following equation (3-1) [10].

$$H = \frac{P_{MAX}}{A_C} \dots\dots\dots (3-1)$$

where H , P_{MAX} and A_C denote the hardness, peak indentation load and contact area, respectively, as illustrated in **Figure 3.1(b)**. The contact area, A_C , can be calculated as a function of the contact depth, h_C , using the equation (3-2).

$$A_C = 24.5h_C^2 \dots\dots\dots (3-2)$$

The contact depth, h_C , can be determined by the following equation (3-3).

$$h_C = h_{MAX} - \epsilon \frac{P_{MAX}}{S} \dots\dots\dots (3.3)$$

where S is the stiffness and ϵ is a geometric constant of the tip, which is considered to 0.75 for the present case. Thus, when the material constants (S and ϵ) of the tip are determined, the nano-hardness can be estimated in the nano-indentation test.

3.2.3. Tensile test

In order to clarify the effect of colony size on mechanical properties in lamellar and bi-lamellar microstructures, tensile tests were carried out at room temperature at a strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$ using a uniaxial tensile machine (SHIMAZU AG-X plus). Micro-tensile specimens with 2 mm in gauge length, 1 mm of gauge width, and 0.5 mm of thickness, illustrated in **Figure 3.2**, were prepared. The specimens were sectioned from the center of the hot compressed specimens with the tensile direction perpendicular to the compression axis.

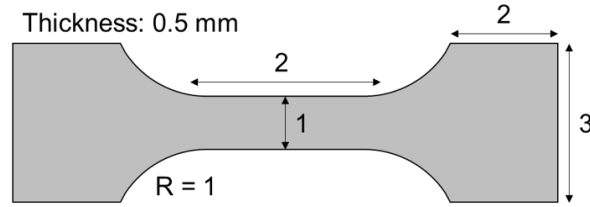


Figure 3.2 Schematic illustration of the micro-tensile test specimen.

The displacement of the gauge part during the tensile test was precisely measured using a digital image correlation (DIC) technique [7]. The surface of the micro-tensile specimen was painted in white first, using a spray paint gun and then particulate black-contrasts were sprayed to make displacement tracking spots randomly distributed, as shown in **Figure 3.3**. Sequential images of the micro-tensile specimen were captured during the tensile test using a charge-coupled device (CCD) camera at a frequency of 10 frames per second (10 fps). From the recorded digital images, a commercial DIC analysis software (Vic-2D™ 2009 powered by Correlated Solutions, Inc.) computed the displacement of the gage part, and then the tensile elongation was readily obtained. In order to estimate a strain rate sensitivity, m , the tensile tests

were also performed at various strain rates ranging from $1.6 \times 10^{-2} \text{ s}^{-1}$ to $1.6 \times 10^0 \text{ s}^{-1}$. The strain rate sensitivity was defined by the following equation (3.5).

$$m = \frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}} \dots\dots\dots (3.5)$$

where σ and $\dot{\epsilon}$ denote the true stress and strain rate, respectively.

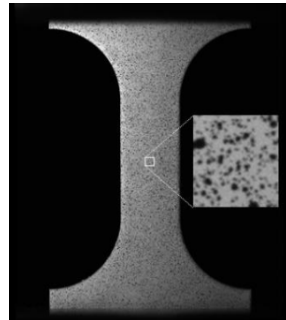


Figure 3.3 Preparation of the tensile specimen for digital image correlation (DIC).

3.3. Results and discussion

3.3.1. Nano-hardness of each phase in lamellar and bi-lamellar microstructures

In order to clarify the difference of mechanical properties of each microstructural component or region in the lamellar and bi-lamellar microstructures, the nano-hardness of α -lamellae and retained- β in the lamellar microstructure, and the nano-hardness of primary α -lamellae and transformed- β regions in the bi-lamellar microstructure were measured. The colony size of the measured lamellar and bi-lamellar microstructures was the largest size among the obtained colony sizes, $234.4 \mu\text{m}$. **Figure 3.4(a)** and **(b)** represent SEM images of the lamellar and the bi-lamellar microstructures, respectively. It was confirmed that the α -

lamellae in the lamellar microstructure and the primary α -lamellae in the bi-lamellar microstructure was etched in chemical polishing, while the retained- β in the lamellar microstructure and the transformed- β in the bi-lamellar microstructure convexly remained. Scanning probe microscopy (SPM) images represented with nano-hardness maps of the corresponding area of the lamellar and the bi-lamellar microstructures are shown in **Figure 3.4(c) and (d)**, respectively. In order to compare the nano-hardness in each phase, nano-hardness histograms in the lamellar and the bi-lamellar microstructures are displayed in **Figure 3.4(e) and (f)**, respectively. The average nano-hardness values of α -lamellae and retained- β in the lamellar microstructure were 4.01 GPa and 2.51 GPa, respectively. The hardness of the α -lamellae was obviously higher than that of the retained- β in the lamellar microstructure, as shown in **Figure 3.4(e)**. In the case of the bi-lamellar microstructure, the average nano-hardness values of primary α -lamellae and transformed- β regions were 4.05 GPa and 3.51 GPa, respectively. It was confirmed that the primary α -lamellae was harder than the transformed- β , as shown in **Figure 3.4(f)**. Comparing the nano-hardness between the lamellar and the bi-lamellar microstructures, the primary α -lamellae showed similar nano-hardness in two different microstructures. In the $\alpha + \beta$ titanium alloys, the solid solution strengthening by substitutional elements (i.e., Al in the Ti-6Al-4V alloy) is the major strengthening mechanism for the α phase [6]. From this result, however, it is considered that the Al element in the α -lamellae was not diffused during inter-critical annealing conducted at $\alpha + \beta$ temperature for obtaining the bi-lamellar microstructure. On the other hand, the nano-hardness of the transformed- β regions in the bi-lamellar microstructure was much higher than that of the retained- β in the lamellar microstructure. It is thought that secondary α -lamellae inside the transformed- β regions in the

bi-lamellar microstructure plays an important role to enhance the hardness of the transformed- β regions. Nano-hardness of the secondary α -lamellae within the transformed- β regions could not be identified due to their fine plate sizes. It is considered that the different hardness of microstructural components in the lamellar and the bi-lamellar microstructures lead to the different deformation responses.

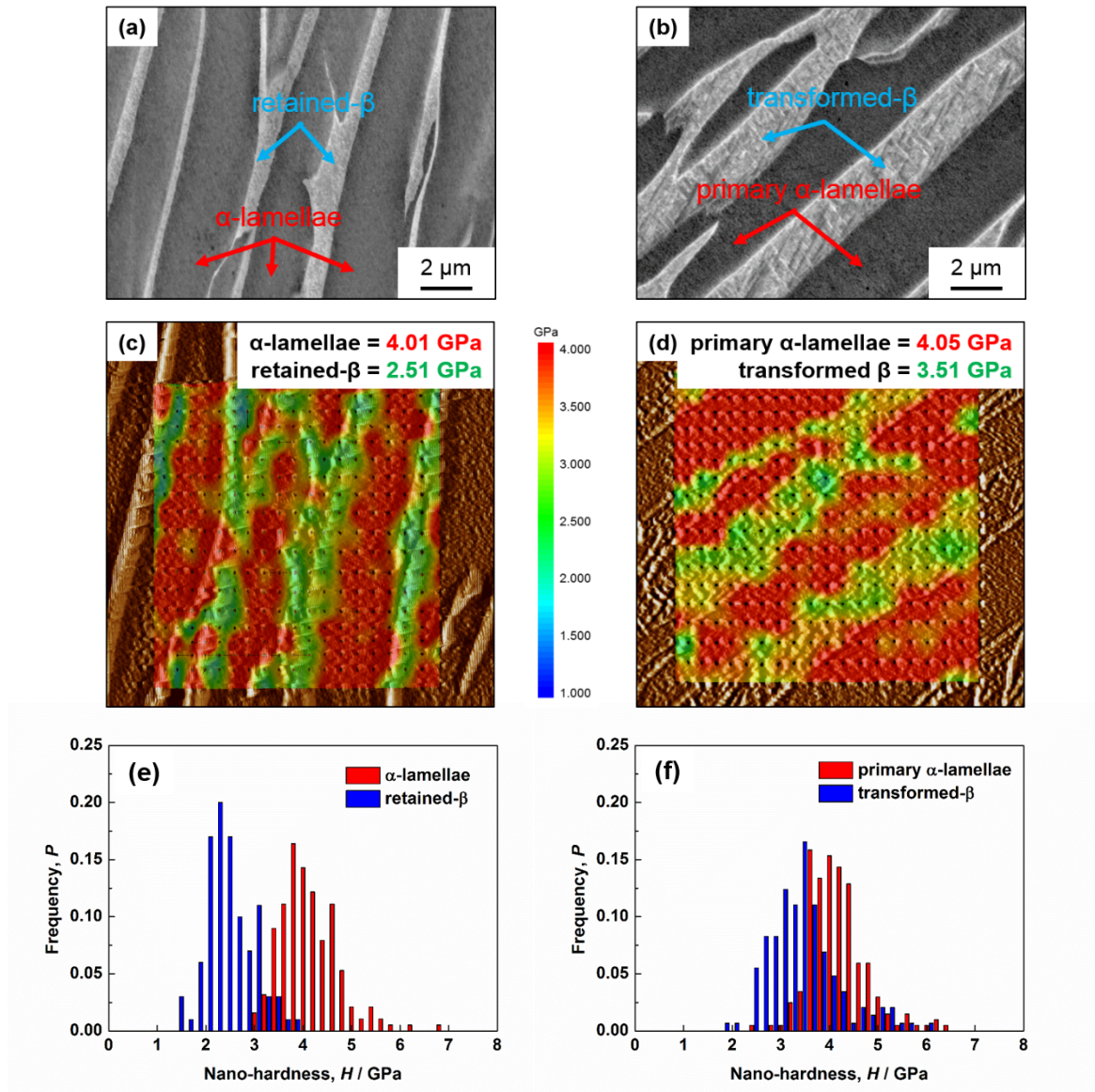


Figure 3.4 (a, b) SEM images, (c, d) scanning probe microscopy (SPM) images with superimposed nano-hardness maps, and (e, f) nano-hardness histogram for the lamellar microstructure (a, c, e), and the bi-lamellar microstructure (b, d, f).

3.3.2. Tensile properties in lamellar microstructures with different colony sizes

Nominal tensile stress-strain curves of the lamellar microstructures with different colony sizes of 234.4 μm , 128.7 μm and 57.5 μm are shown in **Figure 3.5**. The detailed tensile properties of the lamellar microstructures are summarized in **Table 3.1**. It was found that the

yield strength (YS) and the ultimate tensile strength (UTS) increased with decreasing the colony size. The YS in the lamellar microstructures with the colony size of 234.4 μm , 128.7 μm and 57.5 μm was 783.3 MPa, 845.8 MPa and 890.2 MPa, whereas the UTS was 827.3 MPa, 853.3 MPa and 914.6 MPa, respectively. The strain hardening capability which was evaluated from the difference between YS and UTS, was not different in all the colony sizes in the lamellar microstructure. On the other hand, the uniform elongation in the lamellar microstructure slightly increased with decreasing the colony size, while the total elongation had no specific tendency with respect to the colony size.

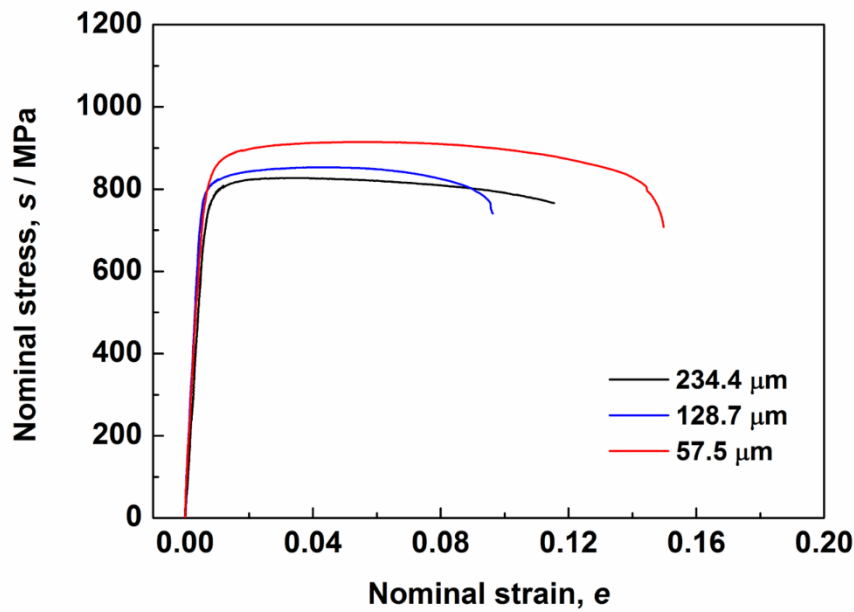


Figure 3.5 Nominal stress-strain curves obtained from the tensile test of the lamellar microstructures with the colony sizes of 234.4 μm , 128.7 μm , and 57.5 μm .

Table 3.1 Tensile properties of the lamellar microstructures with different colony sizes.

Colony size $d_{\text{colony}} / \mu\text{m}$	YS $\sigma_{\text{YS}} / \text{MPa}$	UTS $\sigma_{\text{UTS}} / \text{MPa}$	Total elongation ϵ_t	Uniform elongation ϵ_u	Post-uniform elongation ϵ_{pu}
234.4	783.3	827.3	0.108	0.030	0.078
128.7	845.8	853.3	0.103	0.049	0.054
57.5	890.2	914.6	0.149	0.094	0.055

The tensile strength (YS and UTS) in the lamellar microstructure was plotted as a function of inverse square root of the mean colony size in **Figure 3.6**. It was found that the flow stress followed a Hall-Petch relationship with respect to the colony size. The Hall-Petch relationship is generally expressed in the equation (3.4).

$$\sigma = \sigma_0 + k_d d^{-\frac{1}{2}} \dots\dots\dots (3.4)$$

where σ , σ_0 , k_d and d denote the strength, friction stress, Hall-Petch slope and mean grain size, respectively. The Hall-Petch relationship is well known to express strengthening by grain boundaries. It is noteworthy that the colony boundaries in the lamellar microstructure act as grain boundaries in typical polycrystalline microstructures of metallic materials. In the typical metallic materials which follow the Hall-Petch relationship, ultrafine grained materials often show limited ductility due to the occurrence of plastic instability at early stage of tensile deformation [8]. Interestingly, however, the total elongation in the lamellar microstructure did not decreased even though the colony size decreased, as shown in **Figure 3.7**. The uniform elongation gradually increased with decreasing the colony size, meanwhile the post-uniform elongation (i.e., elongation to fracture after necking) showed no specific tendency depending

on the colony size. Lütjering [4] reported a drastic decline of tensile elongation at higher cooling rates after heat-treatment in β regions (i.e., smaller colony sizes) due to the transition of fracture mechanism from a type with dimple fracture surfaces into a inter-crystalline dimple type fracture. Even in this study, it could be considered that the fracture mechanism was the main reason why the post-uniform elongation was independent with respect to the colony size in the lamellar microstructure. Hence, the relationship between the mechanical response and fracture mechanism in the lamellar microstructure will be discussed in **Chapter 4**.

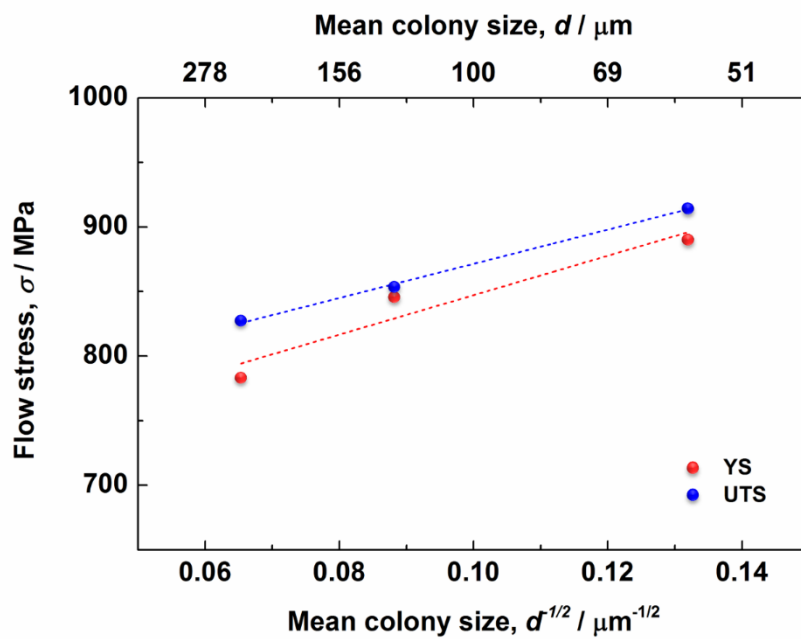


Figure 3.6 Yield strength (YS) and ultimate tensile strength (UTS) of the lamellar microstructures plotted as a function of the inverse square root of the mean colony size.

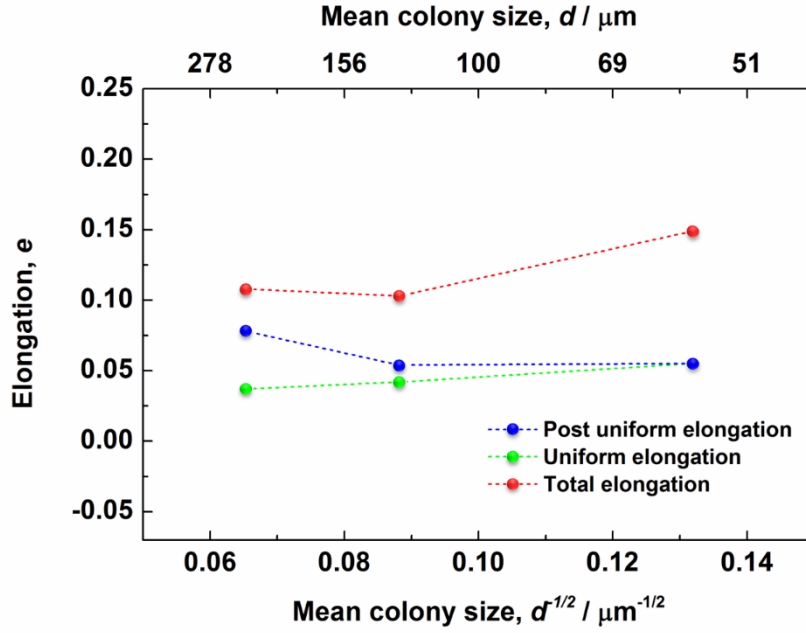


Figure 3.7 Changes of tensile elongations of the lamellar microstructures plotted as a function of the inverse square root of the mean colony size.

3.3.3. Tensile properties in bi-lamellar microstructures with different colony sizes

Nominal tensile stress-strain curves of the bi-lamellar microstructures with different colony sizes of 234.4 μm , 128.7 μm and 57.5 μm are plotted in **Figure 3.8**. The detailed tensile properties of the bi-lamellar microstructures are summarized in **Table 3.2**. YS in the bi-lamellar microstructure with the colony size of 234.4 μm , 128.7 μm and 57.5 μm was 1046.6 MPa, 994.3 MPa and 940.7 MPa, whereas UTS was 1103.4 MPa, 1116.1 MPa and 1085.7 MPa, respectively. The tensile strength of the bi-lamellar microstructures became obviously higher than that of the lamellar microstructures regardless of the colony size. This result is considered to be related to the introduction of the transformed- β regions composed of fine secondary α -lamellae with various crystallographic orientations. On the other hand, uniform elongation of the specimens with the colony size of 234.4 μm , 128.7 μm and 57.5 μm was 0.026, 0.054 and

0.093, whereas post-uniform elongation was 0.005, 0.051 and 0.098 corresponding to the colony size of 234.4 μm , 128.7 μm and 57.5 μm , respectively. Consequently, the total elongation of the bi-lamellar specimens greatly increased with decreasing the colony size.

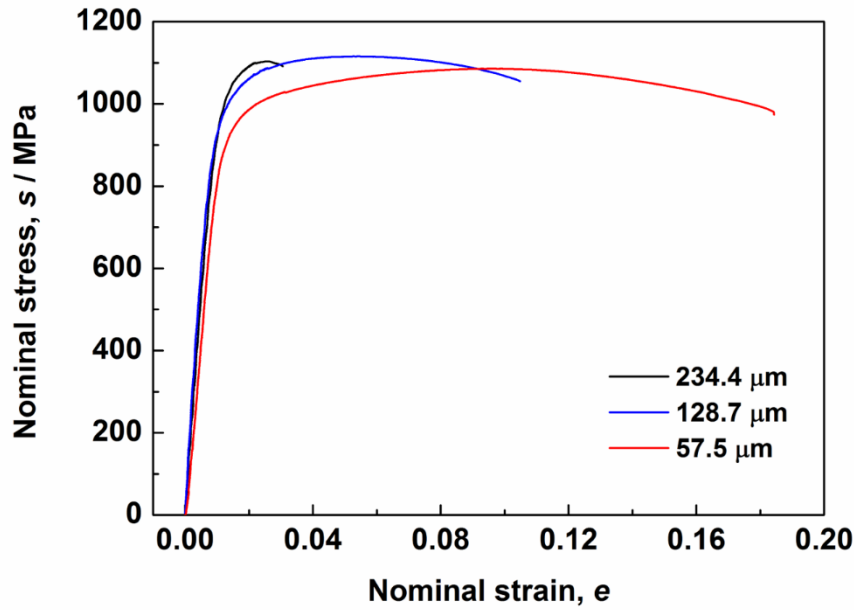


Figure 3.8 Nominal stress-strain curves of the bi-lamellar microstructures with the colony sizes of 234.4 μm , 128.7 μm , and 57.5 μm .

Table 3.2 Tensile properties of the bi-lamellar microstructures with different colony sizes.

Colony size $d_{\text{colony}} / \mu\text{m}$	YS $\sigma_{\text{YS}} / \text{MPa}$	UTS $\sigma_{\text{UTS}} / \text{MPa}$	Total elongation ϵ_t	Uniform elongation ϵ_u	Post-uniform elongation ϵ_{pu}
234.4	1046.6	1103.4	0.031	0.026	0.005
128.7	994.3	1116.1	0.105	0.054	0.051
57.5	940.7	1085.7	0.191	0.093	0.098

The strengths (YS and UTS) of the bi-lamellar microstructures were plotted as a function of inverse square root of the mean colony size in **Figure 3.9**. It was found that the variation of the flow stress with respect to the colony size did not follow the Hall-Petch relationship, different from the lamellar microstructures (**Figure 3.6**). The YS of the bi-lamellar microstructure gradually decreased with decreasing the colony size, while the UTS was nearly constant regardless of the colony size. This result indicated that the strain hardening capability, the difference between YS and UTS, increased with decreasing the colony size, which was in contrast to the case of the lamellar microstructure. Tensile elongation values of the bi-lamellar microstructures were plotted as a function of inverse square root of the mean colony size in **Figure 3.10**. Both uniform elongation and post-uniform elongation increased with decreasing the colony size, resulting in significantly increased total elongation with decreasing the colony size in the bi-lamellar microstructures. It was revealed that the effect of colony size on tensile elongation in the lamellar microstructure and the bi-lamellar microstructure was definitely different.

It is believed that the changes of the post-uniform elongation, i.e., the local elongation after necking, strongly depends on the strain rate sensitivity, m , which is also an important parameter to determine formability of materials [9-10]. Kim et al. [10] reported that ultrafine-grained Al alloy (AA8011) showed large post-uniform elongation which was attributed to the higher strain rate sensitivity than the coarse-grained specimens.

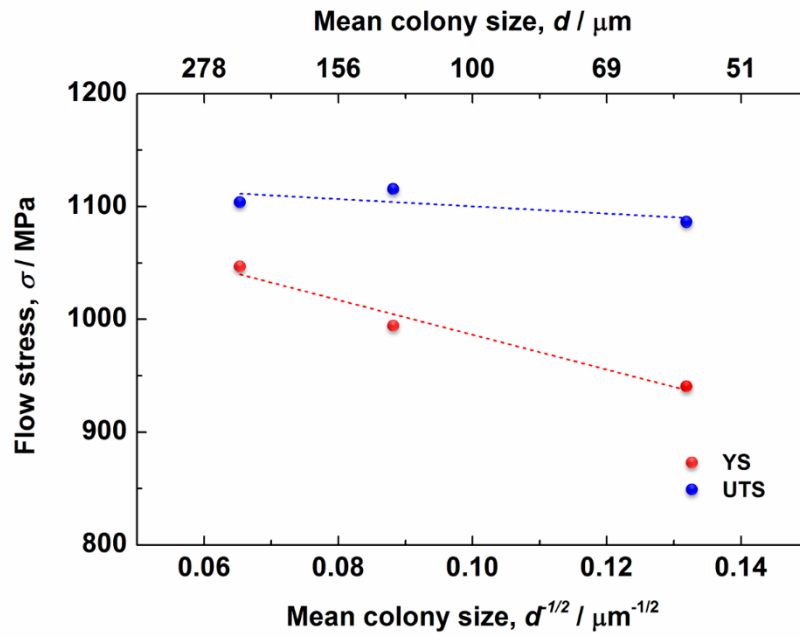


Figure 3.9 Yield strength (YS) and ultimate tensile strength (UTS) plotted as a function of the inverse square root of the mean colony size in the bi-lamellar microstructure.

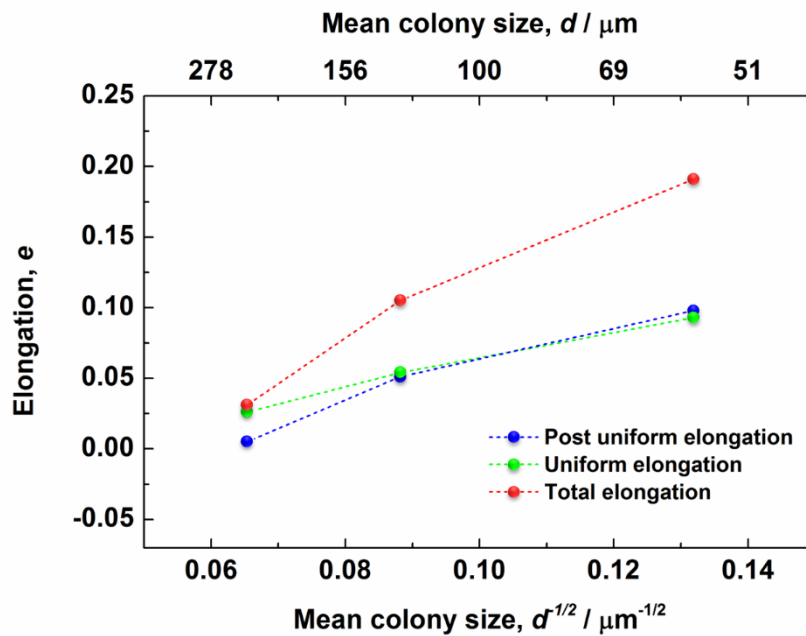


Figure 3.10 Changes of elongation values as a function of the inverse square root of the mean colony size in the bi-lamellar microstructure.

In order to understand the reason for the significantly increased post-uniform elongation with decreasing the colony size in the bi-lamellar microstructure, a strain rate sensitivity in the bi-lamellar microstructure with different colony sizes was investigated. Generally, the strain rate sensitivity is measured by changing the strain rate at a stage of uniform elongation during tensile test (i.e., strain rate jump test). However, since the stage of uniform elongation in the bi-lamellar microstructure was very short (**Figure 3.8**), the strain rate sensitivity in this study was measured by performing tensile tests at various strain rates ranging from $1.6 \times 10^{-2} \text{ s}^{-1}$ to $1.6 \times 10^0 \text{ s}^{-1}$. After that, the true stress at UTS was plotted as a function of the strain rate, as shown in **Figure 3.11**. According to the equation (3.5), the strain rate sensitivity could be defined as the slope in **Figure 3.11**. It was found that the strain rate sensitivity (m) was nearly identical for all the specimens and irrespective of the colony size. The strain rate sensitivity in the colony size of 234.4 μm and 128.7 μm was corresponding to 0.0408 and 0.0430, respectively. These results indicate that the reason for the increased post-uniform elongation in the bi-lamellar microstructure with decreasing the colony size should be attributed to another mechanism rather than increased strain-rate sensitivity. The reason for the increased post-uniform elongation by the colony size refinement in the bi-lamellar microstructure will be discussed from another viewpoint in **Chapter 4**.

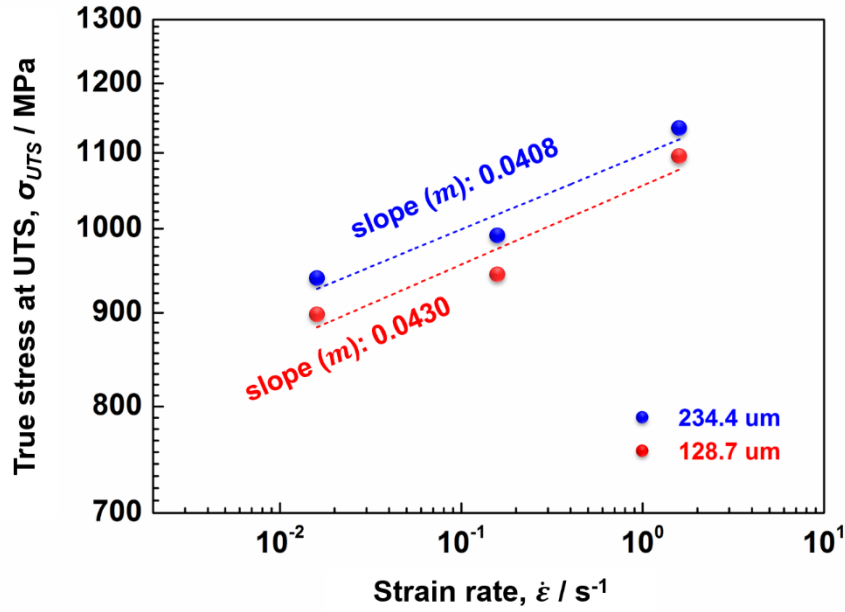


Figure 3.11 Double logarithmic true stress at UTS-Strain and strain rate plot to evaluate strain rate sensitivity (m) for the bi-lamellar microstructures with the colony sizes of 234.4 μm (blue dots and blue broken linear fitting line) and 128.7 μm (red dots and blue broken linear fitting line).

3.4. Conclusion

In this chapter, nano indentation test and uniaxial tensile test were performed to clarify the effect of colony size on tensile properties in lamellar and bi-lamellar microstructures. The results obtained in this chapter are summarized as follow:

1. Nano-hardness of microstructural components in the lamellar and bi-lamellar microstructures was measured using the nano indentation test. The hardness of α -lamellae was higher than that of thin retained- β in the lamellar microstructure. In the bi-lamellar microstructure, the primary α -lamellae was harder than the transformed- β regions. Comparing

the lamellar and the bi-lamellar microstructures, the hardness of primary α -lamellae was nearly identical in both microstructures, while the hardness of transformed- β regions composed of secondary α -lamellae in the bi-lamellar microstructure was higher than that of retained- β in the lamellar microstructure.

2. Mechanical properties of the lamellar microstructures with the average colony size of 234.4 μm , 128.7 μm and 57.5 μm were examined by room temperature uniaxial tensile test at a strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$. The strengths (yield strength, ultimate tensile strength) were enhanced with decreasing the colony size. The uniform elongation of the lamellar microstructure increased slightly with reducing the colony size, but the post-uniform elongation was nearly constant regardless of the colony size.

3. Mechanical properties of the bi-lamellar microstructures with the average colony size of 234.4 μm , 128.7 μm and 57.5 μm were examined by uniaxial tensile test at room temperature. All kinds of strength of the bi-lamellar microstructures was obviously higher than that in the lamellar microstructures. The uniform elongation and the post-uniform elongation of the bi-lamellar microstructure were greatly improved with decreasing the colony size.

4. In order to understand the reason for the improved post-uniform elongation with decreasing the colony size in the bi-lamellar microstructure, the strain rate sensitivity of the bi-lamellar microstructures was measured using tensile tests at various strain rates ranging from $1.6 \times 10^{-2} \text{ s}^{-1}$ to $1.6 \times 10^0 \text{ s}^{-1}$. The strain rate sensitivity of the bi-lamellar microstructure was measured

to be approximately 0.04, which was nearly identical regardless of the colony size. Hence, it was concluded that the strain rate sensitivity was not the reason for the increased post-uniform elongation of the bi-lamellar microstructures with decreasing the colony size in the Ti-6Al-4V alloy.

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CHAPTER 4

CHARACTERISTICS OF CRACK FORMATION IN LAMELLAR AND BI-LAMELLAR MICROSTRUCTURES WITH VARIOUS COLONY SIZES

4.1. Introduction

The effect of colony size on tensile properties in lamellar and bi-lamellar microstructures was investigated in **Chapter 3**. It was found that the post-uniform elongation in the lamellar microstructure was nearly constant regardless of the colony size, while that in the bi-lamellar microstructure was significantly improved with decreasing the colony size. Kim et al. [1] reported in their study on ultra-fine grained Al alloy that the large post-uniform elongation of ultrafine-grained Al alloy was attributed to the higher strain rate sensitivity than coarse-grained material. However, it was found that the strain rate sensitivity of the present Ti-6Al-4V alloy having the bi-lamellar microstructure was nearly constant ($m \sim 0.04$) even though the colony size decreased. These results indicated that the reason for the increased post-uniform elongation with decreasing the colony size in the bi-lamellar microstructure should be related to the fracture behavior. It is known that crack initiation takes place at initial stage of post-uniform elongation (i.e., necking), and crack propagation continues during the post-uniform deformation.

In this **Chapter 4**, the following two subjects are discussed. The formation of micro-crack in the lamellar and bi-lamellar microstructures with different colony sizes is firstly investigated

to understand the crack initiation behaviors. Then, the effect of colony size on following fracture behaviors including crack propagation is demonstrated to find out the reason for the different mechanical response, especially the post-uniform elongation, between the lamellar microstructure and the bi-lamellar microstructure with decreasing the colony size.

4.2. Experiment procedure

4.2.1. Materials

Tensile fractured specimens of Ti-6Al-4V alloy having the lamellar and bi-lamellar microstructures with different colony sizes of 234.4 μm , 128.7 μm and 57.5 μm , which were fabricated in **Chapter 2** were observed to characterize micro-crack formation including initiation and propagation.

4.2.2. Micro-crack observation

In order to characterize micro-crack formation, areas near the fracture surfaces of tensile fractured specimens were observed using field emission scanning electron microscopy (FE-SEM, JEOL-6700F). The specimens were mechanically polished by emery paper (#120 – 4000), and then electronically polished in a solution of 10 % perchloric acid (HClO_3) and 90 % methanol (CH_3OH) at -30 °C. Micro-cracks were categorized into four types based on the possible crack initiation site and possible crack propagation path in both lamellar and bi-lamellar microstructures schematically illustrated in **Figure 4.1**. The detailed descriptions are given below:

- **Type I:** elongated across α -lamellae (**Fig. 3.9(a)**)
- **Type II:** elongated along α -lamellae interface (**Fig. 3.9(b)**)
- **Type III:** elongated along colony boundary (**Fig. 3.9(c)**)
- **Type IV:** elongated along grain boundary- α (**Fig. 3.9(d)**)

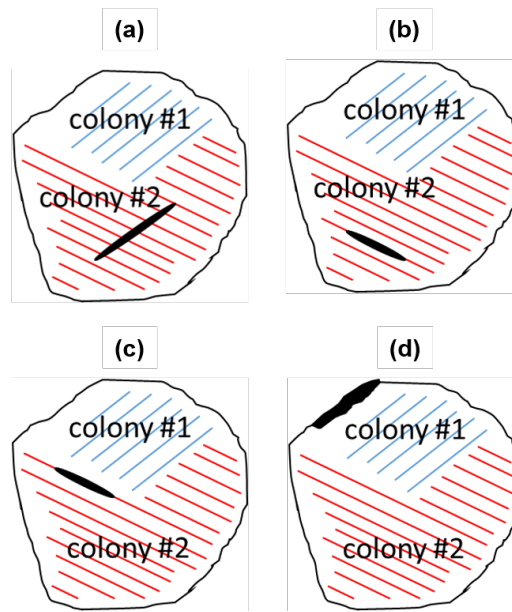


Figure 4.1 Schematic illustrations of the way for micro-crack classification. Each illustration indicates that the micro-crack elongated (a) across α -lamellae; Type I, (b) along α -lamellae interfaces; Type II, (c) along colony boundaries; Type III and (d) along grain boundary- α ; Type IV.

Over than 100 number of micro-cracks were classified based on the micro-crack classification in **Figure 4.1**, and statistically analyzed to quantitatively compare the formation of micro-crack in the lamellar and bi-lamellar microstructures.

4.2.3. Main-crack characterization

Main-cracks of the fractured specimens were observed using EBSD to find out the crack propagation behavior and the crystallographic orientation dependence against the adjacent colonies in the lamellar and bi-lamellar microstructures. In order to prohibit the damage in the main-crack during the mechanical polishing, the fracture surfaces of the specimens were protected using a conductive cold mounting resin, a mixture of dendritic copper powder (Technovit 5000, Kulzer GmbH) and methyl methacrylate ($C_5H_8O_2$), as shown in **Figure 4.2**. The mounted specimens were mechanically polished by emery paper (#120 – 4000), and then electronically polished in a solution of 10 % perchloric acid ($HClO_3$) and 90 % methanol (CH_3OH) at $-30\text{ }^{\circ}C$. Main-cracks in the lamellar and bi-lamellar microstructures were exhibited by inverse pole figure (IPF) map using commercial TSL OIM Analysis software (Orientation Imaging Microscopy v7, EDAX Inc.).

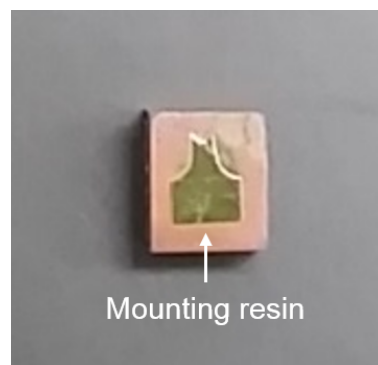


Figure 4.2 Tensile fractured specimen protected by conductive cold mounting resin.

4.2.4. Micro-DIC technique in microstructure

In order to understand the interrelation between microscopic tensile deformation behavior and crack formation in the lamellar and the bi-lamellar microstructures, a micro-DIC technique was applied. The tensile specimens were electronically polished prior to the tensile test to obtain clear microstructure images by SEM. In order to compute the strain distribution in the microstructures, the displacement tracking spot was imprinted as a square-shaped grid by focused ion beam (FIB) with Ga source, as shown in **Figure 4.3**. Park [2] confirmed that the FIB grids are well recognized in the DIC software. Thereafter, the tensile test was performed and interrupted at different strain levels for obtaining the SEM images. The strain distribution in the lamellar and the bi-lamellar microstructures with the colony size of $57.5\text{ }\mu\text{m}$ was investigated by comparing the images of non-deformed and deformed microstructures using commercial DIC analysis software.

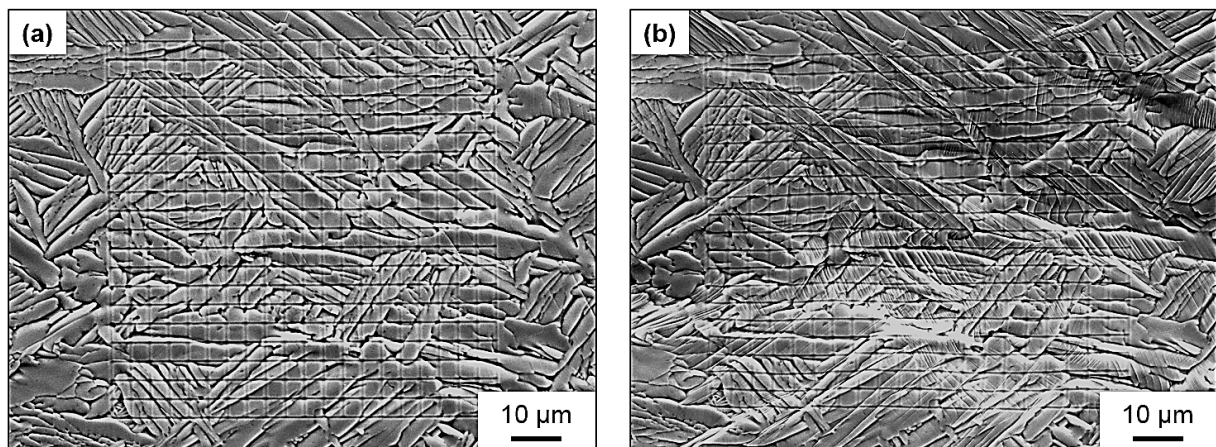


Figure 4.3 SEM images of specimens (a) non-deformed and (b) deformed to 6.7 % strain during the tensile test in lamellar microstructure with colony size of $234.4\text{ }\mu\text{m}$.

4.3. Results and discussion

4.3.1. Characterization of micro-crack formation in lamellar microstructure

Different types of micro-crack formation in lamellar microstructures were given in **Figure 4.4**. It was found that micro-cracks in lamellar microstructures were elongated (a) across α -lamellae; Type I, (b) along α -lamellae interface; Type II and (c) along colony boundary; Type III, respectively. The crack formation was classified into four types which were indicated in **Figure 4.1**, but the crack formation was not observed at the grain boundary- α (Type IV) in all the colony size of the lamellar microstructure. The observed micro-cracks propagated independently or with combining each other. **Figure 4.5** indicates the micro-crack propagation combined by the type I micro-crack and the type II micro-crack. It was found that the crack propagation path was diverted when the crack formation type was changed.

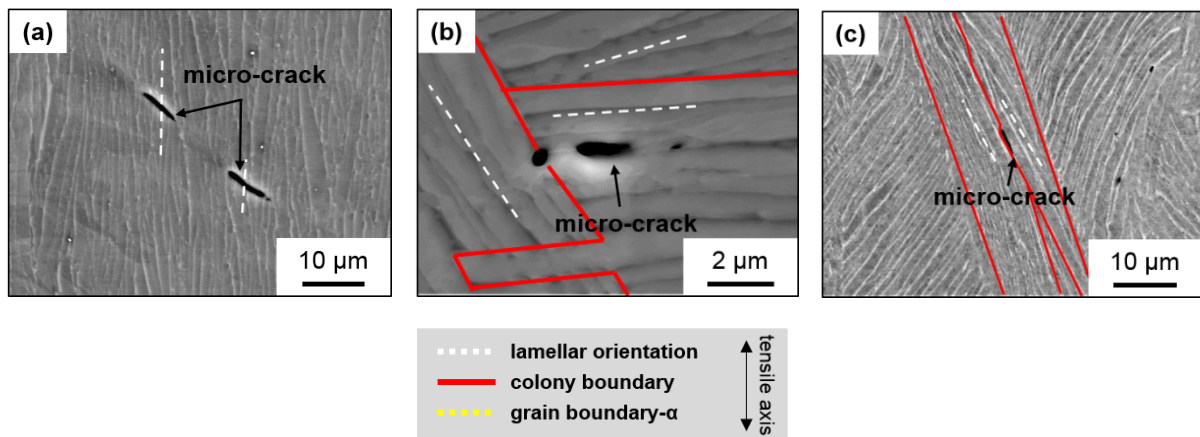


Figure 4.4 SEM images of micro-crack formation in lamellar microstructure. The micro-cracks elongated (a) across α -lamellae; type I, (b) along α -lamellae interfaces; type II and (c) along colony boundary; type III.

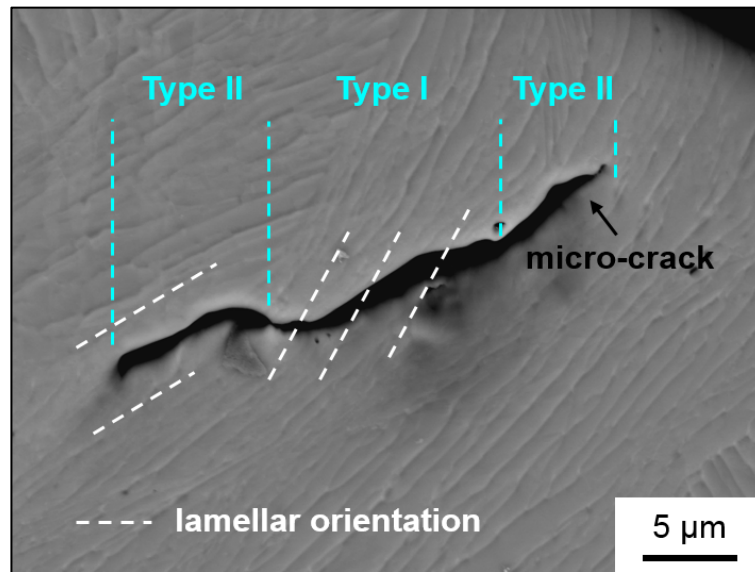


Figure 4.5 SEM image of micro-crack propagation in lamellar microstructure combined by type I (across alpha lamellae) and type II (along lamellae interface).

In order to compare the micro-crack formation in the lamellar microstructures with respect to the colony size, the micro-cracks observed by SEM were statistically analyzed in terms of average length and number fraction in each type of micro-crack formation. **Figure 4.6(a)** and **(b)** exhibit the histograms for average length of micro-cracks and for number fraction of micro-cracks in accordance with their formation types in the lamellar microstructures with different colony sizes, respectively.

Type I micro-crack (elongated across α -lamellae) was measured having the largest average length among the three micro-crack formation types. It was found that the average length of type I micro-crack decreased with decreasing the colony size. The average length of the type I micro-crack in the lamellar microstructures with the colony size of 234.4 μm , 128.7 μm and 57.5 μm was 11.6 μm , 7.9 μm and 5.7 μm , respectively. On the other hand, type II micro-crack (elongated along α -lamellae interface) was found to be the smallest average length,

approximately 1.6 μm . This result indicates that the micro-cracks difficult to elongate (i.e., crack propagation) along the α -lamellae interface. In addition, average length of type III micro-crack (elongated along colony boundary) was 5.2 μm , 4.8 μm and 2.1 μm in the lamellar microstructures with the colony size of 234.4 μm , 128.7 μm and 57.5 μm , respectively.

Number fraction in all the type of micro-crack formation in the lamellar microstructure was shown in **Figure 4.6(b)**. It was found that a large number of micro-cracks were observed at the α -lamellae interface (type II). On the other hand, type I micro-crack were observed to be the least number fraction among the three types of micro-crack formation in the lamellar microstructure. The number fraction of each micro-crack formation was irrelevant to the colony size.

From the results of the largest average length and the smallest number fraction in the type II micro-crack, it can be inferred that micro-cracks easily initiate at the α -lamellae interfaces, but difficult to propagate along the α -lamellae interface. Whereas, the largest average length and the least number fraction of the type I micro-crack indicate that the micro-cracks easily grow across the α -lamellae. This tendency of the crack formation was similar in all the colony size in the lamellar microstructure. Consequently, it could be concluded that the micro-crack formation in the lamellar microstructure is the same irrelevant to the colony size, and micro-cracks preferentially initiate at the α -lamellae interfaces and then predominantly propagate across the α -lamellae.

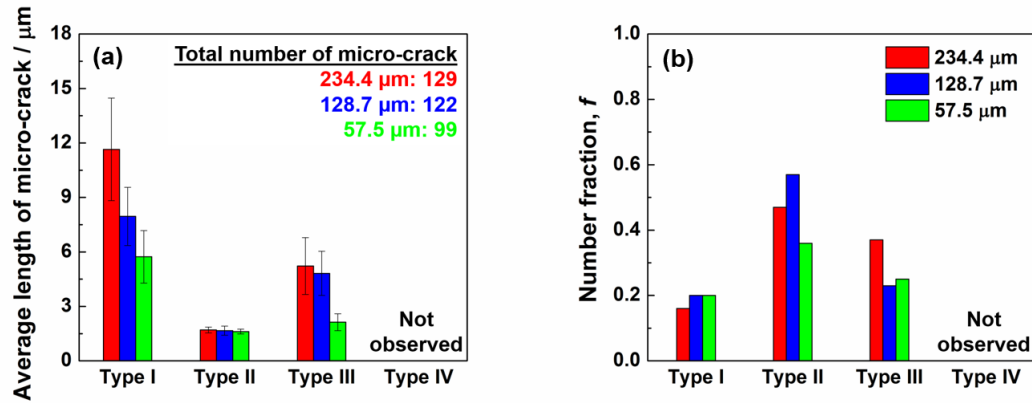


Figure 4.6 Histograms with respect to (a) average length and (b) number fraction of micro-cracks in lamellar microstructures with different colony size. The colony size of 234.4 μm , 128.7 μm and 57.5 μm indicated by red column, blue column and green column, respectively.

4.3.2. Characterization of main-crack formation in lamellar microstructure

Micro-crack formation in the lamellar microstructure was quantitatively characterized in the previous section. Since the micro-crack formation of metallic materials is an early stage of cracking (i.e., crack initiation), there is a limit to perfectly understand the origin of global crack propagation behavior. Hence, the crack propagation behavior of Ti-6Al-4V alloy having lamellar microstructure was investigated by taking the EBSD at the main-crack of tensile fractured specimens. The observed main-cracks in the lamellar microstructure with the colony size of 234.4 μm and 57.5 μm are shown in **Figure 4.7(a)** and **(b)**, respectively. Tensile axis is the vertical direction of the images. The white arrows within the EBSD images indicate the crack propagation path. As concluded from the result in **Figure 4.6**, the crack path in the lamellar microstructure predominantly propagates across the α -lamellae (type I). Moreover, the crack was found to propagate across colony boundary, but immediately be diverted toward other direction, as indicated by the red arrow in **Figure 4.7(a)**. On the other hand, the main-

crack in the lamellar microstructure with the colony size of 57.5 μm was observed to propagate not only across α -lamellae (type I) but also along colony boundary (type III). This result is considered to be due to the increased number of the colony boundaries in the smaller colony size. These results could be concluded that the crack for failure in the lamellar microstructure predominantly propagates across the α -lamellae but the colony boundary also act as an another crack propagation path in the smaller colony size. In order to more effectively understand the fracture mechanism in the lamellar microstructure, the fractograph in the fractured specimen with the lamellar microstructure having the colony size of 234.4 μm was observed, as shown in **Figure 4.8**. It was found that the fractograph of the observed specimen was mostly flat and covered by dimple patterns. In general, the flat fracture surface is accounted as evidence for the brittle fracture accompanied by transgranular fracture which means the crack propagates toward internal of the grains. Nevertheless, the fractograph of the present alloy having the lamellar microstructure was mostly covered by the dimple pattern which is the representative trace for ductile fracture. Consequently, the fracture mechanism of lamellar microstructure is considered as the ductile fracture accompanied by transgranular-like fracture.

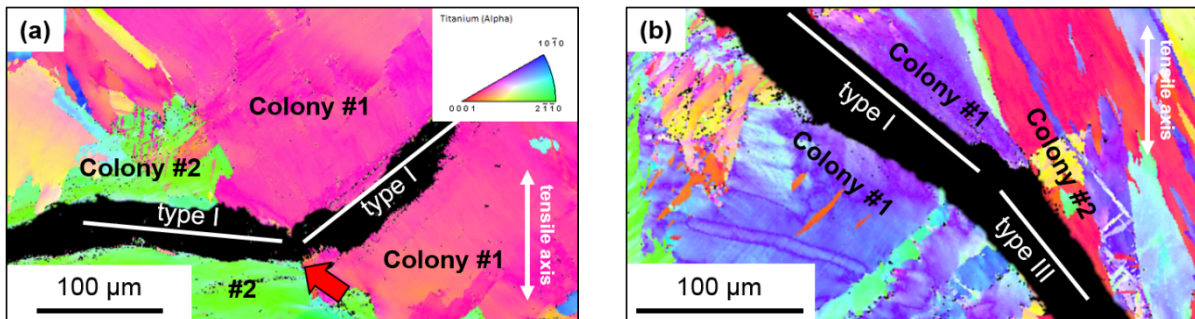


Figure 4.7 EBSD IPF maps of main-cracks in lamellar microstructures with colony sizes of (a) 234.4 μm and (b) 57.5 μm .

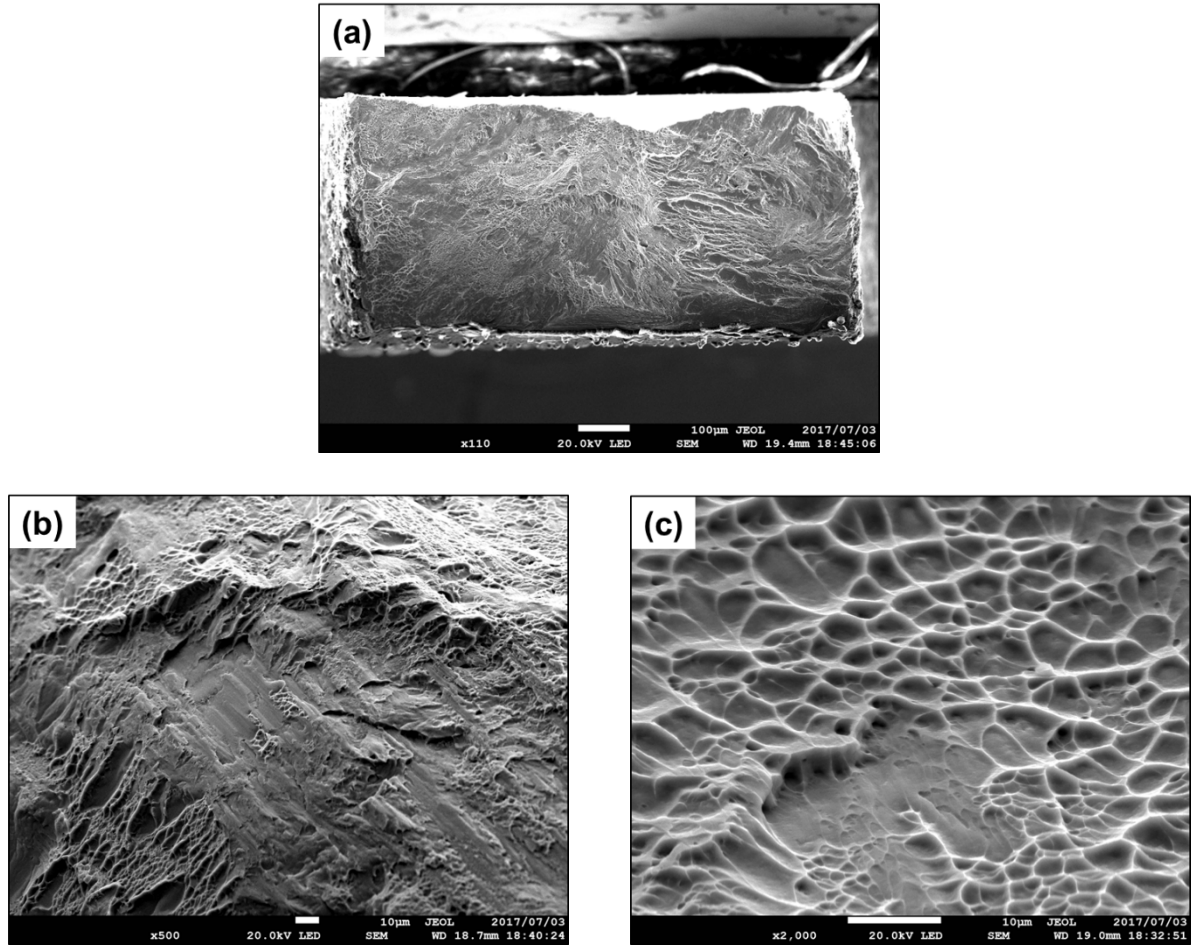


Figure 4.8 SEM images showing the (a) fractographs of tensile fractured specimen having the lamellar microstructure with the colony size of 234.4 μm . (b) flat surface and (c) dimple pattern.

In order to investigate the crystallographic orientation relationship against the colonies near the main-crack, a slip plane trace analysis was applied. Slip planes of the α phase in the titanium alloy are well known as $\{0002\}$ basal plane, $\{10\bar{1}0\}$ prismatic plane and $\{11\bar{2}0\}$ pyramidal plane [3]. Since the basal and prismatic slip planes can be activated most easily, the basal and the prismatic slip planes traces in the colonies near the main-crack were observed. The interrelation between the slip plane traces and the main-crack was investigated by measuring the angle between them. **Figure 4.9** shows the histograms for the frequency of

angles between slip plane traces and main-crack. The angles of the basal plane trace and the prismatic plane trace are corresponding to the red column and the blue column, respectively. It was found that the angle of prismatic plane traces showed a higher frequency of the angle ranging from 0° to 20° and the angle of basal plane traces were widely distributed ranging from 0° to 90° . In other words, the prismatic plane traces were nearly parallel to the main-crack, while the basal plane traces were irrespective of the main-crack. Biroasca and his coworkers [4] reported that crack propagation in a Ti-6246 alloy with lamellar microstructure is parallel to basal plane traces when the crack propagates across α -lamellae. In the present study, however, the prismatic plane trace considered as the major slip plane for the deformation in the Ti-6Al-4V alloy was obviously nearly parallel to the main-crack. The crystallographic orientation dependence of the main-crack is an another representative evidence for the transgranular type of fracture. Hence, it can be concluded based on the result of fractograph and main-crack observation that the fracture in the lamellar microstructure is certainly accompanied by transgranular-like fracture.

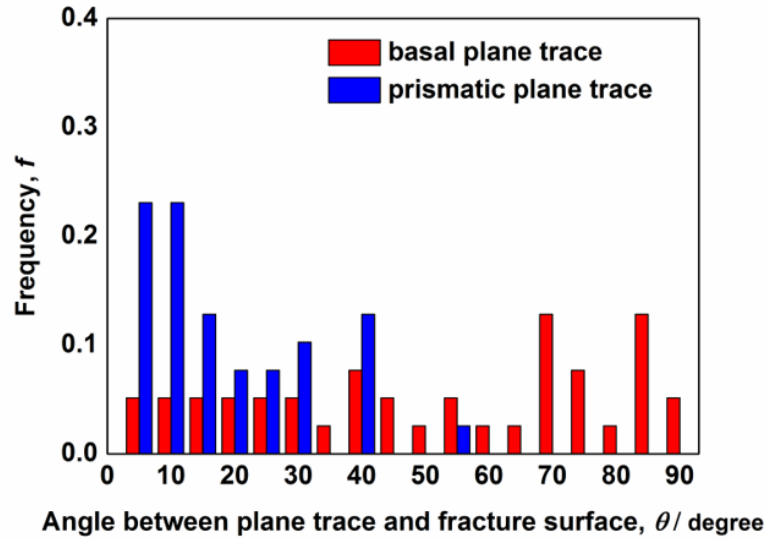


Figure 4.9 SEM images of micro-crack formation in bi-lamellar microstructure. The micro-cracks elongated (a) along α -lamellae; type II, (b) along colony boundary; type III and (c) along grain boundary- α ; type IV.

4.3.3. Relationship between crack propagation and deformation behavior in lamellar microstructure

It was revealed that main-crack in lamellar microstructure propagated predominantly across α -lamellae and diverted when the crack reached colony boundary. Crystallographic relationship between the main-crack and adjacent colonies at the main-crack were statistically analyzed by slip plane trace analysis. It was found that prismatic plane traces were nearly parallel to the main-crack. Since the prismatic plane is the major slip plane in the Ti-6Al-4V alloy for the deformation, it is expected that the crack propagation in the lamellar microstructure might be related to deformation behavior. **Figure 4.10** shows plastic strain maps obtained by the micro-DIC technique in the lamellar microstructure with the colony size of $57.5 \mu\text{m}$ at the applied strain condition of (a) 0 %, (b) 6.7 % and (c) 8.7 % expressed globally

in von-Mises equivalent strain ranging from 0 to 0.25. The strain maps were overlapped with the tensile-deformed SEM images. It was revealed that plastic strain was concentrated in several regions, as shown in **Figure 4.10(b)**. Larger plastic strain can be identified red colored region in the strain maps. The plastic strain was gradually propagated with increasing the applied strain. It was noted that the plastic strain propagates across α -lamellae and colony boundaries (black dotted lines), as indicated by the red colored arrow in **Figure 4.10(b)** and **(c)**. This tendency of microscopic strain propagation is a good agreement with the tendency of the main-crack propagation behavior in the lamellar microstructure. **Figure 4.11** shows SEM image of the slip transfer in the lamellar microstructure. It was obviously observed that the slip line appeared across α -lamellae and colony boundary during the tensile deformation.

As we confirmed in **Chapter 2**, the α -lamellae and the retained- β share the habit plane along $\{0002\}_{\alpha} \parallel \{110\}_{\beta}$ based on the BOR. It means that the α -lamellae and the retained- β within the identical colony are semi-coherent and leads to the easier slip transfer which means the slip takes place across the α -lamellae. The deformation behavior in the lamellar microstructure of titanium alloys was reported by several researchers to be derived by a slip transfer behavior [3, 5-8]. These results can be good evidence for the reason why main-crack in the lamellar microstructure propagate across α -lamellae predominantly.

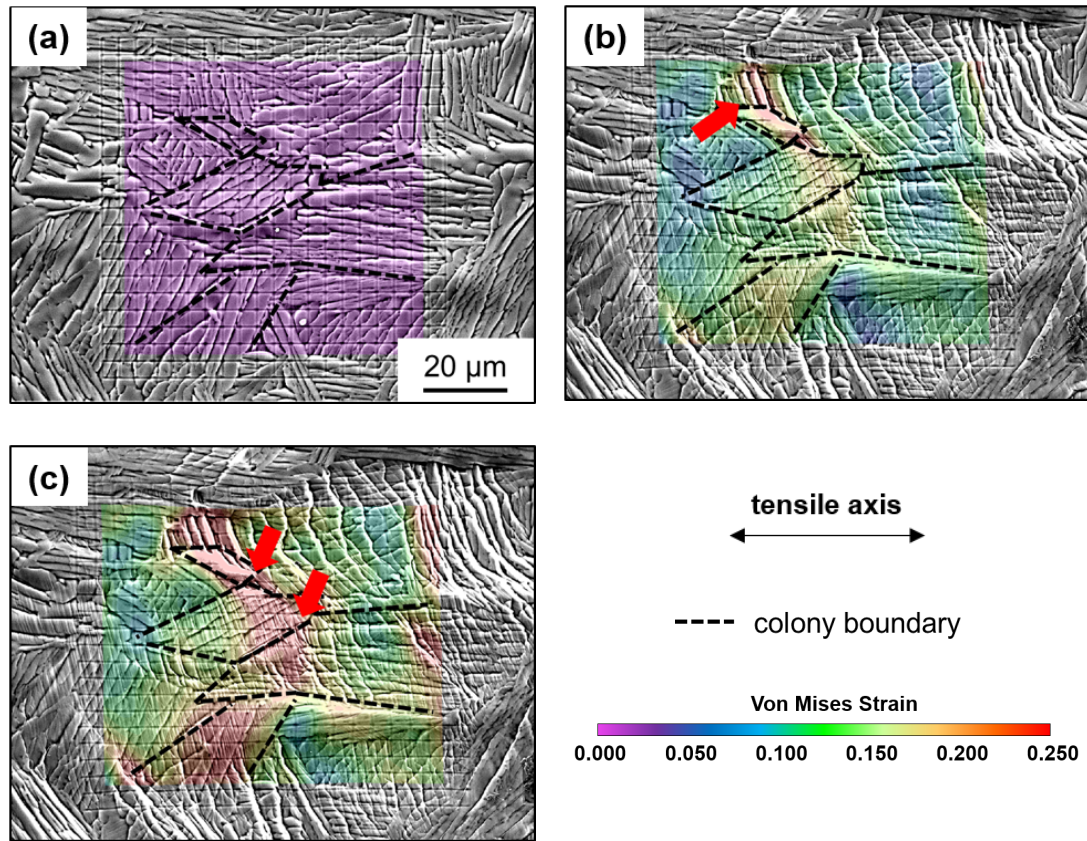


Figure 4.10 Equivalent strain maps obtained through micro-DIC technique of (a) 0 %, (b) 6.7 % and (c) 8.7 % tensile-deformed lamellar microstructure with the colony size of 57.5 μm .

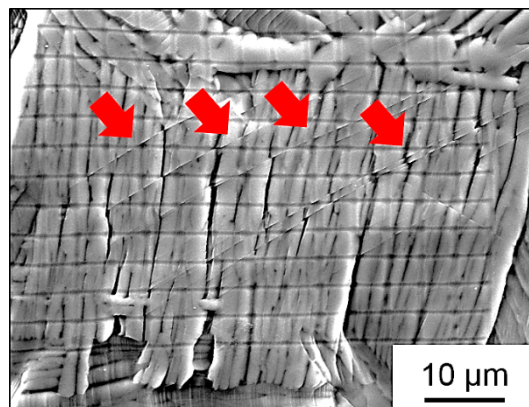


Figure 4.11 SEM image of the slip transfer in the lamellar microstructure. Red colored arrows indicate slip lines.

4.3.4. Characterization of micro-crack formation in bi-lamellar microstructure

Different types of micro-crack formation in bi-lamellar microstructures were given in **Figure 4.12**. It was found that micro-cracks in bi-lamellar microstructures were elongated (a) along α -lamellae (hereafter primary α -lamellae) interface; Type II, (b) along colony boundary; Type III and (c) along grain boundary- α ; Type IV, respectively. Contrary to the micro-crack formation in the lamellar microstructure, type I micro-crack (elongated across α -lamellae) was not observed in all the colony size of the bi-lamellar microstructure. This result represents that the micro-crack formation behavior in the lamellar microstructure and the bi-lamellar microstructure is different. It is considered that secondary α -lamellae in-between primary α -lamellae in the bi-lamellar microstructure act as an obstacle to prohibit the crack propagation across α -lamellae.

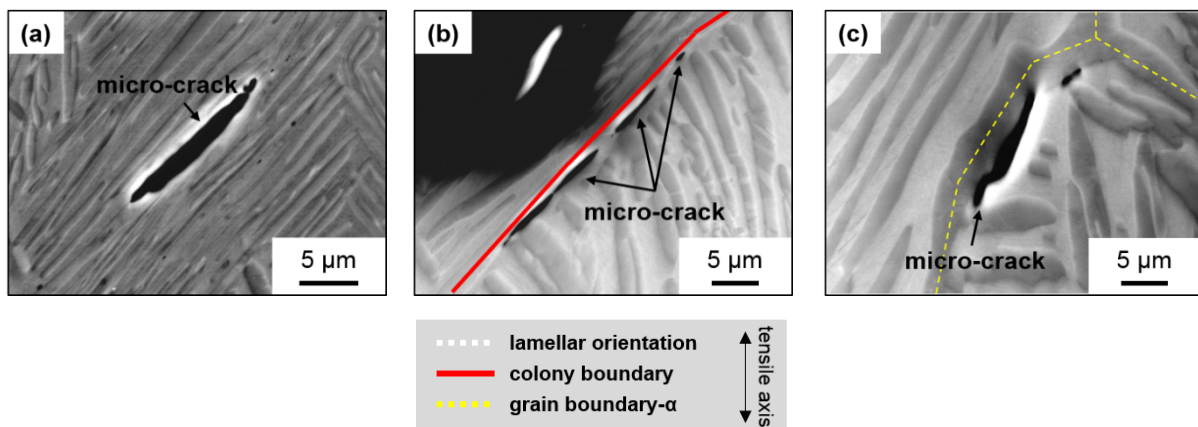


Figure 4.12 SEM images of micro-crack formation in the bi-lamellar microstructure. The micro-cracks elongated (a) along α -lamellae; type II, (b) along colony boundary; type III and (c) along grain boundary- α ; type IV.

In order to compare the micro-crack formation in the bi-lamellar microstructures with respect to the colony size, the micro-cracks in the bi-lamellar microstructures were statistically analyzed in the same manner with the lamellar microstructure. **Figure 4.13(a)** and **(b)** exhibit the histograms for average length of micro-cracks and for number fraction of micro-cracks in accordance with the formation types in the bi-lamellar microstructures with different colony sizes, respectively.

Type II micro-crack (elongated along primary α -lamellae interface) was measured having the smallest average length among the three micro-crack formation types. The average length of the type II micro-crack in the colony size of 234.4 μm , 128.7 μm and 57.5 μm was nearly the same which was approximately 2 μm . On the other hand, the average length of type III and type IV micro-crack showed a similar value but longer than the type I micro-crack. The average length of all the micro-crack type in the bi-lamellar microstructure seemed to be smaller than that of the type I micro-crack in the lamellar microstructure, which was considered as a predominant crack propagation path in the lamellar microstructure. This result indicates that the micro-crack in the bi-lamellar microstructure more difficult to elongated (i.e., crack propagation) than that in the lamellar microstructure.

Number fraction in all the type of micro-crack formation in the bi-lamellar microstructure was shown in **Figure 4.13(b)**. It was found that a number of micro-cracks were observed at the primary α -lamellae interface (type II). This tendency was observed in the lamellar microstructure, as well. From the results of the smallest average length and the largest number fraction in the type II micro-crack which elongated along the primary α -lamellae interface, it can be inferred that micro-cracks easily initiate at primary α -lamellae interfaces, but difficult

to propagate along the primary α -lamellae interface. Contrary to the lamellar microstructure, however, the number fraction of the type II micro-crack in the bi-lamellar microstructure drastically decreased with decreasing the colony size. On the other hand, the number fraction of type III micro-crack which elongated along colony boundary increased with decreasing the colony size. In particular, type III micro-crack was not observed in the largest colony size of the bi-lamellar microstructure. These results indicate that the micro-cracks in the bi-lamellar microstructure with larger colony size preferentially initiate at the primary α -lamellae interfaces. With decreasing the colony size, however, the micro-cracks in the bi-lamellar microstructure seems to initiate at not only the primary α -lamellae interfaces but also colony boundaries. Moreover, it is considered that micro-cracks propagate along the primary α -lamellae interface, colony boundary and grain boundary- α , because the average length of type I, type II and type II micro-cracks was nearly similar.

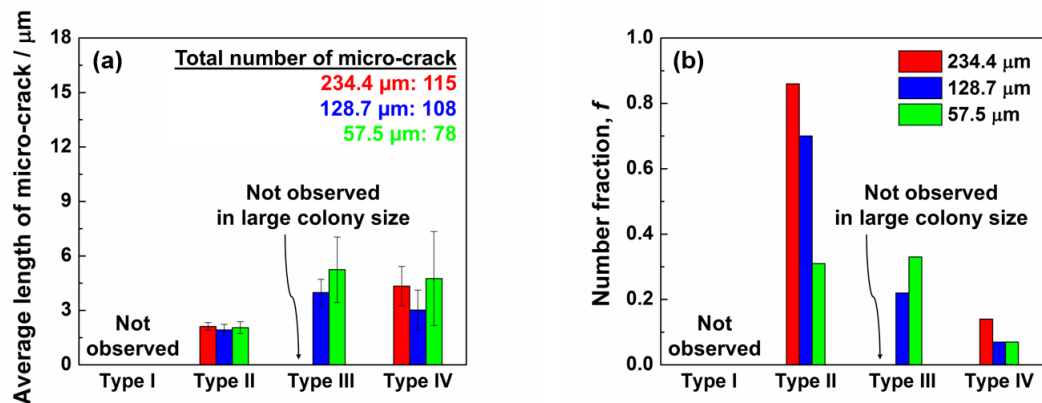


Figure 4.13 Histograms with respect to (a) average length and (b) number fraction of micro-cracks in the bi-lamellar microstructures with different colony size. The colony size of 234.4 μm , 128.7 μm and 57.5 μm indicated by red column, blue column and green column, respectively.

4.3.5. Characterization of main-crack formation in bi-lamellar microstructure

The crack propagation behavior of Ti-6Al-4V alloy having bi-lamellar microstructure was investigated by taking the EBSD in the main-crack of tensile fractured specimens. The observed main-cracks in the bi-lamellar microstructure with the colony size of 234.4 μm and 57.5 μm are shown in **Figure 4.14(a)** and **(b)**, respectively. Tensile axis is the vertical direction of the images. The white arrows within the EBSD images indicate the crack propagation path. Crack propagation in the bi-lamellar microstructure was expected to propagate along the primary α -lamellae interface (type II), colony boundary (type III) and grain boundary- α (type IV) from the result of **Figure 4.13**. However, it was revealed that main-cracks in the bi-lamellar microstructures with the colony sizes of 234.4 μm and 57.5 μm propagate only along the colony boundaries, as shown in **Figure 4.14(a)** and **(b)**. Unfortunately, however, it is difficult to distinguish the colony boundary or the grain boundary- α at the main-crack of the fractured specimen because the boundary morphology (such a grain boundary- α) is damaged. Hence, the colony boundary and the grain boundary- α would be considered as the same microstructural parameter, boundary components, hereafter. The main-crack propagation path diverted when the crack reached the boundary components, as indicated by the red arrow in **Figure 4.14**. The boundary components were the predominant main-crack propagation path in the bi-lamellar microstructure regardless of the colony size. In the same manner with the lamellar microstructure, the fractograph of fractured specimen having the bi-lamellar microstructure with the colony size of 234.4 μm was observed, as shown in **Figure 4.15**. Contrary to the fractograph of the lamellar microstructure, the fractograph of bi-lamellar microstructure was more rugged due to the intergranular fracture-like morphologies. Even if the fractograph was

nearly similar with the intergranular fracture, the fractograph was mostly covered by the dimple patterns. Consequently, the fracture mechanism of the bi-lamellar microstructure is considered as the ductile fracture accompanied by intergranular-like fracture.

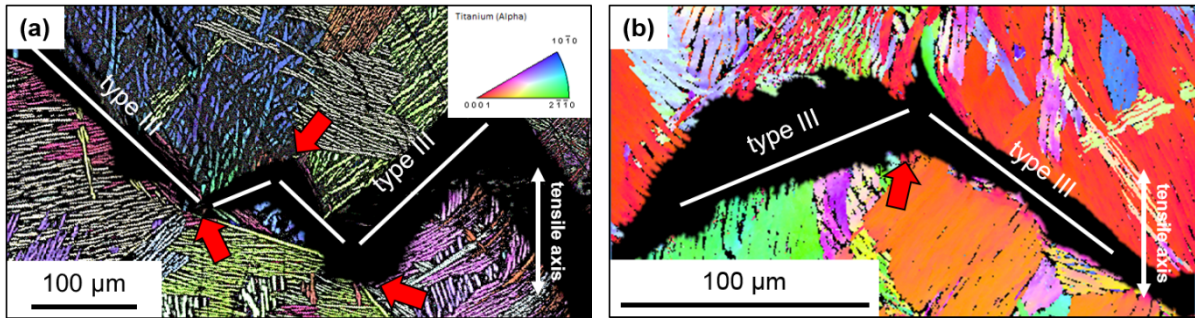


Figure 4.14 EBSD IPF maps of main-cracks in bi-lamellar microstructures with colony sizes of (a) 234.4 μm and (b) 57.5 μm.

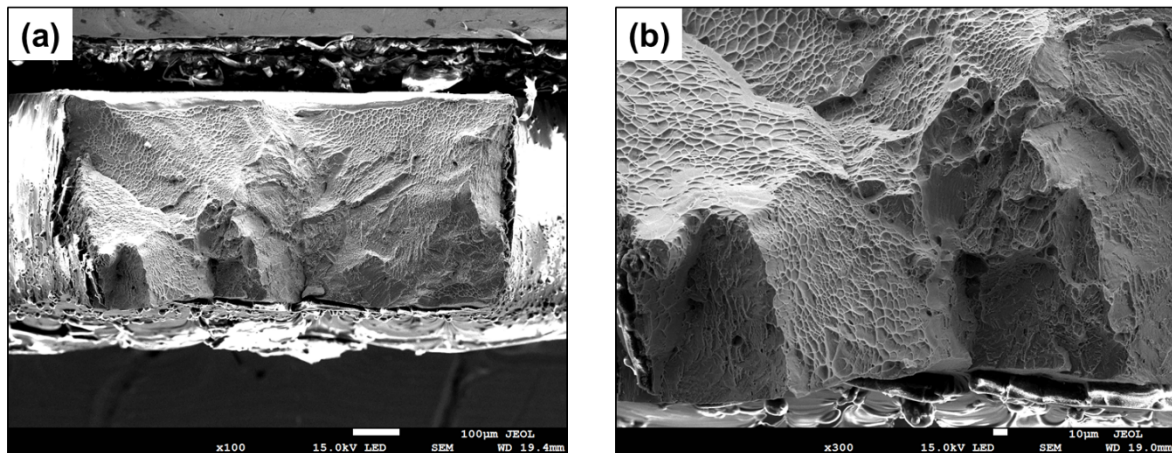


Figure 4.8 SEM images showing the (a) fractographs of tensile fractured specimen having the bi-lamellar microstructure with the colony size of 234.4 μm. (b) intergranular-like fracture surface.

The crystallographic orientation relationship between the colonies near main-crack and the main-crack in the bi-lamellar microstructure was investigated in the same manner as that in the

lamellar microstructure. **Figure 4.16** shows histograms for the frequency of the angles between the slip plane traces and the main-crack. The angles of the basal plane trace and the prismatic plane trace are corresponding to the red column and the blue column, respectively. It was found that the angular ranges of the prismatic plane traces and the basal plane traces were widely distributed ranging from 0° to 70° and from 0° to 90° , respectively. The frequency of angular range in both basal and prismatic plane traces did not show higher frequency in any particular angular range, contrary to that in the lamellar microstructure. In other words, the main-crack in the bi-lamellar microstructure is independent with respect to the crystallographic orientation of the adjacent colonies at the main-crack. This result is a typical characteristic of intergranular fracture which means the fracture takes place along the grain boundaries in metallic materials. Hence, the fracture mechanism of the bi-lamellar microstructure in the Ti-6Al-4V alloy is considered as the intergranular-like fracture based on the fractograph observation and crystallographic orientation dependence of the main-crack.

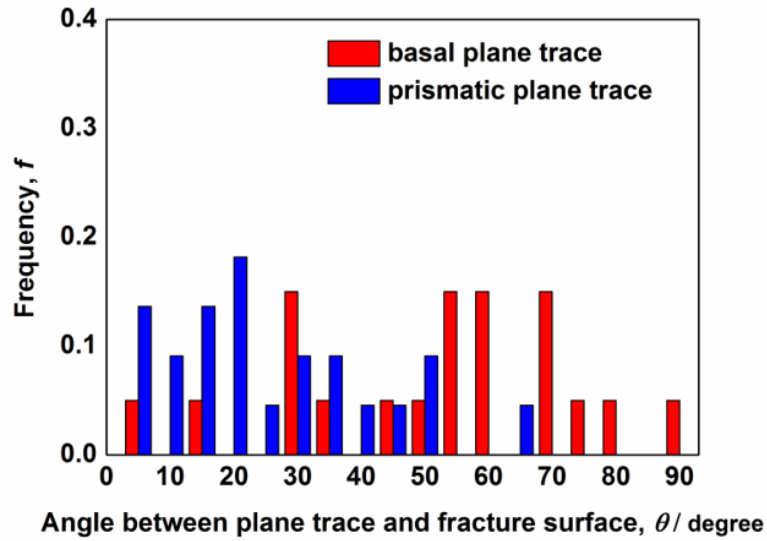


Figure 4.16 SEM images of micro-crack formation in bi-lamellar microstructure. The micro-cracks elongated (a) along α -lamellae; type II, (b) along colony boundary; type III and (c) along grain boundary- α ; type IV.

4.3.6. Relationship between crack propagation and deformation behavior in bi-lamellar microstructure

Main-crack in bi-lamellar microstructure was expected to propagate along α -lamellae and boundary components (colony boundary and grain boundary- α), intergranular-like fracture. Hence, it can be expected that the crack initiation and propagation in the bi-lamellar microstructure take place by stress or strain concentration. **Figure 4.17** shows plastic strain maps obtained by the micro-DIC technique in the bi-lamellar microstructure with the colony size of $57.5 \mu\text{m}$ at the applied strain condition of (a) 0 %, (b) 5.8 % and (c) 9.9 % expressed in von-Mises equivalent strain ranging from 0 to 0.25. It was confirmed that the plastic strain distributed heterogeneously. A number of regions were hardly deformed to the plastic strain larger than 0.25. It was noted that the plastic strain propagates along the longitudinal direction

of the α -lamellae, however, plastic propagation did not pass over the colony boundaries, as indicated by the red colored arrow in **Figure 4.17(c)**. In order to confirm the plastic strain distribution between the primary α -lamellae and the transformed- β , the micro-DIC technique was applied in the higher magnification SEM image of the tensile specimen, as shown in **Figure 4.18(a)**. The SEM image was taken at the tensile strain 2.6 % (just after macroscopic necking). The obtained microstructure is the bi-lamellar microstructure with the colony size of 234.4 μm . Equivalent plastic strain profiles obtained from line analysis (red colored line in **Figure 4.18(a)**) is shown in **Figure 4.18(b)**. It was obviously confirmed the plastic strain was concentrated in the interface between the primary α -lamellae and the transformed- β . The reason for this result is considered to be due to the prohibited slip transfer at the primary α -lamellae interface, as shown in **Figure 4.19**. As confirmed in **Chapter 2**, various crystallographic orientation (i.e., 12 variants) of the secondary α -lamellae within the transformed- β can effectively hinder the slip transfer between secondary α -lamellae and retained- β . Hence, the plastic deformation in the bi-lamellar microstructure is no longer progressed by slip transfer and the plastic strain becomes to concentrate along the primary α -lamellae and/or the boundary components. This tendency of microscopic strain propagation can be the reason why the main-crack propagated along the primary α -lamellae and the boundary components in the bi-lamellar microstructure.

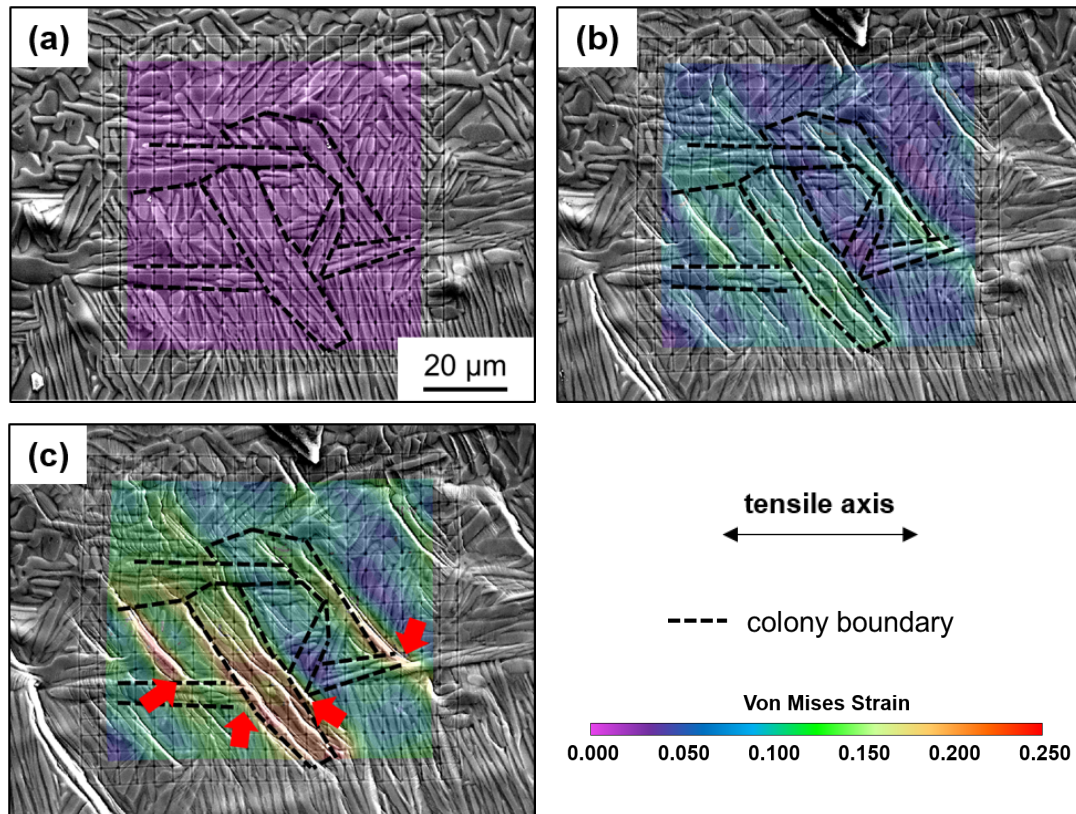


Figure 4.17 Equivalent strain maps obtained through micro-DIC technique of (a) 0 %, (b) 5.8 % and (c) 9.9 % tensile-deformed bi-lamellar microstructure with the colony size of 57.5 μm.

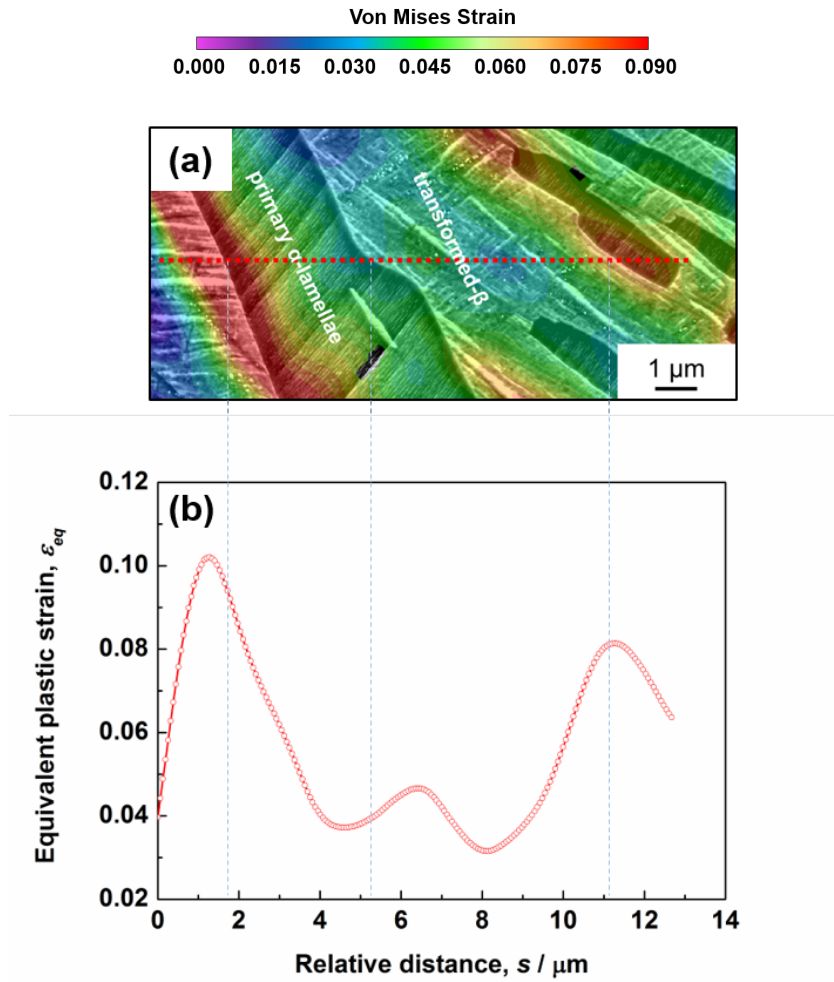


Figure 4.18 (a) Equivalent strain map of bi-lamellar microstructure in higher magnification of SEM image and (b) line profiles of equivalent strain obtained by DIC analysis.

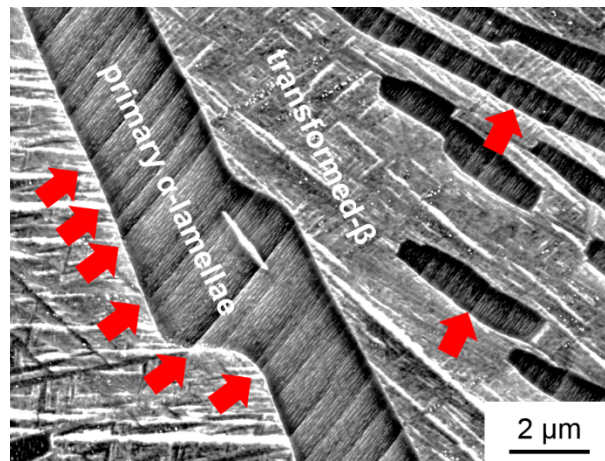


Figure 4.19 SEM image of the interrupted slip transfer in the bi-lamellar microstructure.

4.3.7. Effect of colony size on mechanical properties and crack propagation in lamellar and bi-lamellar microstructures

Based on the results shown in the previous section, the author would like to suggest one possibility with respect to the relationship between fracture mechanism and tensile property (results of **Chapter 3**) in Ti-6Al-4V alloy having lamellar and bi-lamellar microstructures with different colony sizes. It is considered that the different fracture mechanisms in the lamellar and bi-lamellar microstructures lead the different effects on post-uniform elongation during tensile deformation. The crack initiation in ductile materials is well known as owing to extreme plastic deformation at necking part of specimens. After that, the magnitude of post-uniform elongation depends on the crack propagation resistance or the void growth rates of the specimens. In the present case, the interrelation between the fracture mechanism and the effect of colony size in the lamellar and bi-lamellar microstructures on post-uniform elongation was discussed by considering the crack propagation length to an equivalent variant as a void growth rate.

It was revealed that the micro-cracks in the lamellar microstructures with various colony sizes initiated at the α -lamellae interfaces. Moreover, the main-crack in the lamellar microstructure propagated across the α -lamellae and the colony boundaries with parallel to $\{10\bar{1}0\}$ slip plane. **Figure 4.20(a)** and **(b)** present schematic illustrations of the crack initiation and propagation in the lamellar microstructures with larger and smaller colony sizes, respectively. Since the crack in the lamellar microstructure propagates across not only the α -lamellae but also the colony boundaries, the crack propagation lengths for the failure in the larger colony size and in the smaller colony size are not affected or less affected by microstructural components. This results can be the reason why the post-uniform elongation in the lamellar microstructure was nearly constant even in the smaller colony size.

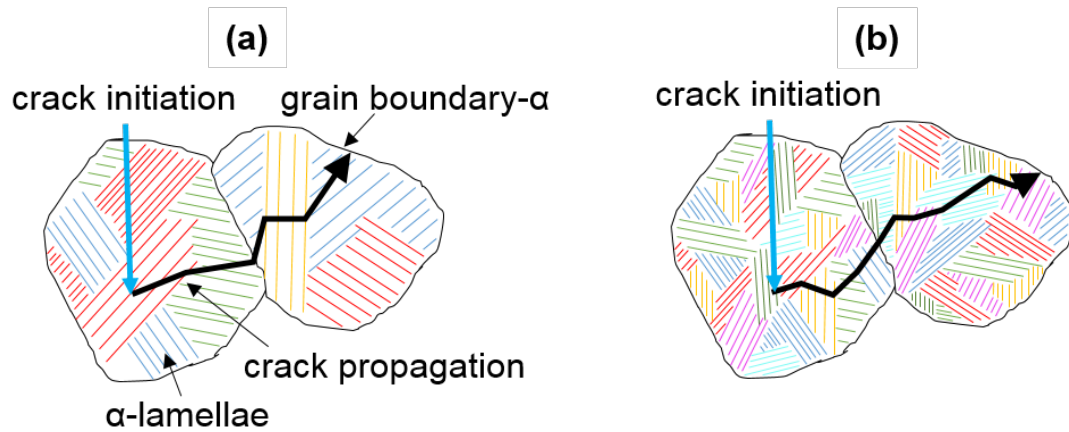


Figure 4.20 Schematic illustration of crack initiation and propagation in lamellar microstructures with (a) larger colony size and (b) smaller colony size.

On the other hand, the crack in the bi-lamellar microstructures propagates along the primary α -lamellae interfaces and/or the boundary components, as described in **Figure 4.21(a)**

and **(b)**, which are the schematic illustrations of the crack initiation and propagation in the bi-lamellar microstructures with larger and smaller colony size, respectively. Since the primary α -lamellae interface and the boundary components are comprised of HAGB, the crack propagation length for the failure in the bi-lamellar microstructures increase with decreasing colony size. It was confirmed that the length density of HAGB increased with decreasing the colony size in **Figure 2.8**. As a result, the colony size refinement in bi-lamellar microstructure leads an increase of the crack propagation length, resulting in enhanced post-uniform elongation.

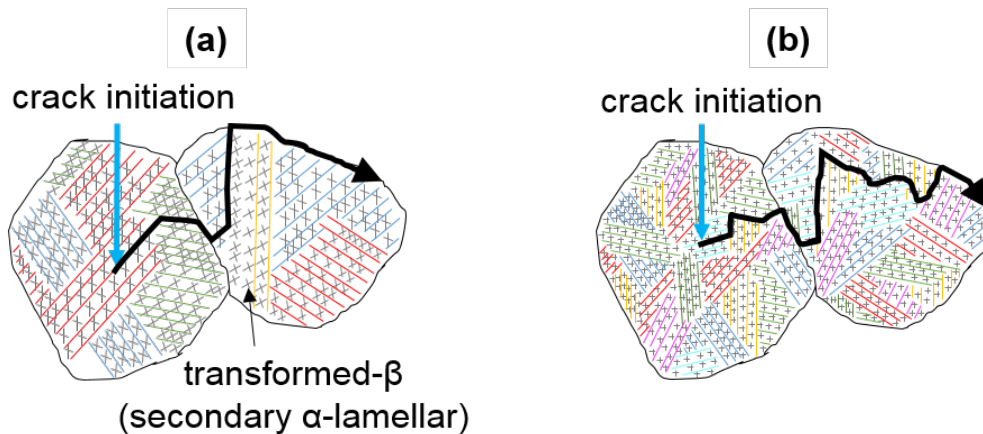


Figure 4.21 Schematic illustration of crack initiation and propagation in bi-lamellar microstructures with (a) larger colony size and (b) smaller colony size.

4.4. Conclusion

In this Chapter, fracture behavior in lamellar and bi-lamellar microstructures having different colony sizes was characterized by micro-crack observation and main-crack observation. In order to understand the crack formation mechanism, deformation behavior in lamellar and bi-lamellar microstructures was investigated using the micro-DIC technique. The

major results of this chapter are summarized as follow:

1. In order to clarify micro-crack formation behavior, micro-crack formation was classified into four types based on the possible micro-crack formation site: Type I; crack elongated across α -lamellae, Type II; crack elongated along α -lamellae interface, Type III; crack elongated along colony boundary and Type IV; crack elongated along grain boundary- α .

2. In lamellar microstructure, three types of micro-crack formation were observed (Type I, II and III). It was found that micro-crack preferentially initiates at α -lamellae interface regardless of the colony size. On the other hand, longer micro-crack mostly elongated across α -lamellae. Main-crack in lamellar microstructure preferentially propagated across α -lamellae. In the smaller colony size in lamellar microstructure, main-crack propagated along colony boundary was also observed. When the main-crack reached colony boundary, the main-crack propagation path was diverted. Fractograph of the lamellar microstructure was mostly flat and was covered by dimple patterns. The main-crack propagating across α -lamellae followed a crystallographic orientation relationship to adjacent colonies in parallel with $\{10\bar{1}0\}$ prismatic plane trace. Based on the result of the fractograph observation and crystallographic orientation dependence of the main-crack, the fracture mechanism in the lamellar microstructure is considered as the ductile fracture accompanied by transgranular-like fracture.

3. Microscopic plastic strain in lamellar microstructure propagates across α -lamellae and colony boundaries. The microscopic strain propagation showed a good agreement with the

tendency of the main-crack propagation behavior in the lamellar microstructure. Slip line during tensile test appeared across α -lamellae and colony boundary. From these result, it was concluded that the main-crack in lamellar microstructure which predominantly propagates across α -lamellae is accompanied by local plastic deformation.

4. In bi-lamellar microstructure, three types of micro-crack formation were observed (Type II, III and IV). It was found that micro-crack preferentially initiates at primary α -lamellae interface in the colony size of 234.4 μm . With decreasing the colony size, on the other hand, the number fraction of micro-crack along colony boundary increased. Main-crack in bi-lamellar microstructure preferentially was found to propagate along boundary components (i.e., colony boundary and grain boundary- α). When the main-crack reached colony boundary, the main-crack propagation path was diverted. Fractograph of bi-lamellar microstructure was more rugged than that in the lamellar microstructure, because the fractograph was composed of intergranular-like fracture surface covered by dimple patterns. It was revealed that main-crack propagation was independent of crystallographic orientation in adjacent colonies from the main-crack. Fracture mechanism of the bi-lamellar microstructure is considered as the ductile fracture accompanied by intergranular-like fracture.

5. Microscopic plastic strain in bi-lamellar microstructure was mainly distributed along colony boundaries. In the higher magnification of SEM image collaborated micro-DIC, it was confirmed that the plastic strain concentrated along primary α -lamellae interface and/or boundary components. It was clearly confirmed that slip transfer was prohibited at the primary

α -lamellae interface. From these result, it was concluded that the main-crack in bi-lamellar microstructure which predominantly propagates along boundary components is accompanied by strain concentration at primary α -lamellae interface and/or boundary components.

6. Combining the result of main-crack propagation behavior and the response of post-uniform elongation (obtained in **Chapter 2**) with respect to the colony size in lamellar and bi-lamellar microstructures, the reason for the different tendency of the post-uniform elongation can be concluded. Since main-crack in lamellar microstructure propagates across not only α -lamellae but also colony boundaries, crack propagation lengths for the failure became almost similar even though colony size decreased. Hence, post-uniform elongation in lamellar microstructure independent of colony size. On the other hand, since main-crack in bi-lamellar microstructures propagates along primary α -lamellae interfaces and/or boundary components (i.e., HAGB), crack propagation length for the failure in bi-lamellar microstructures increase with decreasing colony size. As a result, post-uniform elongation in bi-lamellar microstructure gradually increased with decreasing colony size.

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CHAPTER 5

FRACTURE PROPERTIES IN VARIOUS MICROSTRUCTURES WITH DIFFERENT GRAIN SIZES OR COLONY SIZES

5.1. Introduction

In the previous chapter, crack formation in the lamellar and bi-lamellar microstructures of Ti-6Al-4V alloy was clarified under the micro-crack and the main-crack observation. It was revealed that the fracture properties of titanium alloys are sensitive to the microstructural features. The main-crack in the lamellar microstructure propagates across α -lamellae and colony boundary, but the main-crack in the bi-lamellar microstructure propagates along boundary components (i.e., colony boundary and grain boundary- α). It has been believed that the titanium alloys with lamellar microstructure have superior fracture toughness compared to ones with an equiaxed microstructure. The fracture toughness in the lamellar microstructure was reported to be enhanced by the crack propagation in a zig-zag way for fracture [1-3]. A number of studies suggested that the thickened α -lamellae leads to increase of crack deflection and its fracture toughness [4, 5]. These studies indicate that the thickness of α -lamellae is one of the important parameters to control the fracture toughness of titanium alloys with lamellar microstructure. Several studies have been reported with respect to the fracture toughness in the lamellar microstructure of $\alpha + \beta$ titanium alloys and their microstructural influence, but the influence of microstructural parameters on the fracture toughness is still not sufficiently revealed. Hence, the present study aims to compare the fracture toughness and crack propagation resistance of lamellar and bi-lamellar microstructures with different colony sizes which obtained in the previous chapter. In order to clarify the advantage of lamellar and bi-

lamellar microstructures in fracture properties, fracture toughness and crack propagation resistance in equiaxed microstructure with different grain sizes were investigated, as well.

5.2. Experiment procedure

5.2.1. Fabrication of equiaxed microstructures with various grain sizes

In the same manner with the TMP to obtain the lamellar and bi-lamellar microstructures, TMP for various grain sizes of equiaxed microstructures was carried out through hot compression test using a thermomechanical simulator (Thermecmastor-Z). Cylindrical specimens with 8 mm in diameter and 12 mm in height were prepared from the as-cast billet, as described in **Figure 2.1(c)**. The initial microstructure was used by the specimen having martensite microstructure (**Figure 2.4**) in the outer part (i.e., rapidly cooled zone) of the billet. The TMP for the equiaxed microstructure is schematically shown in **Figure 5.1**. The specimens were heated to 700 °C or 900 °C ($\alpha + \beta$ phase region) and held for 0.3 ks. Subsequently, the specimens were uniaxially compressed to 1.2 of compressive strain at the different strain rates, 10^{-2} s^{-1} or 10^{-4} s^{-1} to control the grain size. And then, the specimens were slowly cooled to 400 °C with a cooling rate of $10 \text{ }^{\circ}\text{C min}^{-1}$. **Figure 5.2** shows the obtained equiaxed microstructures with different grain sizes. The grain sizes of the specimens obtained by the deformations at 900 °C with a strain rate 10^{-4} s^{-1} , at 900 °C with a strain rate 10^{-2} s^{-1} , and at 700 °C with a strain rate 10^{-4} s^{-1} were 7.53 μm , 2.69 μm and 0.63 μm (ultra-fine grain, UFG), respectively.

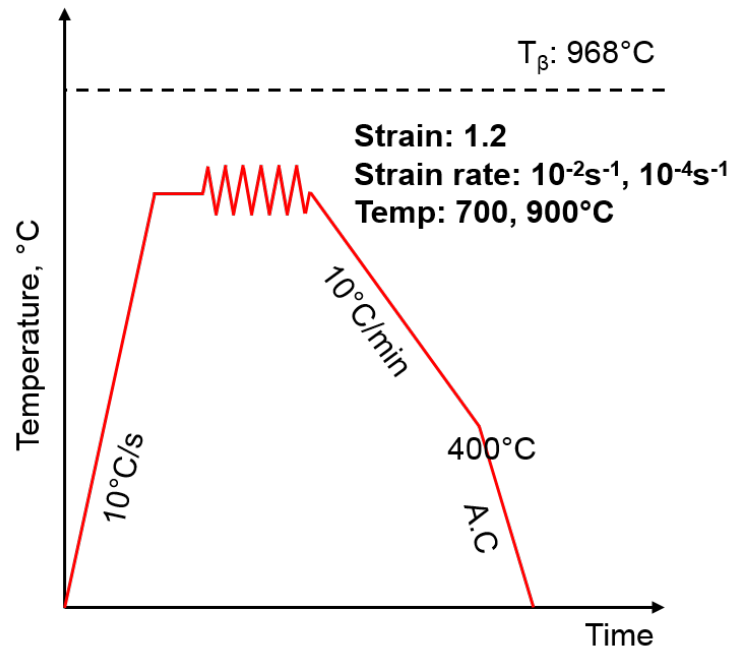


Figure 5.1 Schematic illustrations of thermomechanical processing route to obtain various grain sizes in equiaxed microstructures of Ti-6Al-4V alloy.

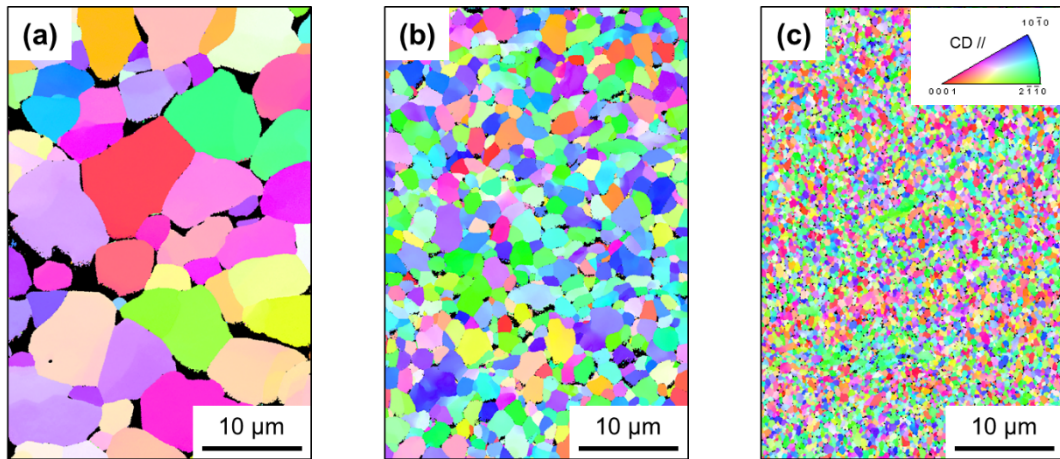


Figure 5.2 EBSD IPF maps of equiaxed microstructures obtained through the TMP at (a) 900 °C with a strain rate 10^{-4} s^{-1} , (b) 900 °C with a strain rate 10^{-2} s^{-1} , and (c) 700 °C with a strain rate 10^{-4} s^{-1} , respectively.

5.2.2. Measurement of fracture properties in various microstructures

5.2.2.1 Three-point bending test

In order to estimate the fracture toughness in equiaxed microstructure with different grain sizes, and in lamellar and bi-lamellar microstructures with different colony sizes, three-point bending tests (opening mode of loading; Mode I) were carried out at room temperature at a cross-head speed of 0.5 mm min^{-1} using a uniaxial tensile machine (SHIMAZU AG-X plus). Miniaturized single edge notched bend (SENB) specimens with 10 mm in support span length (S), 3 mm in width (W), and 2 mm in thickness (B), schematically shown in **Figure 5.3**, were prepared. The single notch in the specimens was fabricated with 0.02 mm in width, 1.3 mm in length, and 0.15 mm in radius. The specimens were designed according to the standard demonstrated in the ASTM E1820 [6].

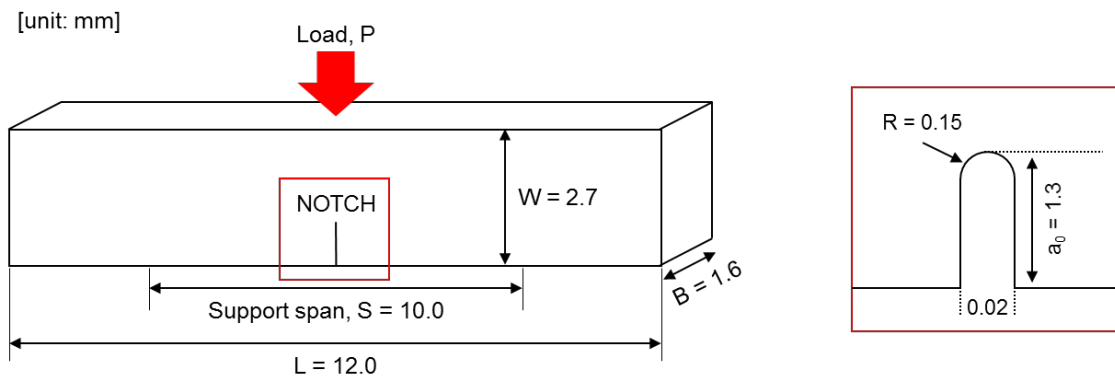


Figure 5.3 Schematic illustrations of the single edge notched three-point bending test specimen.

5.2.2.2 Calculation of J-integral

The fracture property in materials including pre-crack or notch is generally evaluated with

stress intensity factor (K) indicating stress condition at the crack tip or at the notch in linear elastic materials. However, since most metallic materials have large plastic deformation before the fracture, it is inappropriate to estimate the fracture property using the K based on the linear elastic fracture mechanics (LEFM). Hence, in this chapter, J -integral which can be applicable to elasto-plastic fracture mechanics (EPFM) was evaluated to compare the fracture properties in the Ti-6Al-4V alloy with various microstructures. The J -integral means the energy needed for crack propagation to the unit area in elasto-plastic materials. Elasto-plastic fracture toughness parameter, J -integral, can be divided into an elastic component of J -integral, J_{el} , and a plastic component of J -integral, J_{pl} , as following equation (5-1):

$$J = J_{el} + J_{pl} \dots\dots\dots (5-1)$$

The J_{el} is calculated from the stress intensity factor K , elastic modulus E , and Poisson's ratio ν , as follows:

$$J_{el} = \frac{K^2(1-\nu^2)}{E} \dots\dots\dots (5-2)$$

For the SENB specimen at a load, P_i , calculate K , as follows [6]:

$$K_i = \left[\frac{P_{el}S}{BW^{3/2}} \right] f(a/W) \dots\dots\dots (5-3)$$

$$f(a/W) = \frac{3(a/W)^{1/2}[1.99-(a/W)(1-a/W)(2.15-3.93(a/W)+2.7(a/W)^2)]}{2(1+2a/W)(1-a/W)^{3/2}} \dots\dots\dots (5-4)$$

where S , B , W and a denote a length of support span, a thickness of the specimen, a width of the specimen and a crack length, respectively. P_{el} is a load of the intersection of the experimentally obtained load-deflection curve and the 5% secant line (i.e., a straight line from the origin with a slope equal to 95% of initial elastic loading slope) and means the limit load

in the linear elastic theory. The plastic component of *J-integral*, J_{pl} , can be defined as follow:

$$J_{pl} = \frac{\eta A_{pl}}{B(W-a)} \dots \dots \dots (5-5)$$

$$\eta = \sqrt{1 + \left(\frac{a}{W-a}\right)^2} - \frac{a}{W-a} \approx 0.43 \dots \dots \dots (5-6)$$

where A_{pl} and η denote the plastic area under the load-displacement curve and dimensionless constant dependent on the shape of the specimen, respectively. The A_{pl} was defined by means of **Figure 5.4**.

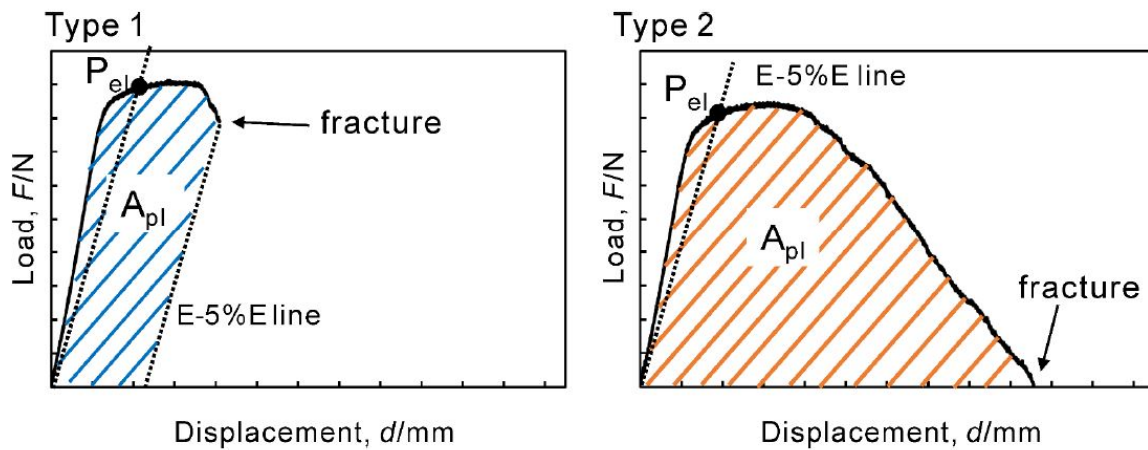


Figure 5.4 Definition of A_{pl} for *J-integral* calculation [7]

5.2.2.3 Crack length measurement for *R-curve*

In order to estimate the crack propagation resistance, *R-curves* should be plotted using calculated *J-integral* as a function of crack extension which means the increment of crack length from the notch tip during the test. Since the plastic zone shape and size on the surface and the internal of the specimen are varied depending on the stress state (i.e., plane strain or

plane stress conditions), the crack length should be measured using an appropriate method [8]. Hence, prior to measure the crack length, the stress state was verified by observing the plastic zone shape at the notch tip using the DIC analysis software. **Figure 5.5** represents the plastic strain map at the notch tip of the specimen with lamellar microstructure. Red and purple colored region indicate strongly deformed region and less deformed region, respectively. Since the stress state of the used specimens was verified as the plane strain condition (butterfly shape of plastic zone) [8], the crack length on the surface and the internal of the specimens were considered as the same. The crack length on the fracture surface was physically measured using an ImageJ software which is a public domain Java image processing program. The images captured by the CCD camera during the bending test was imported to the ImageJ software.

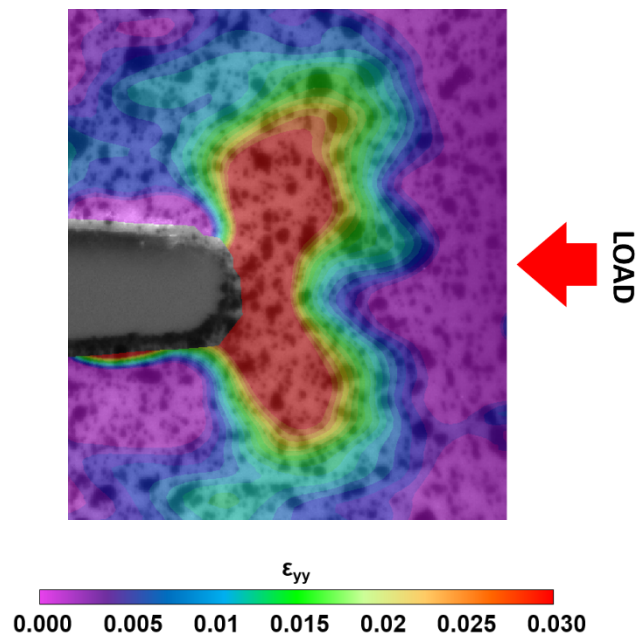
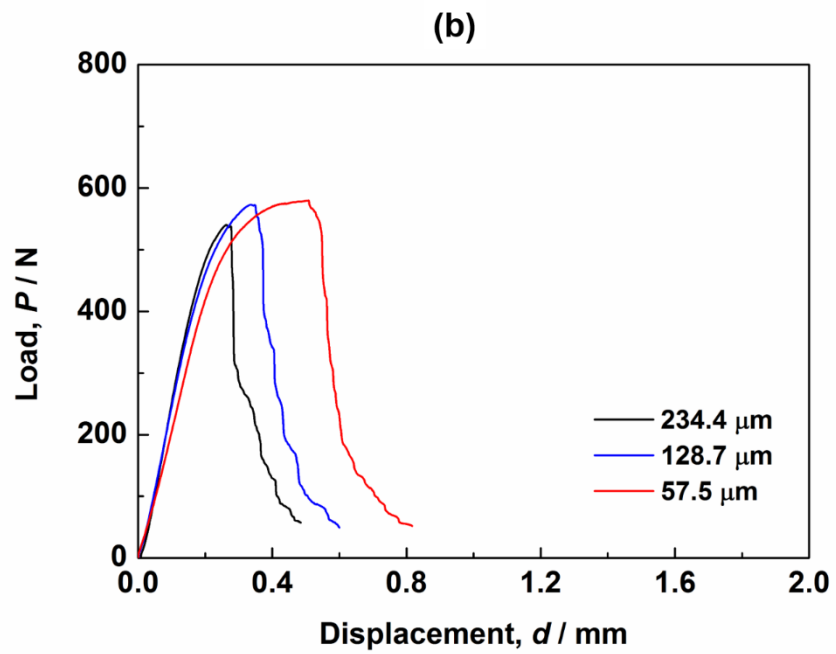
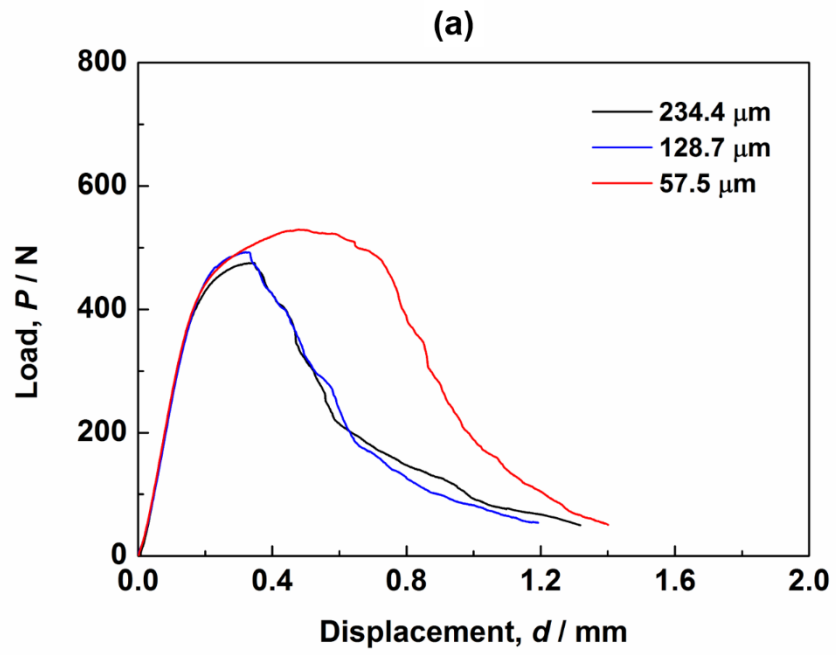


Figure 5.5 Plastic zone shape at the notch tip of the single edge notched bending test specimen obtained using DIC technique. The plastic zone shape was identified to “butterfly shape” which corresponding to the plastic zone shape of plane strain condition.

5.3. Results and discussion

5.3.1. Load-displacement curve obtained by three-point bending tests in various microstructures

Figure 5.6 presents the load-displacement curves obtained by three-point bending tests using the specimens with lamellar and bi-lamellar microstructures with different colony sizes and equiaxed microstructures with different grain sizes. In the lamellar microstructure (**Figure 5.6(a)**), it was found that the maximum load increased with decreasing the colony size. After the maximum load, the applied load in every colony size was gradually decreased. On the other hand, the load decline after maximum load in the bi-lamellar microstructures (**Figure 5.6(b)**) with different colony size was relatively faster than that in the lamellar microstructures. In the equiaxed microstructure (**Figure 5.6(c)**), different load decline behavior was observed with respect to the grain sizes. It was found that the load decline in the specimen with the grain size of 7.53 μm was relatively slower. However, the drastic load decline was observed in the specimens with the grain sizes of 2.69 μm and 0.63 μm .



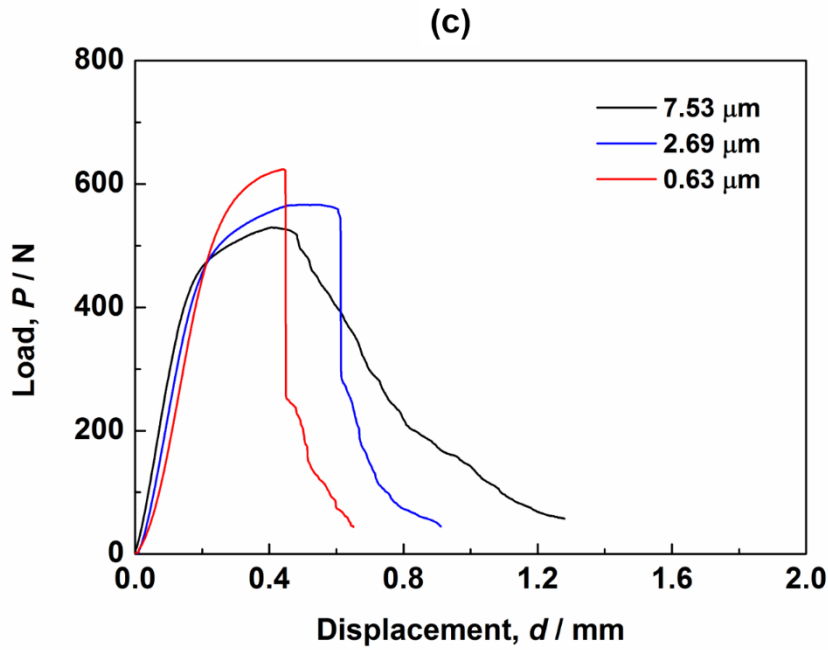


Figure 5.6 Load-displacement curves of (a) lamellar and (b) bi-lamellar microstructures with different colony size ranging from 234.4 μm to 57.5 μm , and (c) equiaxed microstructures with different grain sizes ranging from 7.53 μm to 0.63 μm obtained using three-point bending test at room temperature.

5.3.2. Effect of microstructural parameters on fracture toughness, *J*-integral, in various microstructures

The load decline after maximum load in the load-displacement curve is generally considered to be due to the fracture mechanism, fracture resistance, and fracture toughness. Hence, the fracture toughness, *J*-integral, in the lamellar, the bi-lamellar and the equiaxed microstructures was computed based on the obtained load-displacement curves in each microstructure. **Figure 5.7** exhibits the *J*-integral in the lamellar and the bi-lamellar microstructures with different colony sizes, which corresponds to the red colored x-axis, and the equiaxed microstructure with different grain sizes, which corresponds to the blue colored

x-axis. It was found that the *J-integral* in the lamellar microstructures ($58.7 \sim 90.7 \text{ kJm}^{-2}$) was obviously higher than that in the bi-lamellar microstructures ($42.9 \sim 74.2 \text{ kJm}^{-2}$) of the same level of the colony size. This result might be related to the observed crack propagation behavior in the lamellar and the bi-lamellar microstructures revealed in **Chapter 3**. It is believed that the crack deflection (i.e., increase of crack propagation length) and the termination of crack growth are one of the reasons for improving the fracture toughness [8-11]. The crack propagation in the lamellar microstructure was revealed to propagate across α -lamellae, but to deflect at colony boundaries. On the other hand, the crack propagation in the bi-lamellar microstructure was found to propagate along the boundary components (i.e., colony boundaries and grain boundary- α). It has been reported that the fracture along the boundary components (i.e., intergranular fracture) is harmful to resist the crack propagation due to the concentration of the segregated impurities which leads to the stress concentration [8, 12, 13]. Hence, the different crack propagation mechanism in the lamellar and bi-lamellar microstructures is considered to be the reason for the higher *J-integral* in the lamellar microstructure. Moreover, it was found that the *J-integral* in the lamellar and the bi-lamellar microstructures gradually increased with decreasing the colony size. The reason for this result is considered to be due to the increased number of crack deflection in the smaller colony size because the density of the colony boundaries increases in the smaller colony size in the lamellar and bi-lamellar microstructures. On the other hand, the *J-integral* of the equiaxed microstructure gradually decreased ranging from 76.6 kJm^{-2} to 56.4 kJm^{-2} with decreasing the grain size, resulting in

lower *J-integral* in comparison with that in the smaller colony size of the lamellar and the bi-lamellar microstructures

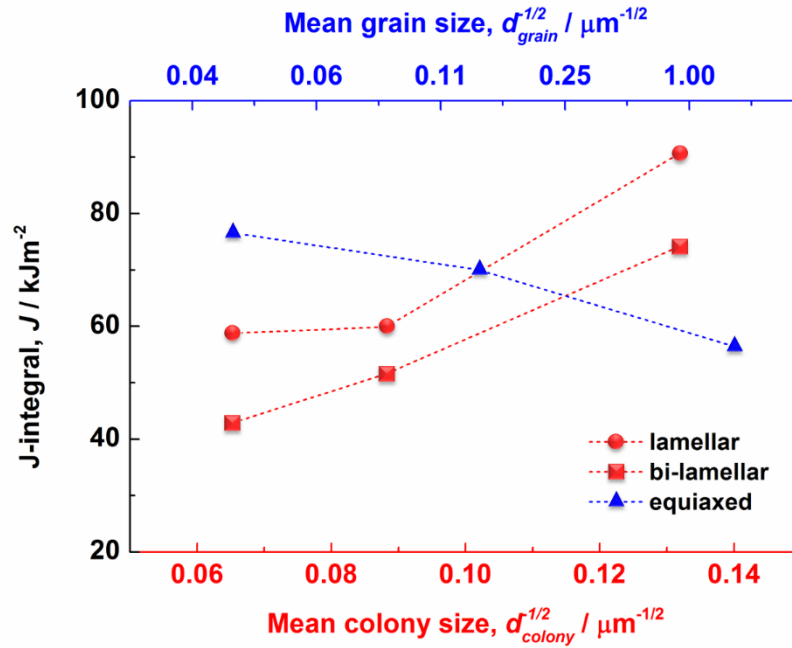


Figure 5.7 Calculated J-integral of the lamellar and bi-lamellar microstructures with respect to the colony size corresponding to the red colored x-axis, and in the equiaxed microstructure with respect to the grain size corresponding to the blue colored x-axis.

Figure 5.8 shows the balance between ultimate tensile strength (UTS) and *J-integral* in the lamellar and bi-lamellar microstructures with different colony sizes and the equiaxed microstructure with different grain sizes. It was clearly found that the smallest colony size (57.5 μm) in the lamellar and the bi-lamellar microstructures showed a good balance between UTS and fracture toughness. It is noteworthy that the colony size refinement in lamellar and bi-

lamellar microstructures has the great potential to possess much higher strength and much higher fracture toughness rather than those of equiaxed microstructure in Ti-6Al-4V alloy.

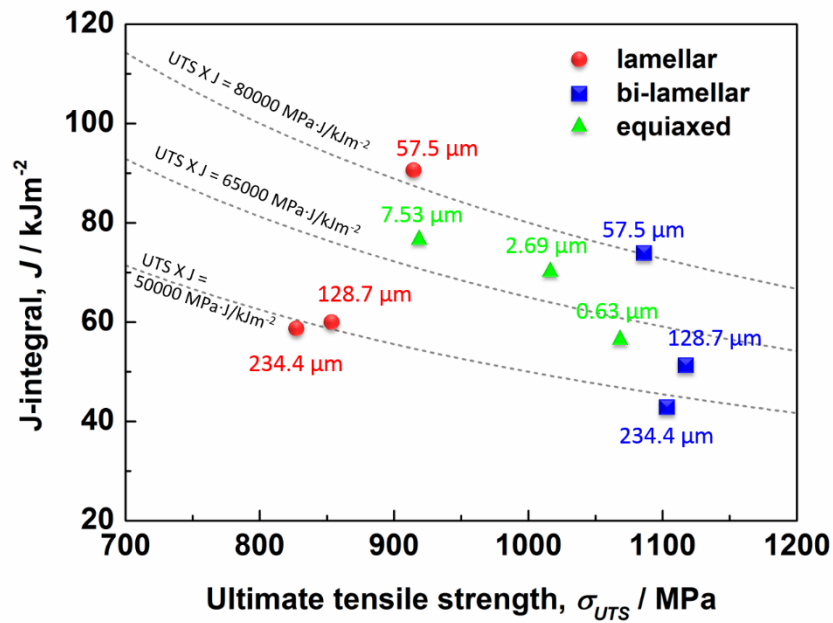


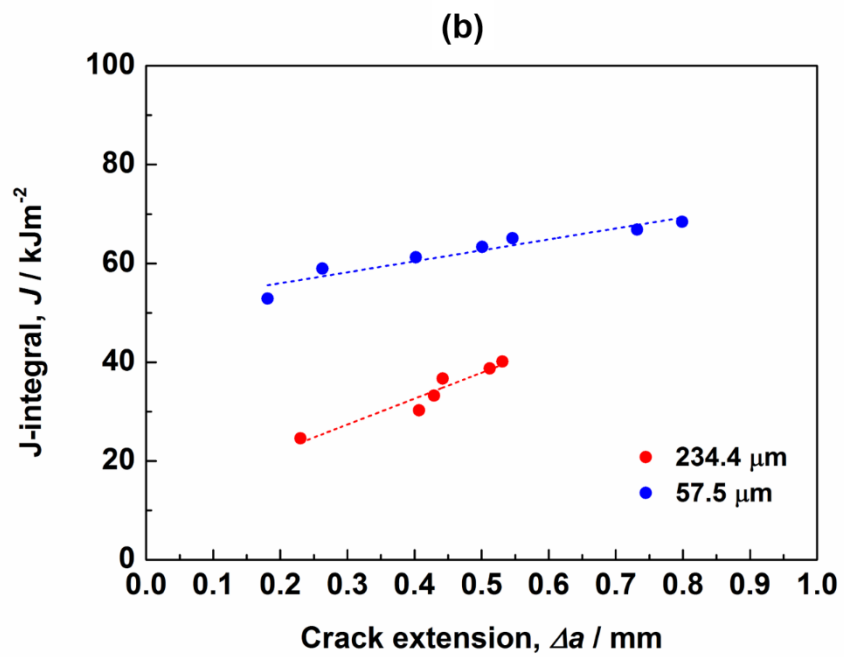
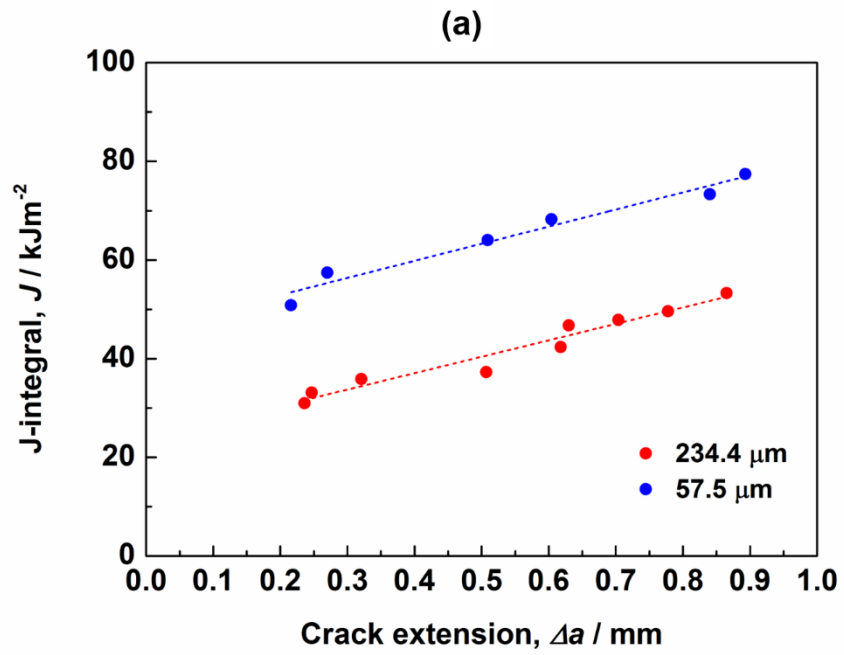
Figure 5.8 Balance between ultimate tensile strength and *J-integral* in the lamellar and bi-lamellar microstructures with different colony sizes, and equiaxed microstructures with different grain sizes.

5.3.3. Effect of microstructural parameters on crack growth resistance in various microstructures (*R-curve*)

Not only the crack propagation mechanism but also the crack propagation resistance is the critical parameter to determine the fracture toughness in materials. Hence, the crack propagation resistance in the lamellar and the bi-lamellar microstructures with different colony sizes, and the equiaxed microstructures with different grain sizes were investigated by the plot

of *J-integral* as a function of the crack extension (Δa). The amount of the *J-integral* increment indicates the crack propagation resistance.

Figure 5.9 exhibits the crack propagation resistance curve (so-called R-curve) of (a) lamellar microstructures, (b) bi-lamellar microstructures, and (c) equiaxed microstructures. Most of the *J-integral* in the R-curves of the lamellar, bi-lamellar and equiaxed microstructures were observed to gradually increase with increasing the crack extension. This shape of R-curves can be represented as a typical R-curve for ductile materials accompanied by stable crack propagation [8]. The R-curves in the lamellar microstructures with respect to the colony size (**Figure 5.9(a)**), the crack propagation resistance (i.e., slope of the fitted line) was nearly the same even though the colony size decreased. This result might be related to the result in **Chapter 4**, the identical crack propagation length for the failure irrespective of the colony size. On the other hand, the crack propagation resistance in the bi-lamellar microstructure (**Figure 5.9(b)**) decreased with decreasing the colony size. This result can be considered to be due to the increase of the crack propagation length for failure with decreasing the colony size in the bi-lamellar microstructures. As concluded in **Chapter 4**, the crack propagation in the bi-lamellar microstructure is predominantly advanced along the boundary components. It was found that the length density of the boundary components (i.e., high angle grain boundaries) increased with decreasing the colony size, and then the crack propagation length also increased. As a result, the fracture toughness in the bi-lamellar microstructure increased with decreasing the colony size. The R-curve of the equiaxed microstructure (**Figure 5.9(c)**) was observed to have less increment of *J-integral* (so-called flat R-curve) with decreasing the grain size, resultant in lower fracture toughness.



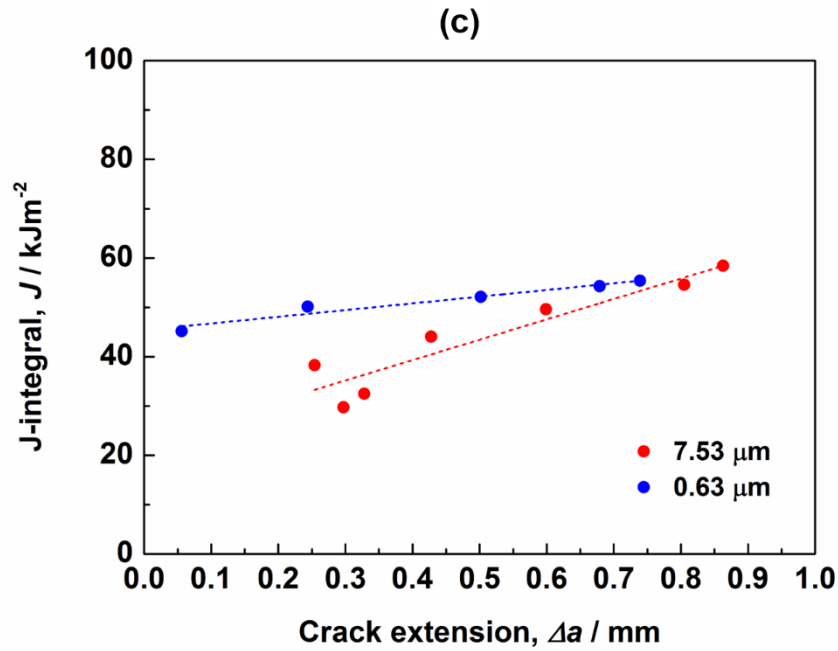


Figure 5.9 Crack propagation resistance curve (R-curve) of (a) lamellar, (b) bi-lamellar microstructures with different colony sizes (234.4 μ m and 57.5 μ m), and (c) equiaxed microstructures with different grain sizes (7.53 μ m and 0.63 μ m) after the single edge notched three-point bending test at room temperature.

5.4. Conclusion

In this Chapter, fracture toughness in lamellar and bi-lamellar microstructures having different colony sizes and equiaxed microstructure having different grain sizes was investigated using single edge notched three-point bending test. The major results of this chapter are summarized as follow:

1. In lamellar microstructure, the applied load after the maximum load gradually increased with decreasing the colony size. On the other hand, the relatively rapid load decline after the maximum load was observed in bi-lamellar microstructures. In the equiaxed microstructure,

load decline behavior was clearly changed in the smaller grain size. It was found that the load decline in the specimen with the grain size of 7.53 μm decreased slowly but that in the grain size of 2.69 μm and 0.63 μm decreased drastically.

2. *J-integral* in lamellar microstructure was obviously higher than that in bi-lamellar microstructures in the same level of the colony size. Crack deflection at colony boundaries in the lamellar microstructure, which concluded in Chapter 4 is considered as the reason for the higher *J-integral* than that of bi-lamellar microstructure. On the other hand, the crack propagation along boundary components (i.e., colony boundaries and grain boundary- α) in the bi-lamellar microstructure is considered as the reason for the lower *J-integral* than that in the lamellar microstructure. In both microstructures, the *J-integral* gradually increased with decreasing the colony size.

3. R-curves of lamellar, bi-lamellar and equiaxed microstructures gradually increased with increasing the crack extension, which represents the fracture in lamellar, bi-lamellar and equiaxed microstructures accompanied by a ductile fracture with stable crack propagation. On the other hand, the equiaxed microstructure with the grain size of 0.63 μm was attributed nearly flat R-curve, which means the fracture accompanied by the unstable crack propagation. In the lamellar microstructures, crack propagation resistance was nearly the same despite the colony size decreased. On the other hand, crack propagation resistance in the bi-lamellar microstructure decreased with decreasing the colony size.

4. Lamellar and bi-lamellar microstructures with the colony size of 57.5 μm presented a good balance between UTS and fracture toughness. This result can suggest that the lamellar and bi-lamellar microstructures have the great potential to possess much higher strength and fracture toughness through the colony size refinement.

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CHAPTER 6

SUMMARY AND CONCLUSION

In this thesis, the effect of colony size of Ti-6Al-4V alloy having lamellar and bi-lamellar microstructures on mechanical properties were systemically investigated. Tensile tests were carried out to evaluate the tensile strength, tensile elongation and fracture behavior after tensile failure, and three-point bending tests were carried out to estimate fracture toughness and crack propagation resistance. The results obtained in the present thesis are summarized as follows:

Chapter 1 introduced the backgrounds of the present study. General use of titanium alloys in industrial fields, the nature of titanium and titanium alloys, representative microstructures and its mechanical properties were described. The objective of the present study was mentioned.

Chapter 2 covered thermomechanical processing to obtain the lamellar and bi-lamellar microstructure with various colony sizes. Various colony sizes of lamellar microstructures were obtained through simple deformation process with various compressive strain at 1050 °C followed by slow cooling with 10 °C min⁻¹ of cooling rate. The colony sizes were achieved ranging from 234.4 µm to 128.7 µm with increasing the compressive strain ranging from 0 to 1.2, but the colony size was saturated to approximately 130 µm when the compressive strain was over 0.3. The finer colony size than the colony size of lamellar microstructures manufactured by simple deformation process was obtained through multiple deformation process. The compression test was performed to 0.6 compressive strain at 700 °C and 1050 °C,

respectively. It was found that β grain size of the multiple deformed specimen was much smaller than that in the simple deformed specimen. As a result, the colony size in the lamellar microstructure refined down to 57.5 μm . Different colony sizes of bi-lamellar microstructures were obtained through inter-critical annealing at 920 $^{\circ}\text{C}$ followed by water quenching. Fine secondary α -lamellae was precipitated inside transformed- β region in-between primary α -lamellae. The different colony sizes of lamellar microstructures obtained through two different types of TMPs were used as initial microstructures, resultant in the various colony sizes in the bi-lamellar microstructures. The colony sizes in the bi-lamellar microstructures were considered to be equal to those in the initial lamellar microstructures, i.e., 234.4 μm , 128.7 μm and 57.5 μm . The crystallographic orientation relationship between the retained- β and the α -lamellae in the lamellar microstructure was revealed to maintain the BOR having $\{0002\}_{\alpha} \parallel \{110\}_{\beta}$ and $\langle 11\bar{2}0 \rangle_{\alpha} \parallel \langle 111 \rangle_{\beta}$. The colony composed of the equivalent crystallographic orientation of α -lamellae was identified to have a total of 12 crystallographic orientations from one β grain. On the other hand, one colony in the bi-lamellar microstructure was composed of the additional 12 variants of secondary α -lamellae maintaining the BOR against identical retained- β in-between the primary α -lamellae.

Chapter 3, Chapter 4 and Chapter 5 are containing the mechanical response of obtained lamellar and bi-lamellar microstructures having different colony sizes.

In **Chapter 3**, nano indentation test and uniaxial tensile test were performed to compare nano-hardness of microstructural components in lamellar and bi-lamellar microstructures and to clarify the effect of colony size on tensile properties in lamellar and bi-lamellar microstructures.

Nano-hardness of microstructural components in the lamellar and bi-lamellar microstructures was measured using the nano indentation test. The hardness of α -lamellae was higher than that of thin retained- β in the lamellar microstructure. In the bi-lamellar microstructure, the primary α -lamellae was harder than the transformed- β regions. Comparing the lamellar and the bi-lamellar microstructures, the hardness of primary α -lamellae was nearly identical in both microstructures, while the hardness of transformed- β regions composed of secondary α -lamellae in the bi-lamellar microstructure was higher than that of retained- β in the lamellar microstructure. Mechanical properties of the lamellar microstructures with the average colony size of 234.4 μm , 128.7 μm and 57.5 μm were examined by room temperature uniaxial tensile test at a strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$. The strengths (yield strength, ultimate tensile strength) were enhanced with decreasing the colony size. The uniform elongation of the lamellar microstructure increased slightly with reducing the colony size, but the post-uniform elongation was nearly constant regardless of the colony size. Mechanical properties of the bi-lamellar microstructures with the average colony size of 234.4 μm , 128.7 μm and 57.5 μm were examined by uniaxial tensile test at room temperature. All kinds of strength of the bi-lamellar microstructures was obviously higher than that in the lamellar microstructures. The uniform elongation and the post-uniform elongation of the bi-lamellar microstructure were greatly improved with decreasing the colony size. In order to understand the reason for the improved post-uniform elongation with decreasing the colony size in the bi-lamellar microstructure, the strain rate sensitivity of the bi-lamellar microstructures was measured using tensile tests at various strain rates ranging from $1.6 \times 10^{-2} \text{ s}^{-1}$ to $1.6 \times 10^0 \text{ s}^{-1}$. The strain rate sensitivity of the bi-lamellar microstructure was measured to be approximately 0.04, which was nearly identical

regardless of the colony size. Hence, it was concluded that the strain rate sensitivity was not the reason for the increased post-uniform elongation of the bi-lamellar microstructures with decreasing the colony size in the Ti-6Al-4V alloy.

In **Chapter 4**, micro-crack formation was classified into four types based on the possible micro-crack formation site: Type I; crack elongated across α -lamellae, Type II; crack elongated along α -lamellae interface, Type III; crack elongated along colony boundary and Type IV; crack elongated along grain boundary- α . In lamellar microstructure, three types of micro-crack formation were observed (Type I, II and III). It was found that micro-crack preferentially initiates at α -lamellae interface regardless of the colony size. On the other hand, longer micro-crack mostly elongated across α -lamellae. Main-crack in lamellar microstructure preferentially propagated across α -lamellae. In the smaller colony size in lamellar microstructure, main-crack propagated along colony boundary was also observed. When the main-crack reached colony boundary, the main-crack propagation path was diverted. Fractograph of the lamellar microstructure was mostly flat and was covered by dimple patterns. The main-crack propagating across α -lamellae followed a crystallographic orientation relationship to adjacent colonies in parallel with $\{10\bar{1}0\}$ prismatic plane trace. Based on the result of the fractograph observation and crystallographic orientation dependence of the main-crack, the fracture mechanism in the lamellar microstructure is considered as the ductile fracture accompanied by transgranular-like fracture. Microscopic plastic strain in lamellar microstructure propagates across α -lamellae and colony boundaries. The microscopic strain propagation showed a good agreement with the tendency of the main-crack propagation behavior in the lamellar

microstructure. Slip line during tensile test appeared across α -lamellae and colony boundary. From these result, it was concluded that the main-crack in lamellar microstructure which predominantly propagates across α -lamellae is accompanied by local plastic deformation. In bi-lamellar microstructure, three types of micro-crack formation were observed (Type II, III and IV). It was found that micro-crack preferentially initiates at primary α -lamellae interface in the colony size of 234.4 μm . With decreasing the colony size, on the other hand, the number fraction of micro-crack along colony boundary increased. Main-crack in bi-lamellar microstructure preferentially was found to propagate along boundary components (i.e., colony boundary and grain boundary- α). When the main-crack reached colony boundary, the main-crack propagation path was diverted. Fractograph of bi-lamellar microstructure was more rugged than that in the lamellar microstructure, because the fractograph was composed of intergranular-like fracture surface covered by dimple patterns. It was revealed that main-crack propagation was independent of crystallographic orientation in adjacent colonies from the main-crack. Fracture mechanism of the bi-lamellar microstructure is considered as the ductile fracture accompanied by intergranular-like fracture. Microscopic plastic strain in bi-lamellar microstructure was mainly distributed along colony boundaries. In the higher magnification of SEM image collaborated micro-DIC, it was confirmed that the plastic strain concentrated along primary α -lamellae interface and/or boundary components. It was clearly confirmed that slip transfer was prohibited at the primary α -lamellae interface. From these result, it was concluded that the main-crack in bi-lamellar microstructure which predominantly propagates along boundary components is accompanied by strain concentration at primary α -lamellae interface and/or boundary components. Combining the result of main-crack propagation behavior and

the response of post-uniform elongation (obtained in **Chapter 2**) with respect to the colony size in lamellar and bi-lamellar microstructures, the reason for the different tendency of the post-uniform elongation can be concluded. Since main-crack in lamellar microstructure propagates across not only α -lamellae but also colony boundaries, crack propagation lengths for the failure became almost similar even though colony size decreased. Hence, post-uniform elongation in lamellar microstructure independent of colony size. On the other hand, since main-crack in bi-lamellar microstructures propagates along primary α -lamellae interfaces and/or boundary components (i.e., HAGB), crack propagation length for the failure in bi-lamellar microstructures increase with decreasing colony size. As a result, post-uniform elongation in bi-lamellar microstructure gradually increased with decreasing colony size.

In **Chapter 5**, fracture toughness in lamellar and bi-lamellar microstructures having different colony sizes and equiaxed microstructure having different grain sizes was investigated using single edge notched three-point bending test. In lamellar microstructure, the applied load after the maximum load gradually increased with decreasing the colony size. On the other hand, the relatively rapid load decline after the maximum load was observed in bi-lamellar microstructures. In the equiaxed microstructure, load decline behavior was clearly changed in the smaller grain size. It was found that the load decline in the specimen with the grain size of 7.53 μm decreased slowly but that in the grain size of 2.69 μm and 0.63 μm decreased drastically. *J-integral* in lamellar microstructure was obviously higher than that in bi-lamellar microstructures in the same level of the colony size. Crack deflection at colony boundaries in the lamellar microstructure, which concluded in Chapter 4 is considered as the reason for the

higher *J-integral* than that of bi-lamellar microstructure. On the other hand, the crack propagation along boundary components (i.e., colony boundaries and grain boundary- α) in the bi-lamellar microstructure is considered as the reason for the lower J-integral than that in the lamellar microstructure. In both microstructures, the *J-integral* gradually increased with decreasing the colony size. R-curves of lamellar, bi-lamellar and equiaxed microstructures gradually increased with increasing the crack extension, which represents the fracture in lamellar, bi-lamellar and equiaxed microstructures accompanied by a ductile fracture with stable crack propagation. On the other hand, the equiaxed microstructure with the grain size of 0.63 μm was attributed nearly flat R-curve, which means the fracture accompanied by the unstable crack propagation. In the lamellar microstructures, crack propagation resistance was nearly the same despite the colony size decreased. On the other hand, crack propagation resistance in the bi-lamellar microstructure decreased with decreasing the colony size. Lamellar and bi-lamellar microstructures with the colony size of 57.5 μm presented a good balance between UTS and fracture toughness. This result can suggest that the lamellar and bi-lamellar microstructures have the great potential to possess much higher strength and fracture toughness through the colony size refinement.

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