

The Effect of Phase Stability on the Grain Growth Behavior of Beta Titanium Alloys

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Abstract

The grain growth kinetics of two beta titanium alloys, Ti-4733 (nominally, Ti-4Al-7Mo-3V-3Cr, in wt.%) and Ti-5553 (nominally, Ti-5Al-5Mo-5V-3Cr) was studied over a temperature range of 850 °C to 1000 °C. The more stable alloy i.e. Ti-4733, showed a smaller average grain size and growth rate compared to Ti-5553 at a same annealing condition. The results showed that the grain size distribution changes during annealing and affects the grain growth behavior so that the uniformity of grain size decreased initially down to a minimum and then increased with increasing temperature. The maximum uniformity of grain size (~ 78%) was achieved at 850 °C for Ti-4733 while this maximum was achieved for Ti-5553 only after annealing at 1000 °C which resulted in a significant grain growth. The grain growth exponent (n) and activation energy (Q) were calculated for the different annealing conditions and it was revealed that n is mainly affected by temperature and Q is generally influenced by time. The n value for Ti-4733 was found to be lower than that for Ti-5553 at all studied temperatures while a higher Q was calculated for Ti-4733. The lower n values and the higher Q were attributed to the solute drag effect and the high Mo content with low diffusivity in Ti matrix.

Keywords: Grain growth kinetics, Beta titanium alloy, Grain size distribution

1. Introduction

β and near β titanium alloys are widely used in the fields of aerospace, biomedical and chemical industries because of their high specific strength and attractive combinations of strength, toughness and fatigue resistance [1,2]. However, one of the disadvantages of β titanium alloys is rapid grain growth at elevated temperatures due to enhanced diffusion in the β phase resulting from its open body centered cubic structure. Grain size is an important determinant of mechanical properties during exposure of an alloy to elevated temperature. Hence, understanding of grain growth behavior is a prerequisite for designing and even controlling the process parameters and consequently final mechanical properties.

It is well-known that the driving force for grain growth is provided by the reduction in free energy due to the decrease in total grain boundary area [3,4]. Grain growth kinetics in the absence of pinning force can be described by measuring the average grain size as a function of time as follows [5,6]:

$$D - D_0 = kt^n \quad (1)$$

where D is the mean grain diameter, D_0 is the initial grain size, k is a constant depend on the grain boundary energy and mobility and independent of time, and n is the grain growth exponent.

The rate constant, k , is actually a function of temperature and activation energy and is expressed as [4]:

$$k = k_0 \exp\left(\frac{-Q}{RT}\right) \quad (2)$$

where k_0 is a material constant related to the specific interfacial energy of grain boundary, Q is the activation energy for grain growth, T is the temperature in Kelvin, and R is the gas constant.

A grain growth exponent of 0.5 was predicted by Burke and Turnbull [4] which has been recognized and commonly used as the theoretical value. However, it has been observed that usually the experimental values of n are less than 0.5 [6–8]. There are several factors affecting the grain growth kinetics which are not taken into account in Eq (1) including but not limited to solute segregation to grain boundaries [9], second phase particles [10–12], presence of a certain texture [13,14] and time depended grain size distribution [15,16].

Many researches have been devoted evaluating the grain growth kinetics of β -Ti alloys. Some of them reported a normal grain growth [8,10] while others [17] have reported a deviation from ideal classic grain growth kinetics. The effect of phase stability on the kinetics of grain growth and the evolution of grain size distribution during the heating of a β Ti alloy have rarely been systematically investigated.

Recently, the authors have designed and introduced the Ti-4733 β alloy with a nominal composition of Ti-4Al-7Mo-3V-3Cr (hereinafter all in wt.%) through the theoretical d-electron method which exhibits high compressive strength (~ 1400 MPa) and excellent compressive deformability ($\sim 35\%$) in the solution treated condition [18]. However, as a newly designed alloy, only few studies on its microstructure and mechanical properties has been done [2,19,20] and many other aspects including grain growth behavior has not been investigated yet. As regards, the present work aims to study the grain growth behavior in Ti-4733 and to compare with its commercial counterpart, Ti-5553, as a reference material, during the annealing treatment. In addition, the evaluation of grain size distribution during the grain growth process is investigated for both alloys.

2. Experimental

The alloys were produced by a double vacuum arc melting process. The chemical compositions of the alloys are listed in Table 1. After homogenization at 1100°C for 4 h, ingots were forged to the square billets with 50×50 mm in dimensions. The billets were then hot rolled at 750°C into the strips with 20 mm in thickness.

Table 1. Chemical composition (wt.%) and stability parameters of the alloys.

Al_{eq}	Mo_{eq}	Ti	N	O	Cr	V	Mo	Al	Alloy
6.14	12.80	Balance	0.021	0.152	2.9	3.1	7.1	4.2	Ti-4733
7.03	11.57	Balance	0.018	0.147	2.9	4.7	4.8	5.2	Ti-5553

For grain growth study, the as-rolled samples were isothermally annealed in a box furnace at 850, 900, 950 and 1000 °C for 15, 30, 60, 120 and 240 min followed by rapid quenching. All the samples grinded to completely remove the α -case formed during the heat treatment. The specimens for optical microscopy were prepared with the standard metallographic procedure and etched in a modified Kroll's reagent (6% HF + 18% HNO₃ + 76% H₂O) to reveal the grain boundaries. Microstructure characterization was performed on an Olympus GX51 light optical microscope. At least ten figures were captured for each sample to minimize the error in grain size measurements. The grain boundaries in all figures were clearly traced to identify the grains and to do the measurements appropriately by Clemex image analysis software. The grain size measurements were done according to ASTM-E112 standard. The uniformity of the β grain size was determined by a grain ratio $\omega = N/N_t$ as described in Refs. [21,22], where N is the number of the grains of which sizes are in the range of 0.6–1.4D (D is the average β grain size) and N_t is the total number of the measured β grains. Obviously, the higher the ratio, the more uniform the grain size distribution. Phase identification was performed using a Rigaku SmartLab 9 kW X-ray diffraction (XRD) unit with Co-K α radiation ($\lambda=0.179$ nm) in the angular (2θ) range of 40–130°.

3. Results

The initial microstructure of the hot-rolled Ti-4733 and Ti-5553 alloys is shown in Fig. 1. The microstructure of both alloys contains elongated and large β grains along with primary α phase. The primary α phase forms during the hot rolling in the $\alpha+\beta$ phase region (750 °C).

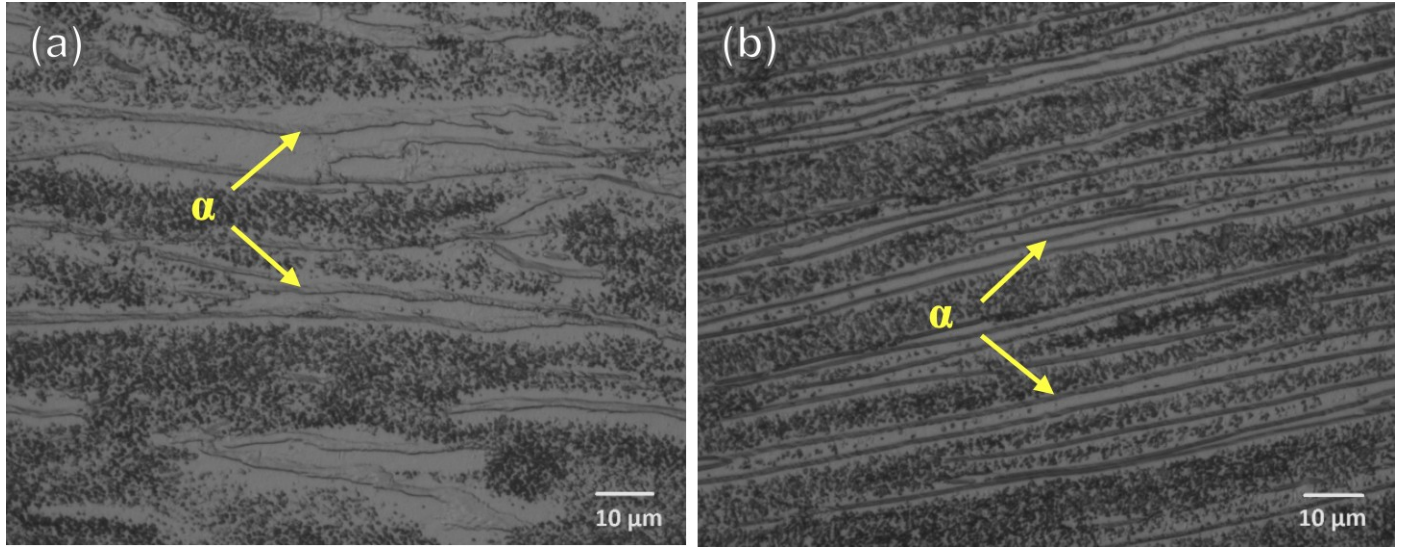


Figure 1. Light optical microstructure of the as-rolled specimens for the a) Ti-5553 and b) Ti-4733 alloys.

Fig.2 shows the phase diagram calculation of the studied samples using JMatPro software. It can be seen that the β -transus temperature (T_β) i.e. when the microstructure is fully β phase is about 784 °C and 811 °C for Ti-4733 and Ti-5552, respectively.

T_β was also theoretically calculated according to the following empirical equation [23]:

$$T_\beta = 882 + 21.1[Al]_{eq} - 9.5[Mo]_{eq} \quad (3)$$

where $[Al]_{eq}$ and $[Mo]_{eq}$ are aluminum and molybdenum equivalents which can be calculated as follows [24]:

$$[Mo]_{eq} = [Mo] + 0.2[Ta] + 0.28[Nb] + 0.4[W] + 0.67[V] + 1.25[Cr] + 1.25[Ni] + 1.7[Mn] + 1.7[Co] + 2.5[Fe] \quad (4)$$

$$[Al]_{eq} = [Al] + 0.17[Zr] + 0.33[Sn] + 10[O] + 10[C] + 20[N] \quad (5)$$

The calculated T_β based on Eq. (3) were about 890 °C for Ti-4733 and 920 °C for Ti-5553 which were higher than JMatPro calculations. To be more precise, a tremendous effort was done to determine T_β using microscopic microstructures for the different annealing conditions. The measured values were approximately 850 °C and 870 °C for Ti-4733 and Ti-5553, respectively. JMatPro was also employed to calculate the continuous cooling transformation (CCT) diagrams as given in Fig. 3. The results indicate that the microstructure of both samples after annealing and rapid quenching is single β phase. In addition, XRD analysis was performed to investigate the accuracy of calculations. As an example, Fig. 4 (a and b) shows the XRD results for both alloys after annealing at 850 °C for 240 min and subsequent quenching. The pattern indicates that Ti-4733 has a single β phase structure while the pattern of Ti-5553 contains a weak peak relating to α phase. In coincidence with XRD result, the optical microstructure of Ti-5553 also confirms the precipitation of alpha phase at grain boundaries as shown in Fig. 4c. It seems that the calculations are slightly far from the reality, most probably the over/underestimation is due to not considering the effect of initial grain size and/or heating rate on critical phase transformation temperatures.

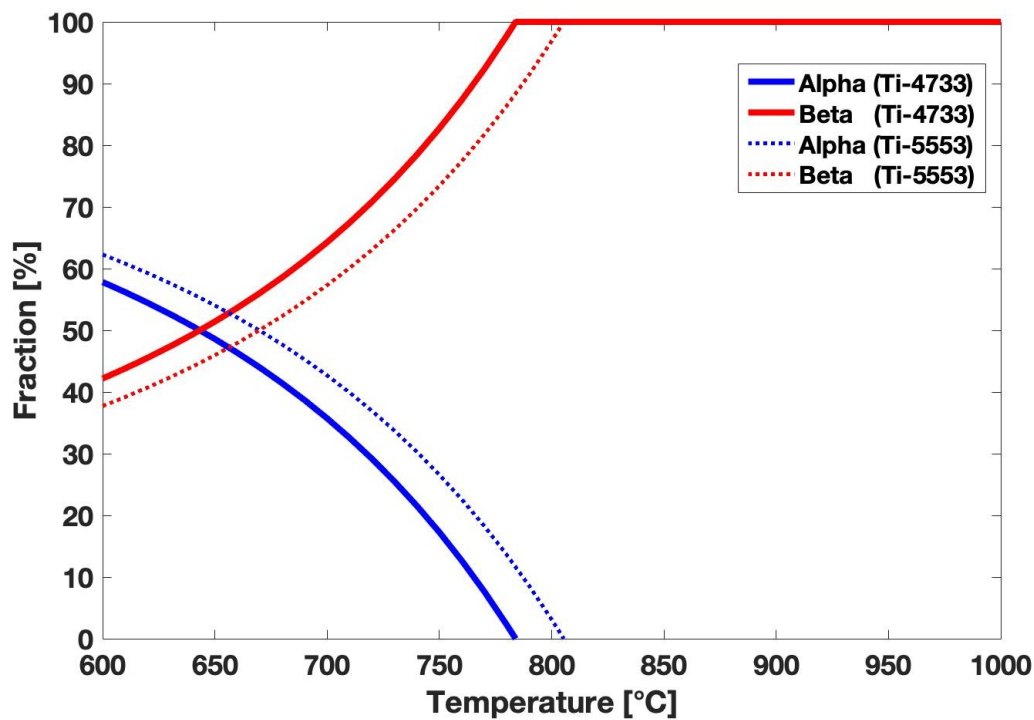


Figure 2. Phase diagram of Ti-4733 and Ti-5553.

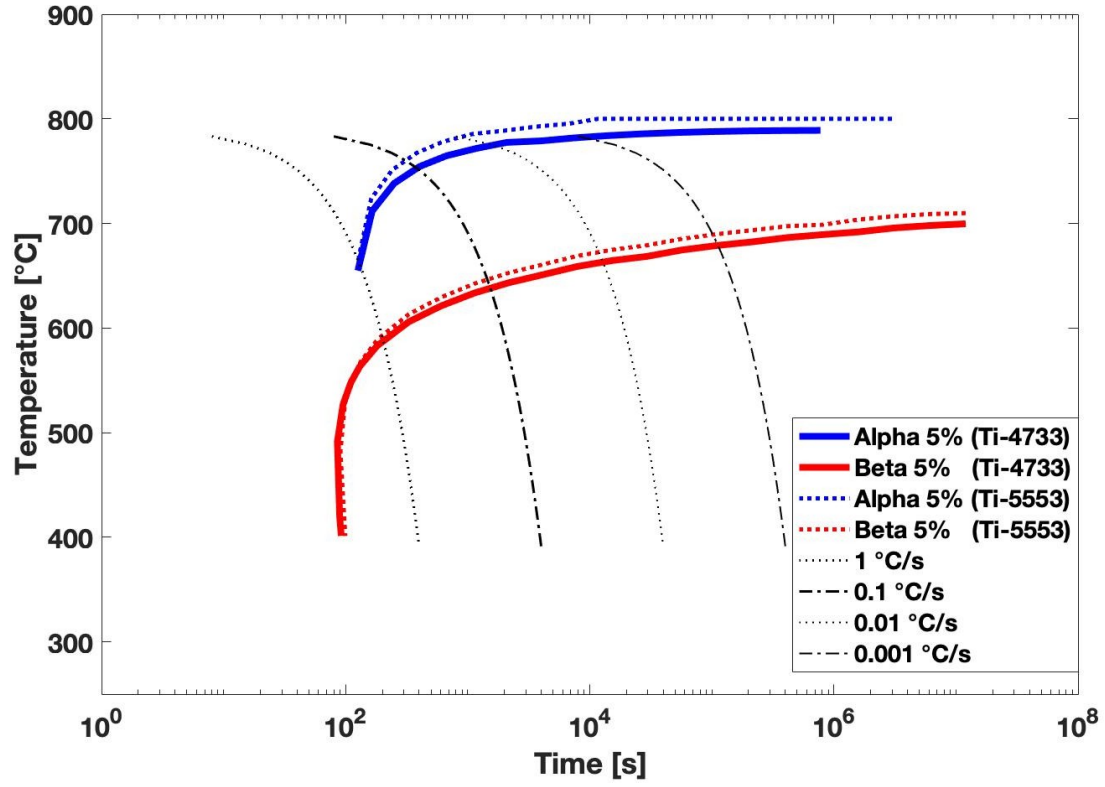


Figure 3. CCT diagram of Ti-4733 and Ti-5553 using JMatPro software.

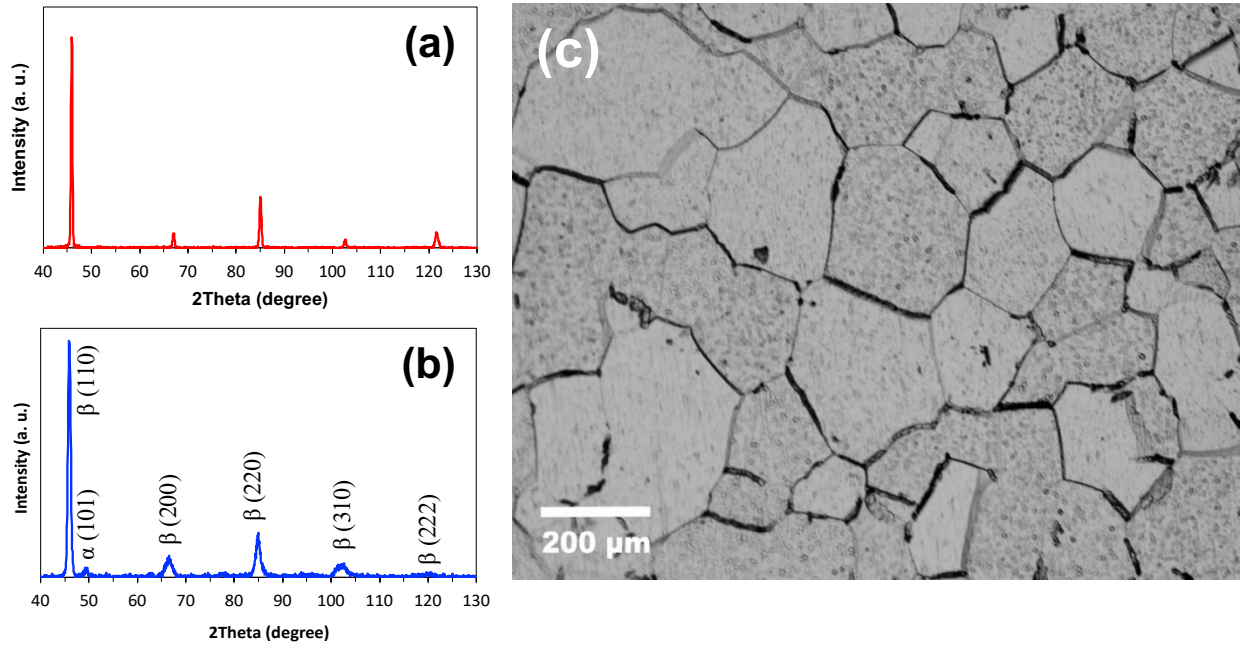


Figure 4. XRD patterns of a) Ti-4733 and b) Ti-5553 after annealing at 850 °C for 240 min and c) formation of α phase at beta grain boundaries confirming the presence of α phase in Ti-5553 annealed at 850 °C for 240 min.

Figures 5-8 show the microstructure of Ti-4733 and Ti-5553 after annealing at 850, 900, 950 and 1000 °C for different holding times. As observed, annealing of the hot-rolled samples resulted in solution of the primary α phase and fully recrystallization of the deformed β phase, so that the annealed microstructures consisted of equiaxed β single phase grains. According to Figs. 5-8, as expected, the grains in both alloys became coarser with increasing the annealing time and temperature. As shown in Figs. 5-8, the β grains in Ti-4733 are smaller than those in Ti-5553 after annealing in a same condition.

To assess the grain size variation, the average grain size values as a function of annealing time and temperature are plotted in Fig. 9. The results showed that the alloys annealed at 850 °C (specifically Ti-5553) exhibited very slow grain growth and actually insensitive to the holding time. This is most probably because of little undissolved alpha phase remained in the beta matrix. As the annealing temperature of 850 °C is close to the T_β of Ti-4733 or even below that for Ti-5553, the presence of small amount of α phase is possible as it is evident from Fig. 4c as well. The α phase acts as an inhibitor of β grain growth and it hinders the grain boundaries movement. However, both alloys annealed at/above 900 °C showed rapid grain growth up to 120 min and slower grain growth afterward. This is due to this fact that with increasing the grain size, the grain boundary area per unit of volume decreases leading to a decrease in interfacial energy per unit volume. Hence, the driving force for additional growth decreases and results in a slower growth kinetic. As seen, the growth rate increases with increasing temperature.

The variation of grain size against time obviously shows that the grain growth of Ti-5553 is faster than that of Ti-4733 in the same annealing condition. After annealing at 1000 °C for 240 min the average grain size was calculated to be 560 and 630 μm for Ti-4733 and Ti-5553, respectively.

Fig. 10 presents the β grain size distribution of the alloys in different annealing conditions determined from the image analysis of corresponding optical micrographs.

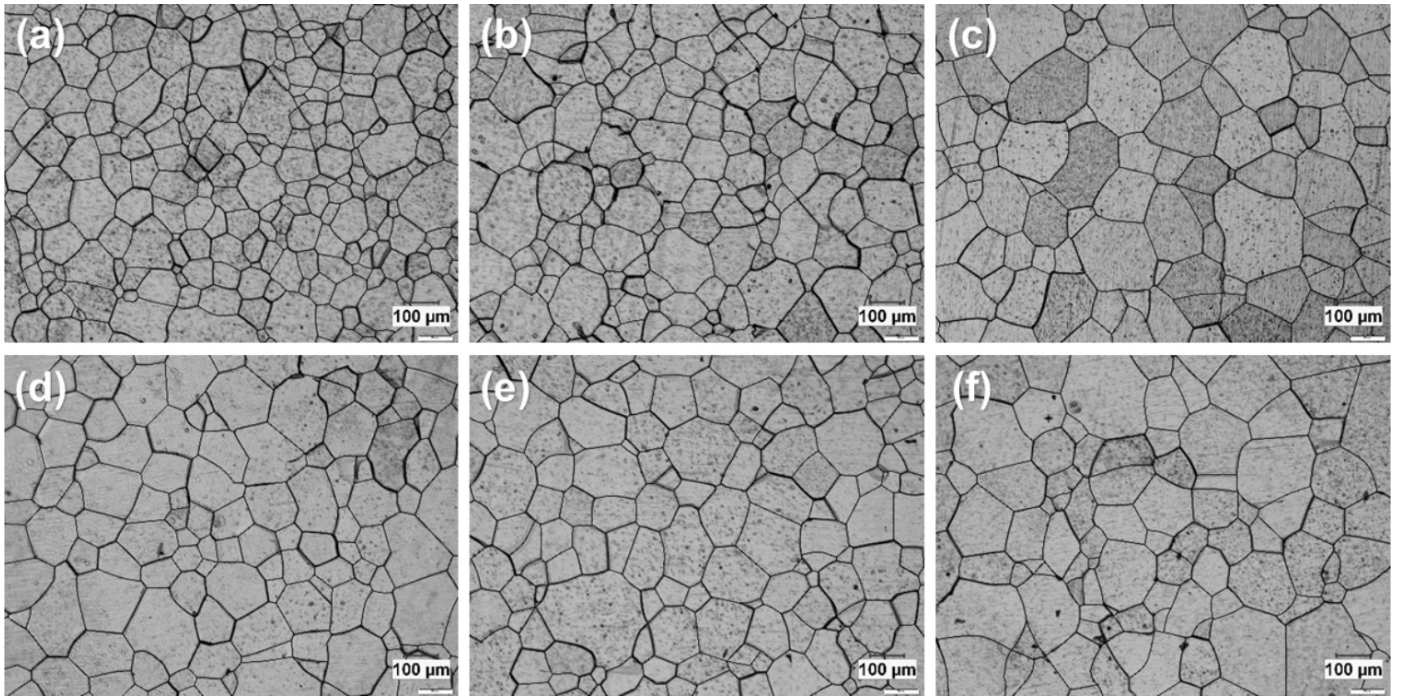


Figure 5. Light optical microstructures of Ti-4733 (a, b, c) and Ti-5553 (d, e, f) samples after isothermal annealing at 850 °C for (a, d) 30 min, (b, e) 60 min and (c, f) 120 min.

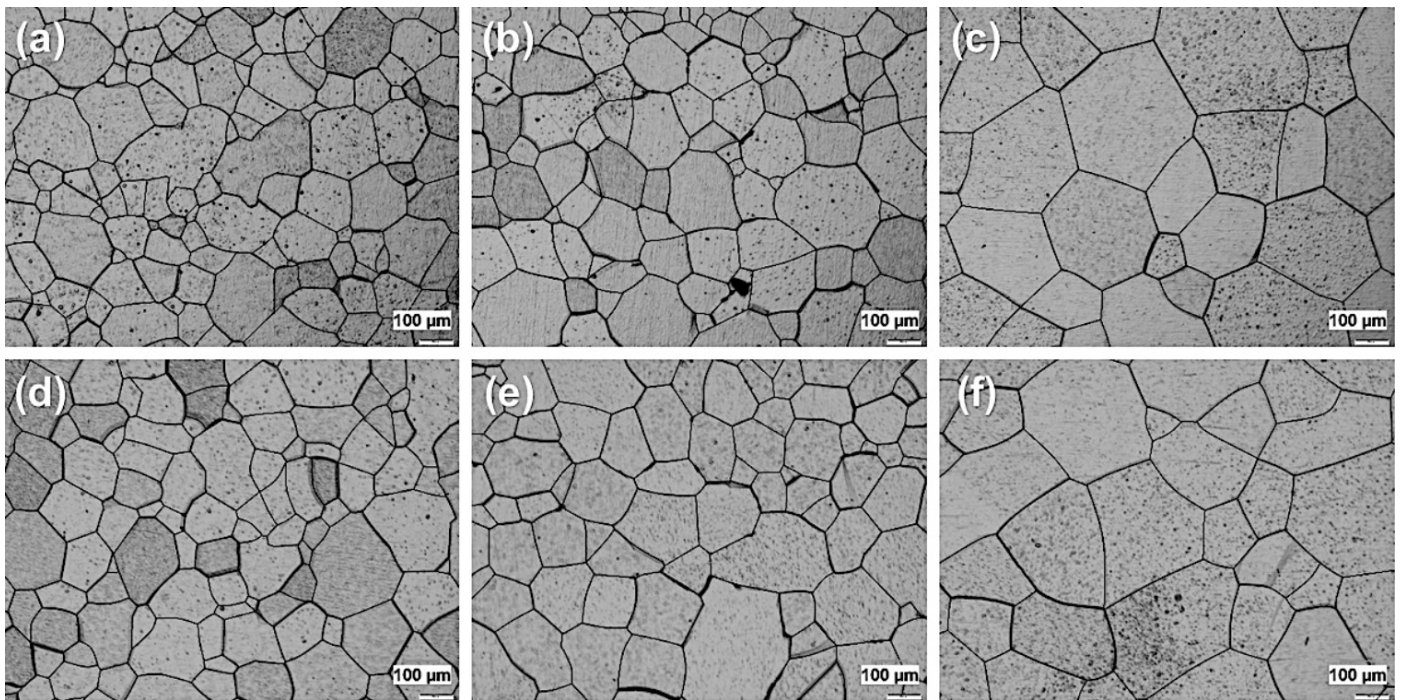


Figure 6. Light optical microstructures of Ti-4733 (a, b, c) and Ti-5553 (d, e, f) samples after isothermal annealing at 900 °C for (a, d) 30 min, (b, e) 60 min and (c, f) 120 min.

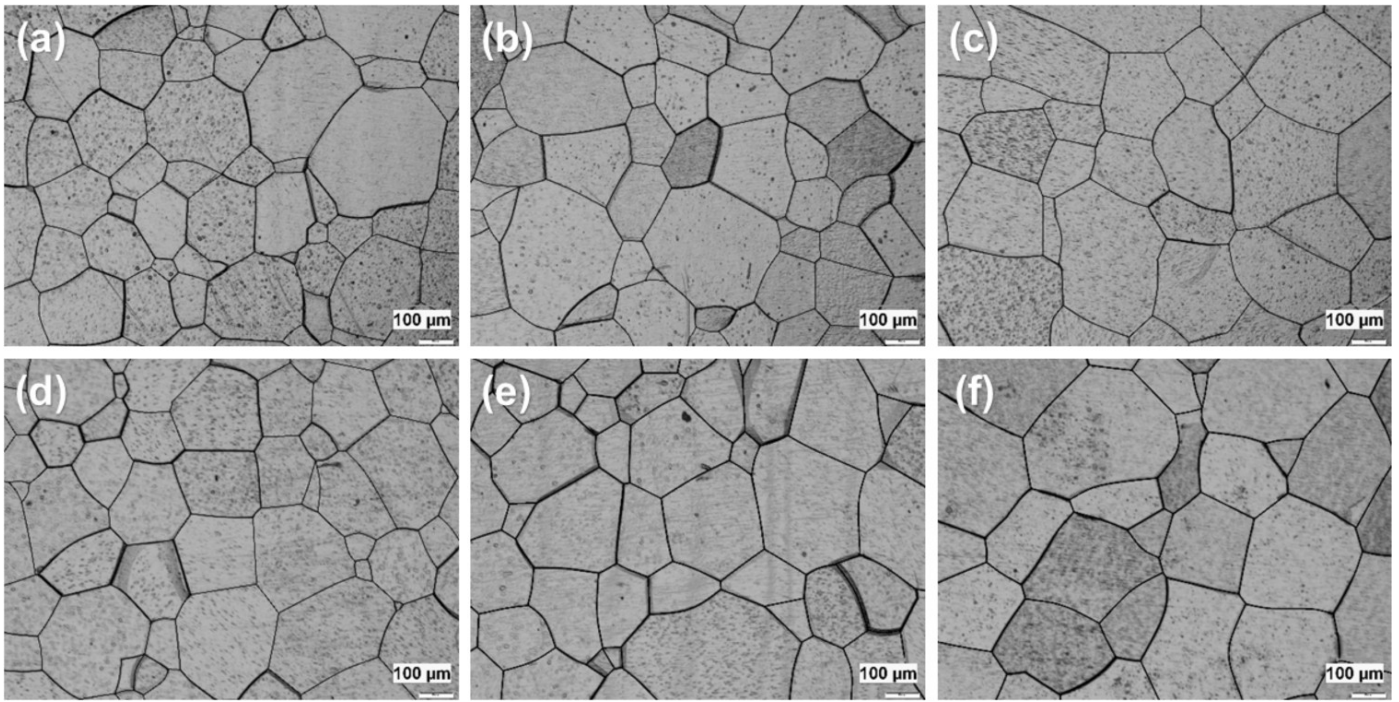


Figure 7. Light optical microstructures of Ti-4733 (a, b, c) and Ti-5553 (d, e, f) samples after isothermal annealing at 950 °C for (a, d) 30 min, (b, e) 60 min and (c, f) 120 min.

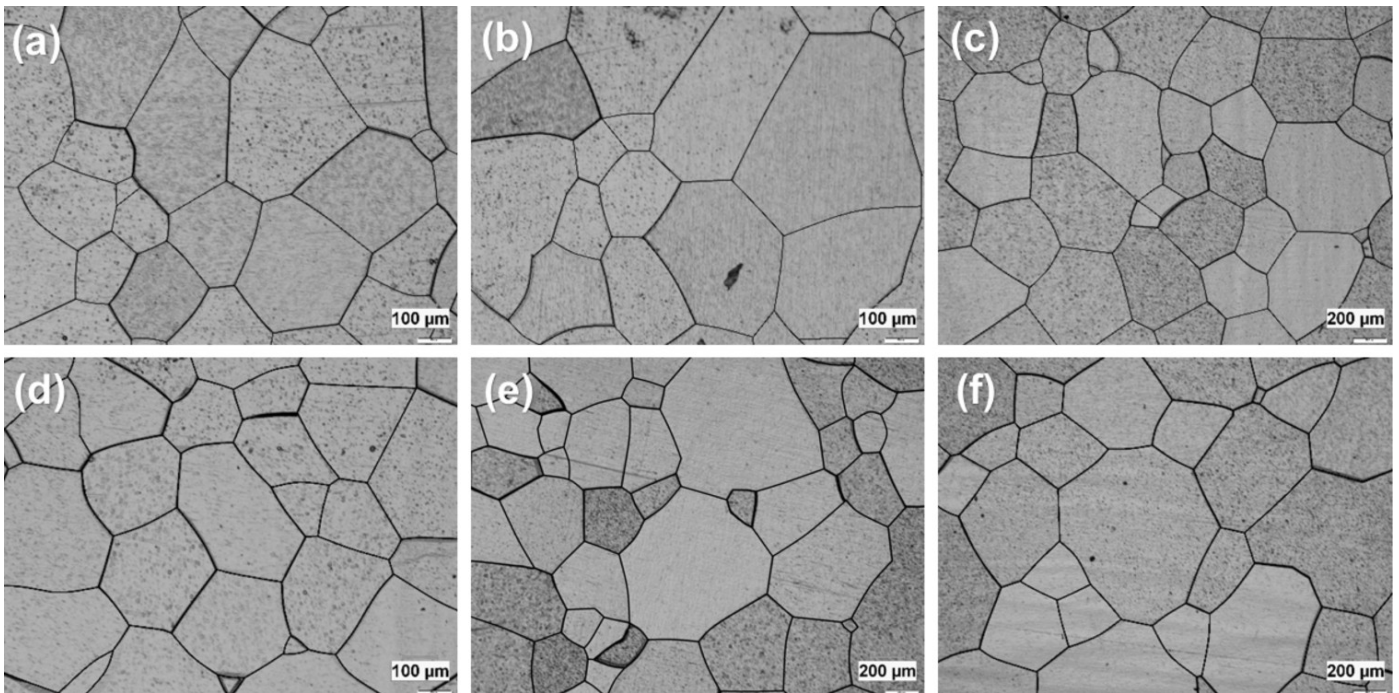


Figure 8. Light optical microstructures of Ti-4733 (a, b, c) and Ti-5553 (d, e, f) samples after isothermal annealing at 1000 °C for (a, d) 30 min, (b, e) 60 min and (c, f) 120 min. Please notice to the different magnification of the images.

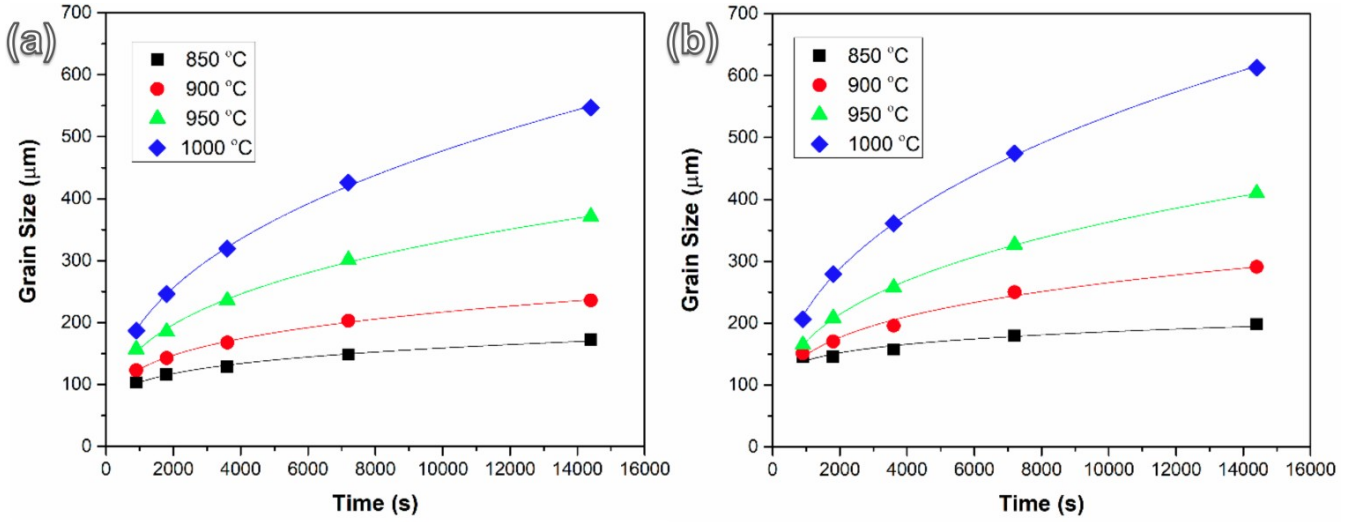


Figure 9. Variation of average grain size as a function of annealing time at different temperatures for a) Ti-4733 and b) Ti-5553.

It is obvious that the distributions are not same before and after grain growth. Moreover, the results show that the uniformity of distribution initially decreases with increasing temperature and the distribution becomes more broaden (specially for durations of 15 and 30 min), while for longer durations (120 and 240 min), the distributions show better uniformity and tighter range i.e. a normal distribution with increasing temperature. In other words, the range of grain size changes and the maximum relative fraction shifts with increasing the time and temperature meaning that the grain size distribution also changes dynamically during heating which may affect the kinetics of grain growth.

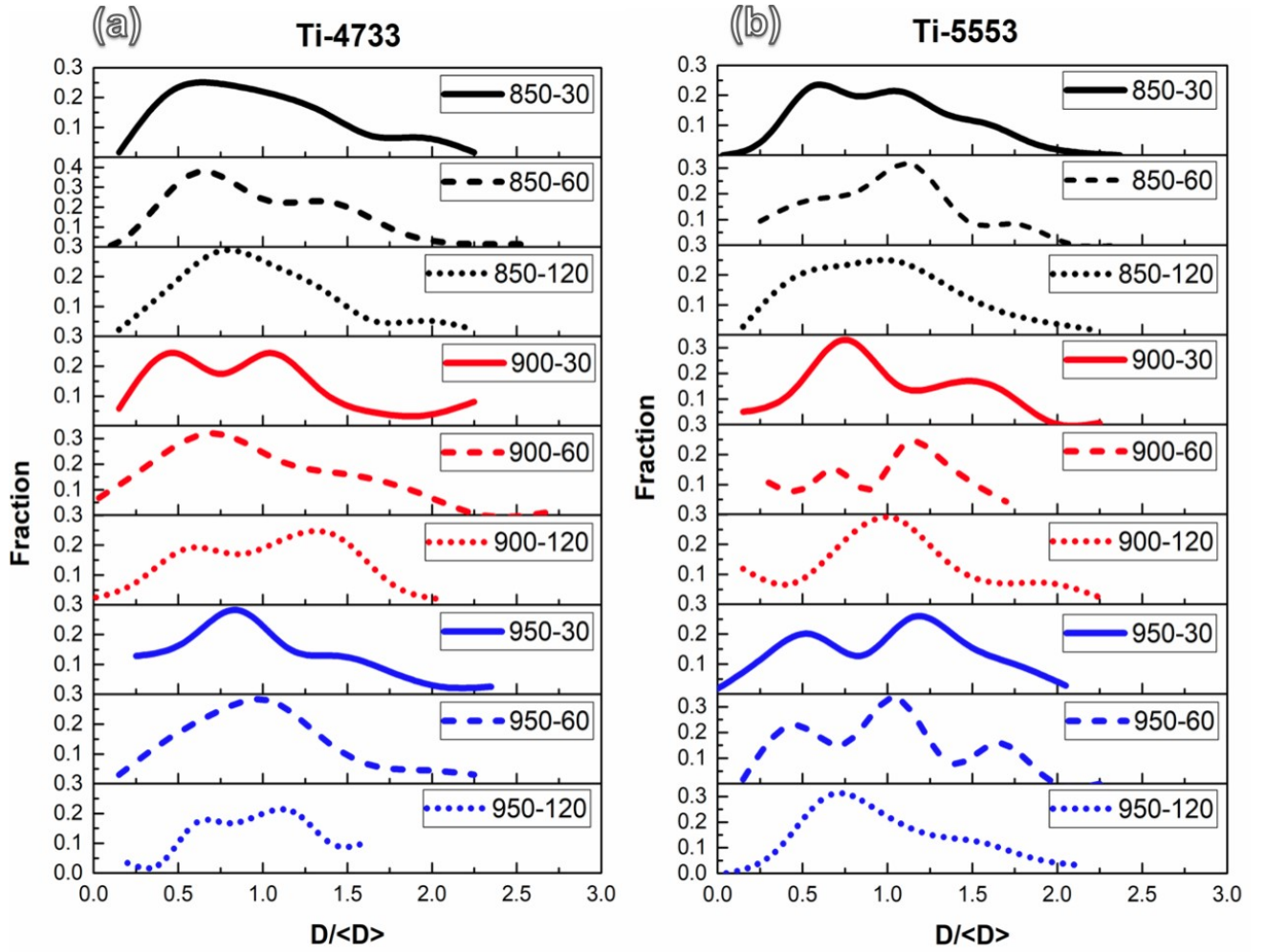


Figure 10. Grain size distribution of Ti-4733 (a) and Ti-5553 (b) samples annealed at different condition.

To better understand and simplify the evolution of grain size distribution, the parameter of grain size uniformity (ω) was considered. Figs. 11 (a and b) show the variations of ω with annealing time at different temperatures for Ti-4733 and Ti-5553, respectively. It can be observed that the trend of the variation of ω is different in different ranges of annealing time so that it first decreases down to a minimum and afterward increases. This minimum is reached at shorter times by Ti-5553 meaning that the grain structure of Ti-5553 becomes nonuniform faster than that of Ti-4733. However, the values of ω for both alloys have similar variations except in the case of Ti-5553 annealed at 850 °C showing an almost stable uniformity. This is attributed to the presence of α phase which prevents the grain growth and keeps the grain size uniformity in a constant level during soaking at 850 °C.

The grain growth rate increases with increasing temperature (as seen in Fig. 9) and the driving force for grain growth makes large grains grow and small grains disappear to reduce the grain boundary area. Accompanying this process is that at a short period of time such as 30 min, the ω decreases with increasing the temperature. After that given time, the grain size tends to be uniform with increasing the time and the higher the temperature the shorter the time is needed to reach this state. Generally, Ti-4733 shows a higher uniformity compared to Ti-5553 at the same annealing condition. It is also worth to mention that the maximum uniformity was achieved at 850 °C for Ti-4733 when the average grain size was 172 μm , while that maximum was achieved for Ti-5553 only at high temperature of 1000 °C which resulted in a large average grain size of 613 μm . This means that a uniform distribution of smaller grain size can be achieved in Ti-4733.

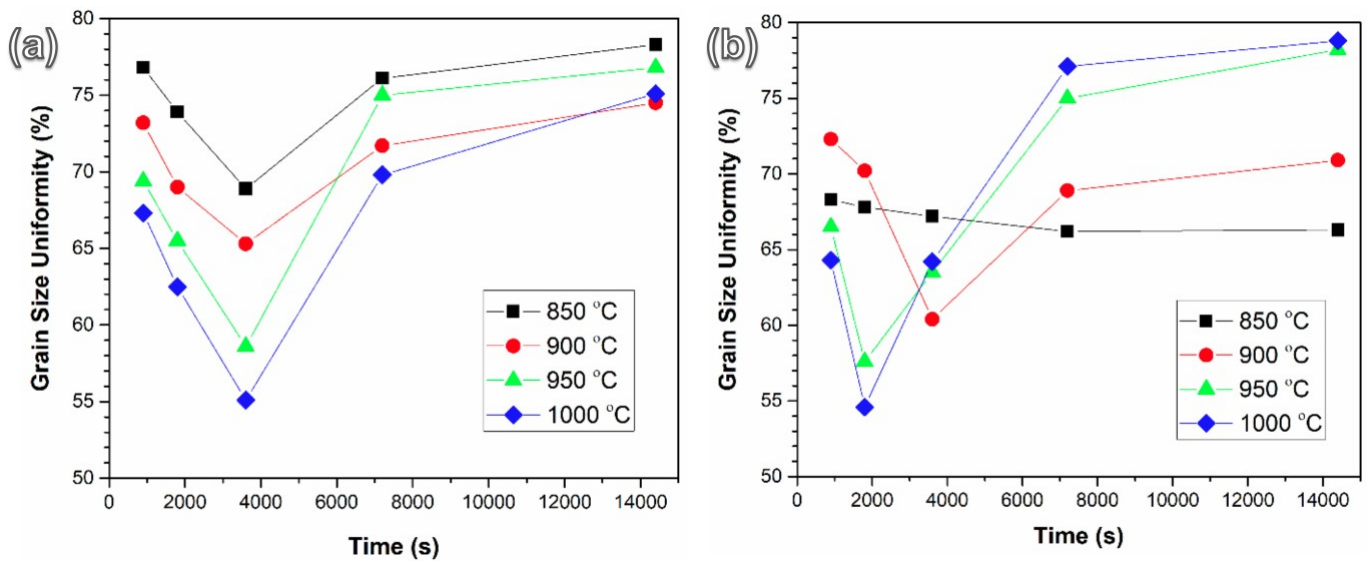


Figure 11. The grain size uniformity as a function of annealing time at different temperatures for a) Ti-4733 and b) Ti-5553.

According to Eq. (1), the grain growth exponent, n , values at different temperatures can be determined by fitting the experimental grain size data as shown in Fig. 12. The calculated n values for both alloys are presented in Fig. 13 and Table 2. These data show that the growth exponent increases with temperature. This behavior which is in agreement with previous works [13,22,25] may be related to the enhancement of diffusion rate with increasing temperature.

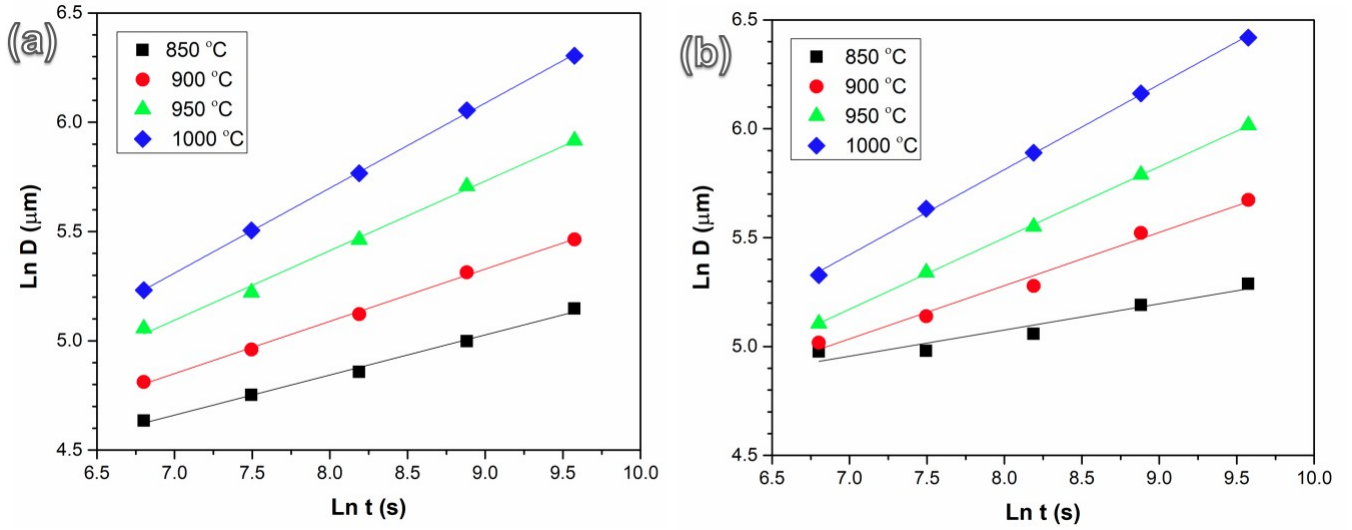


Figure 12: Variation of $\ln D$ as a function of $\ln t$ and corresponding fitted lines at different temperatures for a) Ti-4733 and b) Ti-5553.

In general, the values of grain growth exponent for titanium and its alloys including the present results are high compared to that of other metals and alloys. Aluminum (99.99 wt.%) shows a growth exponent of 0.1 at 400 °C, which increases to 0.3 at 600 °C [26]; for austenite grains in carbon steels (0.8 wt.% C), it increases from 0.1 at 760 °C to 0.23 at 980 °C [26]. In the case of α -Fe, where the crystal structure (bcc, as in the present alloys) is more open and prone to diffusion, the growth exponent is higher so that increases from 0.2 at 600 °C to nearly 0.5 at 800 °C. As can be seen, in all cases the n value determined here are smaller than 0.5 and in all cases the n value for Ti-4733 is lower than that for Ti-5553. The Ti-4733 alloy has a 12.80 Mo_{eq} giving a solute drag effect and consequently leads to the lower n values in comparison to the Ti-5553 alloy. As an exemption, the lower n value of Ti-5553 at 850 °C can be attributed to the presence of α -phase as mentioned before.

The activation energies (Q) for grain growth in both alloys were determined by plotting the k versus $1/T$ as shown in Fig. 14. The activation energies for grain growth at different temperatures determined for Ti-4733 and Ti-5553 are shown in Fig. 15 and Table 2. The calculated values for Q

were in the range of 250-900 kJ/mol and increased with the holding time. The differences in Q is found to be mainly due to annealing temperature or chemical composition of the alloy. The increase of temperature and the decrease of solute content both promote the diffusion of atoms. Because of high solute (specifically Mo) content, the diffusion in Ti-4733 across grain boundary should be more difficult than that in Ti-5553. Therefore, Ti-4733 needs higher activation energy for grain growth. Similar results on the variation of Q with temperature and solute content have been reported for other β Ti alloys [10,22,27,28].

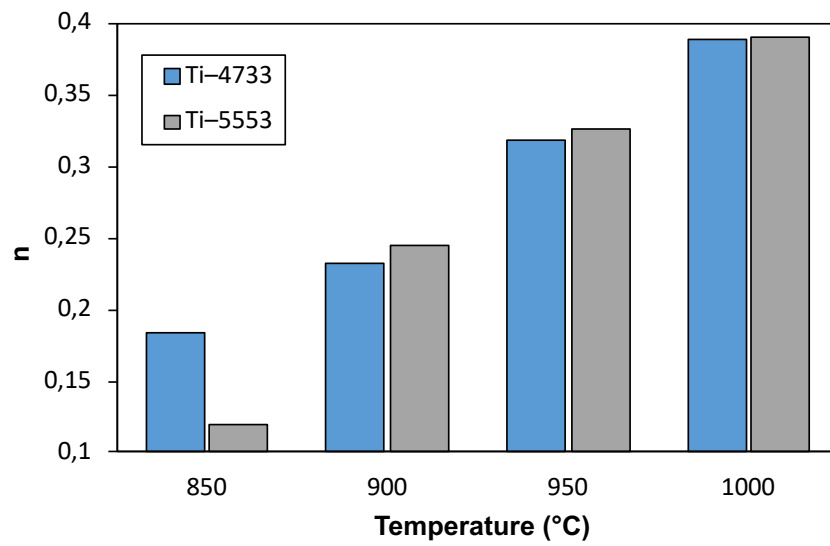


Figure 13. The values of grain growth exponent, n , at different temperatures for Ti-4733 and Ti-5553.

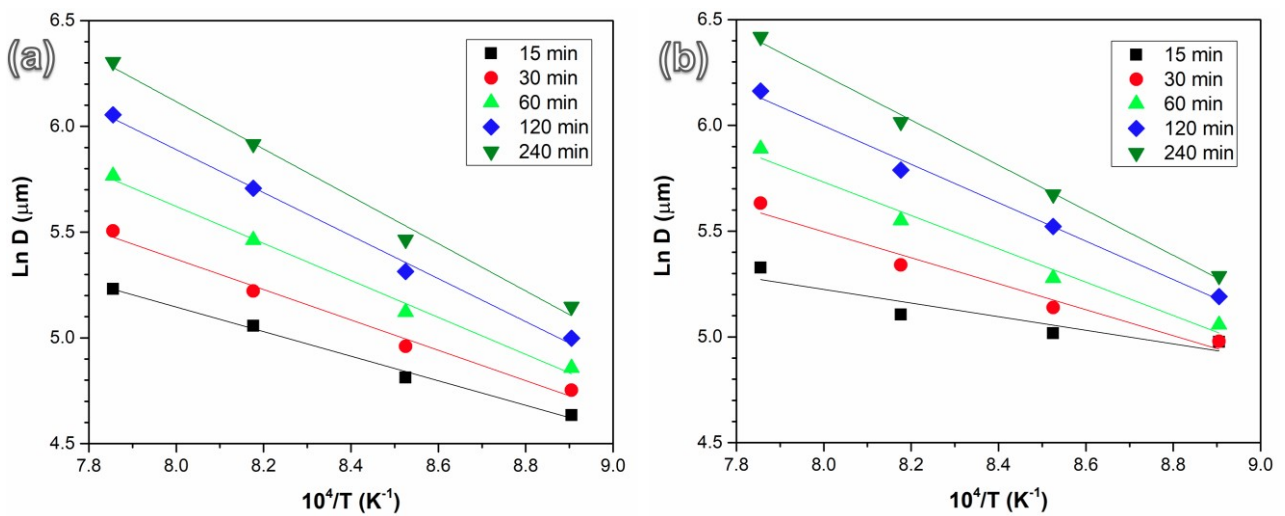


Figure 14. Variation of $\ln D$ as a function of $1/T$ and corresponding fitted lines at different holding times for a) Ti-4733 and b) Ti-5553.

According to Fig. 15, the activation energy increases for both alloys with increasing the holding time while Ti-5553 shows a higher rate of increasing. Some studies [10,22,27] have evaluated the effect of solution time on the activation energy of titanium alloys. They showed the activation energy for grain growth of titanium alloy increases with increasing the solution time. This increasing of the activation energy can be explained in term of “solute drag effect”. At the early stage of annealing, the velocity of grain boundary migration is faster than the diffusion of solute atoms, and the solute atoms do not aggregate in the boundaries. As a result, the grain boundaries are free from the “solute drag effect” and have lower activation energy [27,29]. The driving force of grain growth decreases with increasing the solution time due to increasing the average grain size inducing a decrease in grain boundary area, and then the migration velocity of grain boundary decreases. The amount of solute atom which aggregated at boundaries also increases with decreasing the migration velocity of grain boundary [27,29]. Therefore, enrichment of solute atoms in the boundaries induces the drag effect, and the activation energy for grain growth increases with increasing the solution time.

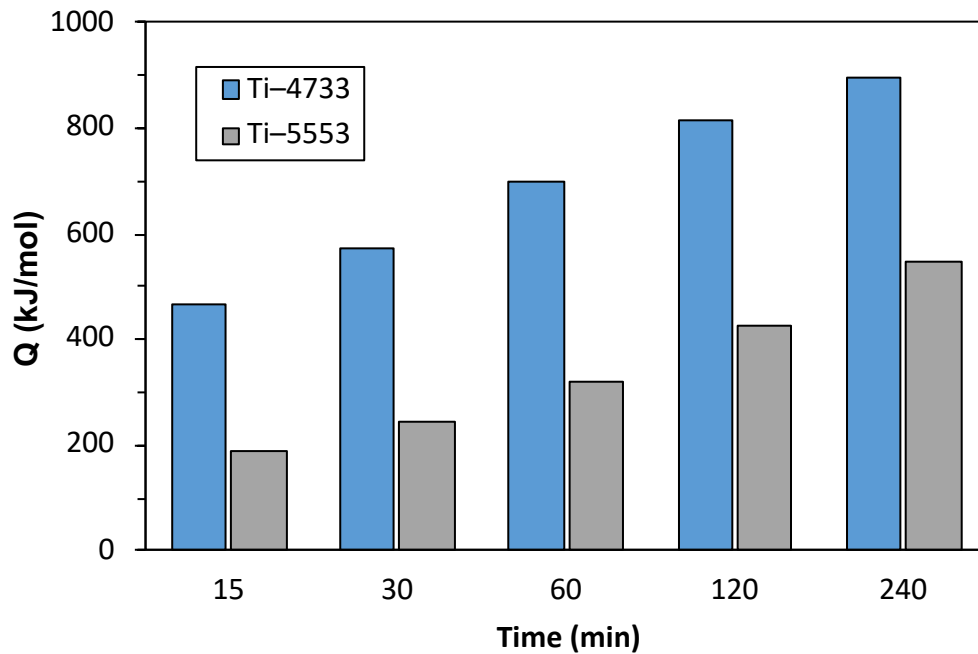


Figure 15. The values of activation energy for grain growth, Q , for Ti-4733 and Ti-5553 for different soaking times.

Table 2 presents Q and n values obtained at different annealing conditions for some β Ti alloys in comparison to those for the present alloys. The variation in the activation energy for different alloys is considerable while the grain growth exponent is almost in a same range. This suggests that Q is more sensitive to chemical composition and annealing condition compared to n. The activation energy for self-diffusion in titanium has been reported to be between 145 to 315 kJ/mol [30] which is lower than the calculated value for the present alloys. The values of 312 and 347 have also been reported for Ti–6Al–4V (in the β phase) [31] and Beta III [32] alloys, respectively. However, a high value of 644 kJ/mol at 1000 °C has also been reported for Ti–2Al–9.2Mo–2Fe by Lee et al. [25]. Similar to the results reported by Lee et al. [25], the high values of Q in the present alloys can be ascribed to the high content of Mo with low diffusivity and the significant solute drag effects in the present alloys retarding the mobility of grain boundaries and increasing the activation energy.

Table 2. Comparison of the activation energy (Q) and grain growth exponent (n) of different β Ti alloys obtained at different annealing conditions with those of the present alloys.

Alloy	T beta (°C)	Temperature (°C)	Time (min)	Q (kJ/mol)	n	Ref.
Beta Ti	883	900-1000	5-120	20	0.05-0.5	[10]
TG6	1045	$T_{\beta}+10-30$	5-180	120.4- 212.5	0.35-0.38	[24]
Ti17	895	$T_{\beta}+10-30$	5-180	290.5- 378.5	0.27-0.3	[24]
Ti–2Al–9.2Mo– 2Fe	827	820-1000	30-720	442.8- 644.3	0.09-0.22	[25]
TC4-DT	975	985, 995, 1005	2-120	86.8-129.7	0.34-0.35	[28]
Ti-20Mo	710	650-900	15-360	272.16.00	0.26 (at 800 °C)	[27]
Beta21S	807	810-960	30-600	320	0.1-0.125	[12]
Beta21S-0.1B	>810	810-960	30-600	914	0.02-0.06	[12]
Ti-4733	850	850-1000	30-240	465-895	0.18-0.39	Present work
Ti-5553	870	850-1000	30-240	258-853	0.12-0.39	Present work

4. Conclusions

The grain growth behavior of two β Ti alloys with different levels of phase stability was studied during annealing at the temperature range of 850-1000 °C for different durations of 15 to 240 min. The main results are summarized as follows:

- 1) Ti-4733 showed a smaller average grain size and slower grain growth rate compared to Ti-5553 in all annealing conditions which was attributed to the higher β phase stability of Ti-4733.
- 2) The grain growth exponent, n , increased with increasing temperature and was found to be in the range of 0.18-0.39 for both alloys while Ti-4733 showed a smaller n value in almost all annealing conditions.
- 3) The activation energy of grain growth, Q , showed a time dependency and was found to be in the range of 465-895 kJ/mol for Ti-4733 and 187-547 kJ/mol for Ti-5553. The high value of Q for Ti-4733 was attributed to the solute drag effect and the high content of Mo with low diffusivity in this alloy.
- 4) It was shown that the uniformity of grain size decreased initially down to a minimum and then increased with increasing temperature.
- 5) The maximum uniformity of grain size ($\sim 78\%$) was achieved at 850 °C for Ti-4733 with an average grain size of 172 μm while this maximum was achieved for Ti-5553 only after annealing at 1000 °C which resulted in a large average grain size of 613 μm .

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