

## STUDY OF ISOTHERMAL PHASE TRANSFORMATION BEHAVIOR OF TC6 TITANIUM ALLOY DURING HEAT TREATMENT

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**Abstract-** We report the phase transformation behavior of TC6 titanium alloy to establish isothermal phase transformation model and constitutive equation in hot working process of TC6 titanium alloy. The phase diagram of the alloy was obtained by thermodynamic calculation, and the effect of temperature on the distribution of each phase was calculated. The phase transition point of TC6 titanium alloy was observed at 985°C temperature. The kinetics showed that the amount of  $\alpha$  phase decreased gradually and the quantity of  $\beta$  phase increased with increasing temperature. The isothermal transformation kinetics involved the temperature between 750°C and phase transition temperatures with different holding times. According to kinetics, different double heat treatment processes in two stages were designed and performed. With the increase of holding temperature of 1<sup>st</sup> stage heat treatment, primary  $\alpha$  phase was decreased and the decreasing rate accelerated at higher temperature. The characterization of both  $\alpha$  and  $\beta$  phase can be effectively controlled by double heat treatment process in two stages. Thus makes it possible to obtain proper process to get different properties of this alloy that needed.

**Keywords:** TC6 titanium alloy, Heat treatment, Isothermal phase transformation, microstructure.

### 1. INTRODUCTION

Two phase titanium alloys are widely used in aerospace industry due to their high specific strength, fatigue resistance and excellent corrosion resistance [1-5]. In spite of high cost of titanium alloys, their properties have promoted their use in a variety of applications in other fields such as- automotive, energy, power generation, biomedical and sport industries [3-5]. The mechanical properties of two phase alloys are highly affected by microstructural characteristics, which may be controlled by thermal and thermo-mechanical treatments [1-3]. So, the precise understanding of  $\alpha$  to  $\beta$  phase transformation kinetics is prime needed for the optimization of functional design of these alloys. In this perspective, recent attempt is to study the isothermal phase transformation behavior of TC6 titanium alloy during heat treatment process, which is a two phase alloy. It possesses good ductility and toughness, high relative strength and good resistance against heat and corrosion. These properties make it an ideal choice for many aerospace applications, such as- aerofoil blade and disc in the aviation and aerospace industry [6]. This research is concerned with the isothermal phase transformations and phase transformation kinetics that occur in TC6 titanium alloy. Researchers have already investigated the deformation behavior of TC6 titanium alloy and mechanical properties were evaluated [6-14]; either experimentally or using simulation. G. Li et al. [15] studied the 3-dimensional microstructure based

mechanical behaviour of TC6 titanium alloy, where they influenced the  $\alpha$  and  $\beta$  phase contained micromechanical behaviour. The microstructure and properties of TC6 titanium alloy is dependent on  $\alpha$  and  $\beta$  phase as well as their transformation kinetics, which is not studied significantly. Moreover, researchers have studied the phase transformation behavior as well as kinetics of some Ti alloys [16, 17]. But those behaviours are still unexplored for TC6 titanium alloys. So, the aim of this study is to compare the changes of isothermal transformation kinetics at different temperature at different time interval at different cooling conditions. The phase transformation behavior of TC6 titanium alloy has been investigated in order to establish the isothermal phase transformation model and constitutive equation during hot working process. The phase diagram of the alloy was gained by thermodynamic calculation process, and the influence of temperature on the distribution of each phase was calculated. The phase transformation temperature of TC6 alloy was determined by metallographic method which was 985°C temperature. The effect of temperature on the element of each phase was obtained by the phase diagram simulation. Lamellar microstructure was originated upon cooling from  $\beta$  phase and the primary  $\alpha$  remained unchanged through the transformation of  $\beta$  phase. Based on thermodynamic-kinetic research, it is disclosed that the amount of  $\alpha$  phase decreased gradually and the quantity of  $\beta$  phase increased when the temperature increased.

The phase transformation rate was rapid above 750°C. If the temperature holds in the  $\alpha$ - $\beta$  field for a long time, it resulted in the growth of primary  $\alpha$  phase at the expense of  $\beta$  until the equilibrium phase was observed. The study of isothermal transformation kinetics involved the temperature between 750°C and phase transition temperatures with different holding times. The phase transition time to reach equilibrium was evaluated by quantitative characterization of microstructures. At 750°C, 800°C, 850°C, 900°C, 950°C temperature, the time of phase transition to obtain the equilibrium conditions were 180 min, 150 min, 125 min, 90 min and 80 min respectively. Isothermal transformation kinetics models at each temperature were built, with drawn TTT curves. According to kinetics, different double heat treatment processes in two-phase section were designed and performed. The contents and patterns of each phase in TC6 alloy were quantitatively analyzed. Primary  $\alpha$  phase was deeply controlled by first step of heat treatment and with the increase of holding temperature of first step heat treatment primary  $\alpha$  phase was decreased and the decreasing rate accelerated at higher temperature can also influence the content of primary  $\alpha$  phase. Lamellar  $\alpha$  phase and  $\beta$  phase can also be controlled effectively by second step of heat treatment. So, the characterization of both  $\alpha$  and  $\beta$  phase can be effectively controlled by double heat treatment process in two steps. Thus proper process controlled investigation facilitates to the development various applications of TC6 titanium alloy.

## 2. EXPERIMENTAL

The as purchased test material was cylindrical sized TC6 titanium alloy, belongs to  $\alpha + \beta$  type titanium alloy, where the composition was Ti-6.29Al-1.42Cr-0.42Fe-2.71Mo-0.33Si. The content of the alloy elements were measured by inductively coupled plasma spectrometer analyzer and distillation. The specific components are shown in Table 1.

Table 1 Chemical Composition of TC6 titanium alloy (wt %)

Al	Cr	Fe	Mo	Si	C	N	H	O	Other
6.29	1.42	0.42	2.71	0.33	≤0.10	0.05	≤0.015	≤0.1	≤0.1(total≤0.4)

At the beginning of the experiment, JMat Pro software was used in order to calculate the isothermal transformation thermodynamic, where the setting initial and final temperatures were 0°C and 1800°C with temperature interval of 15°C. The phases of TC6 titanium alloy such as-  $\alpha$ -phase,  $\beta$  phase, Ti3Al and liquid phases were observed by calculating the isothermal transformation thermodynamics. For experiment, initially the supplied cylindrical samples were cut into 10 mm height. The heat treatment was performed by a high temperature resistance furnace (SX2-10-13, Zhejiang Sujing Purification Equipment Co., Ltd., Zhejiang, China). Its rated temperature was 1300°C, the heating rate was 200-300°C/h and rated power was 10kW. Before the test, a standard thermocouple and temperature potentiometer was set to ensure that the temperature

deviation within  $\pm 5^\circ\text{C}$ . As titanium was easily oxidized at high temperatures, it was necessary to coat the surface of samples with an anti-oxidant before the heat treatment process. After completing the heat treatment, the samples were cooled by different methods, such as- water, furnace or air cooling. To know the  $\beta$  transformation temperature the samples were heated from 925-1010°C with 20 min holding time, and the heated samples were cooled by water quenching, while later air cooling and furnace cooling were carried out to improve the alloy property in double heat treatment process. Before observing the microstructure of heated samples, it was required to polish those samples. Sandpapers of different grades 150, 400 and 600 were used to polish the samples. After grinding with sandpapers, the samples were polished by an automatic polishing machine by using 3.5  $\mu\text{m}$  particle size diamonds polishing liquid. The samples were polished until the surface became bright and invisible the scratches. After finishing polishing a mixture of chemical HF: HNO<sub>3</sub>: H<sub>2</sub>O = 2: 3: 95 was used for etching of the specimen. Etching time was 10-20s prior to observe the microstructure of the sample after heat treatment. Thus the samples were prepared on a good metallurgical sample for Zeiss optical microscope observation and different microstructures were observed. By observing  $\alpha$  and  $\beta$  percentage of each sample, transformation kinetics was observed and phase transformation behaviour was studied. Image Pro Plus software was used to calculate  $\alpha$  and  $\beta$  volume fraction to describe the kinetics of TC6 titanium alloy. To know the phase transformation behaviour of TC6 titanium alloy heat treatment was carried out below the  $\beta$  phase transformation temperature such as- 750°C, 800°C, 850°C and 900°C with different holding times ranging from 5 to 210 min, where the heated samples were cooled by water quenching. Furthermore, the heat treatment was designed in two stages. At first, samples were heated for 20 min at four different temperatures such as- 960°C, 965°C, 970°C and 975°C, where the cooling process was water quenching. In this stage enough samples were prepared to continue the second stage heat treatment. In second stage, same samples were reheated for 60 min with seven different temperatures such as- 920°C, 930°C, 935°C, 940°C, 945°C, 950°C and 955°C. In the second stage, all reheated samples were furnace cooled and air cooled separately. The flow diagram of heating procedure is given in Fig. 1. In both stages samples were analysed microstructurally in order to know  $\alpha$  and  $\beta$  phase transformation behavior.

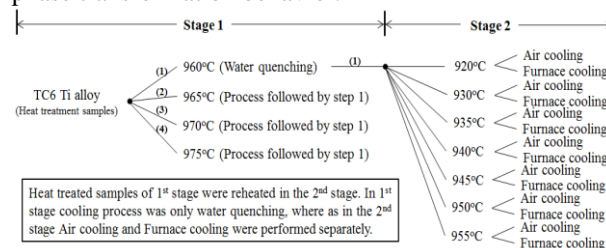


Fig.1 Heat treatment design to observe the microstructure

## 3. RESULTS AND DISCUSSION

### 3.1 Effect of temperature on the properties of

## each phase

JMat Pro thermodynamic software was used to simulate the actual composition of the alloy. The phase diagram was studied in the temperature range of 0°C to 1800°C which is shown in Fig. 2. The phases of TC6 titanium alloy are  $\alpha$ -phase,  $\beta$  phase, Ti3Al and liquid phases. The simulation result shows that the content of  $\alpha$  phase was almost constant below 500°C. Above 500°C  $\alpha$  phase content gradually decreased and it decreased rapidly when the temperature was above 700°C. As the temperature was gradually increased from room temperature to about 500°C, the phase is almost the same as before. The content of  $\beta$  phase above 500°C temperature gradually increased. After 700°C its content increased rapidly. This is due to the following reason: at 500°C;  $\alpha$  phase had opposed insufficient driving force at  $\beta$  phase transition point. In this point, each phase remained relatively stable. As the temperature rose above 500°C,  $\alpha$  phase gradually transforms to  $\beta$  phase and the conversion rate was very slow. When the temperature was above 700°C,  $\alpha$  phase to  $\beta$  phase transition occurs in very fast rate. The simulated alloy phase diagram can characterize the quantitative relationship between heat treatment temperature and the contents between the each phase. When the temperature increased gradually  $\alpha$  phase shifted to  $\beta$  phase. So, from Fig. 2(a) and 2(b), the phase transition temperature lies between 700°C to 1000°C. If the temperature continued to rise, the content of  $\beta$  phase became saturated. The alloy phase was only stable up to 1693°C temperature. The liquid is produced by mixing the alloy to  $\beta$  + liquid two phases. If the temperature continued to rise, at 1721°C all the phases turn to the liquid phase. But it also needs the actual temperature during the heat treatment process to develop the choice of the references. So if it is needed to adjust the alloy content of  $\alpha$  and  $\beta$  phase. Thus the alloy achieves the required performance.

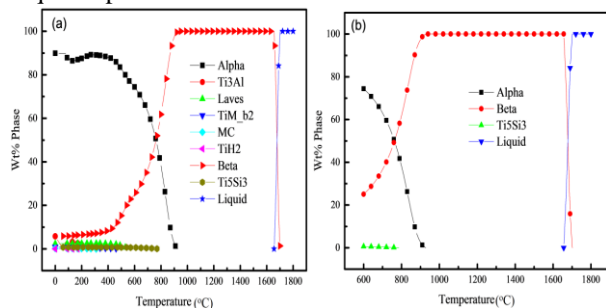


Fig. 2 Equilibrium phase diagram of TC6 Titanium alloy according to (a) mass fraction and (b) atomic weight fraction.

## 3.2. Measurement of $\beta$ transformation temperature point

For determining the phase transition temperature of the alloy, some temperatures around the phase transition point (predicted phase transformation temperature from simulation) were selected to precise the thermal expansion method. By using the optical method, the microstructure of the alloy after isometric heat treatment was measured from 925°C - 1010°C. The interval of the temperatures was 5°C with holding time 20 min and the

cooling process was water quenching. The specific heat treatment system is shown in Table 1.

Table 1: Metallography of  $\beta$  phase transition temperature of the heat treatment parameters

Sample No.	Heating temperature (°C)	Holding time (min)	Cooling process
1	925	20	Water quenching
2	935	"	"
3	945	"	"
4	955	"	"
5	960	"	"
6	970	"	"
7	975	"	"
8	980	"	"
9	985	"	"
10	990	"	"
11	995	"	"
12	1000	"	"
13	1005	"	"
14	1010	"	"

After quenching the reduction of  $\alpha$  phase content in the microstructure was observed. TC6 titanium alloy changed greatly between 980°C-990°C temperature and  $\alpha$  phase disappeared within this temperature range and it was determined that the phase transition temperature was 985°C. Before starting the  $\beta$  phase transition temperature both equiaxed  $\alpha$  phase and transformed  $\beta$  phase were observed, where the heat treated temperatures were 965°C, 970°C and 975°C, as shown in Fig.3. The increased  $\alpha$  phases were observed in lowered heat treated samples. At  $\beta$  transition point temperature rapid water quenching was done, which led to a martensitic transformation of  $\beta$  phase, resulting in a very fine needle like microstructure, as shown in Fig. 3(i). The  $\alpha$  equiaxed and lamella microstructure were observed above the  $\beta$  transition temperature such as- 990°C, 995°C, 1000°C, 1005°C and 1010°C, where the equiaxed microstructures are the result of a recrystallization process and lamellar microstructure was generated upon cooling from  $\beta$  phase field. So as to finalize; the measured phase transition temperature of TC6 titanium alloy was 985°C.

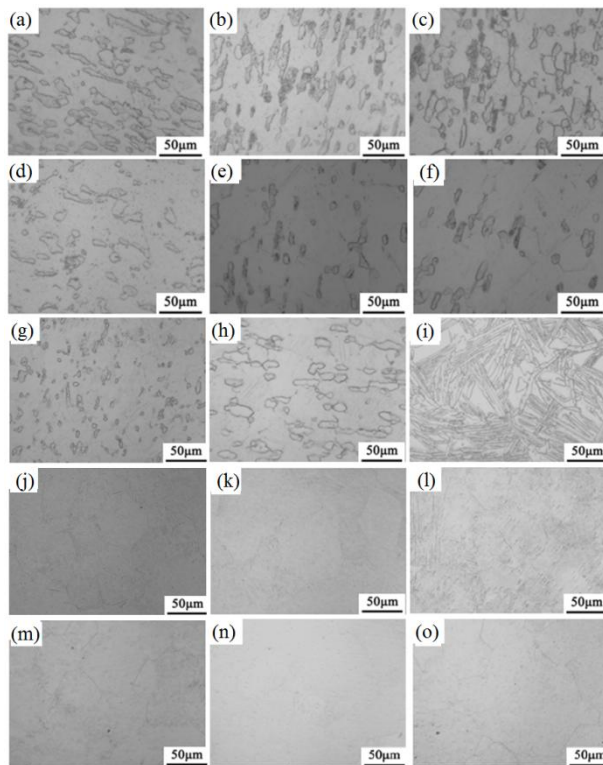


Fig. 3 Microstructure observations after heat treatment at different temperatures- (a) 925°C, (b) 935°C, (c) 945°C, (d) 955°C, (e) 960°C, (f) 965°C, (g) 970°C, (h) 975°C, (i) 980°C, (j) 985°C, (k) 990°C, (l) 995°C, (m) 1000°C, (n) 1005°C and (o) 1010°C

### 3.3 Isothermal phase transformation behavior at different temperatures

The isothermal phase transformation behavior of TC6 titanium alloy was studied microstructurally and were characterized by binding Image-pro Plus software for quantitative characterization. As with increasing temperature gradually all  $\alpha$  phases of TC6 titanium alloy shifted into the beta phase and at 985°C all the alpha phases turned into beta phases, the isothermal phase transformation behaviour was studied at below the  $\beta$  transformation temperatures such as- 750°C, 800°C, 850°C, 900°C and 950°C. For each temperature different holding times, 2-210 min were applied followed by water quenching. The selected holding times contained high and low-temperature flexibility. The basic principle was that the farther away from the phase transition point, the selected interval time was large and total time was longer to ensure that the transformation from  $\alpha$  phase to  $\beta$  phase became steady. After heat treatment process the microstructures were analyzed and the volume fractions of  $\alpha$  and  $\beta$  were calculated using Image-pro Plus software. In order to explain the lower and higher temperature heat treatment effect, 750°C and 900°C will be discussed elaborately.

The simulation results obtained in section 3.1 predicted that the transition rate of  $\alpha$  began to change rapidly at 750°C temperature. The first equilibrium structure was subjected to a heat treatment at different times of incubation after annealing at 750°C temperature. The specific heat treatment system was followed by

different holding times, such as- 5°C, 10°C, 20°C, 30°C, 45°C, 60°C, 90°C, 120°C, 150°C, 180°C and 220°C, where after heat treatment water quenching was performed. As the phase transition point of TC6 was 985°C, 750°C is far away from the phase transition point. So the selected time interval was large. Balance structure after annealing was subjected to a heat treatment at different times of incubation at 750°C.

At 750°C temperature with different incubation times the resulted microstructures of TC6 titanium alloy are shown in Fig. 3. With the extension of the holding time,  $\beta$  phase volume fraction was increased and finally tended to a constant amount. The microstructure of the samples contained  $\alpha$  and  $\beta$  phase. The proportion of phase changed unlikely, but with the naked eye can't be seen the microstructure clearly and the fraction of the phases significantly changed in the respective adjacent point in time.

After incubation at 750°C temperature at different times, the volume fraction of  $\alpha$  phase was calculated by using Image-pro Plus software for quantitative characterization, which is shown in Fig. 5(a). Fig. 5(a) represents the changes of  $\alpha$  volume fraction with different holding time, where with increasing the holding time,  $\alpha$  volume fraction decreased, but the slope of the curve was small because of slow decreasing rate with time. After incubation of 180 min and longer, the volume fraction of  $\alpha$  phase did not change so much but had a stable value. According to this trend it could be judged, in this alloy the phase transition at 750°C incubated balance time was 180 min. The volume fraction of primary  $\alpha$  phase microstructure was calculated by using Image-pro Plus software. The change of the  $\alpha$  volume fraction with the holding time was decreased when the time was increased from 5 min to 150 min and further increasing the time to 210 min,  $\alpha$  volume fraction remained in stable value which is shown in Fig. 5(a).

The non-homogeneous kinetic theory of phase transitions, when the external conditions (temperature or pressure) changes, the system will be in a metastable state and the metastable phase has a tendency to be transformed into one or a few relatively stable new phase. In  $\alpha + \beta$  phase type TC6 titanium alloy, with the holding time and a certain degree of super cooling,  $\alpha$  phase gradually changed to  $\beta$  phase. The contents with the non-homogeneous phase gradually increased to  $\beta$  phase until the transition ends in an equilibrium state. The relationship between the volume fractions of  $\beta$  phase with the respective holding time is drawn. Thus it was obtained non-uniform alloy phase transformation kinetics at the same temperature, namely "S-Curve".

The non-uniform phase transformation kinetics of TC6 titanium alloy at 750°C is shown in Fig. 5(b). The

amount of  $\beta$  phase transition was  $f = \frac{x_t - x_0}{x_{eq} - x_0}$ , where  $x_t$

was at any one time for the incubation,  $\beta$  is the phase volume fraction at the different holding temperature,  $x_{eq}$  was the lower temperature of incubation, when thermodynamic equilibrium of phase transition of  $\beta$  phase; the volume fraction of the phase (maximum volume),  $x_0$  was the beginning point for heating

temperature to achieve the thermodynamic equilibrium of  $\beta$  phase. At 750°C, with the holding time,  $\beta$  volume fraction increased gradually, but turned into the smaller slope of the curve, which showed a slow increase with time. After 150 min incubation time and longer, the volume fraction of beta phase inversion didn't increase rapidly, but stayed stable value. According to this trend could be judged, in the alloy phase transition at 750°C incubated balance time was 150 min.

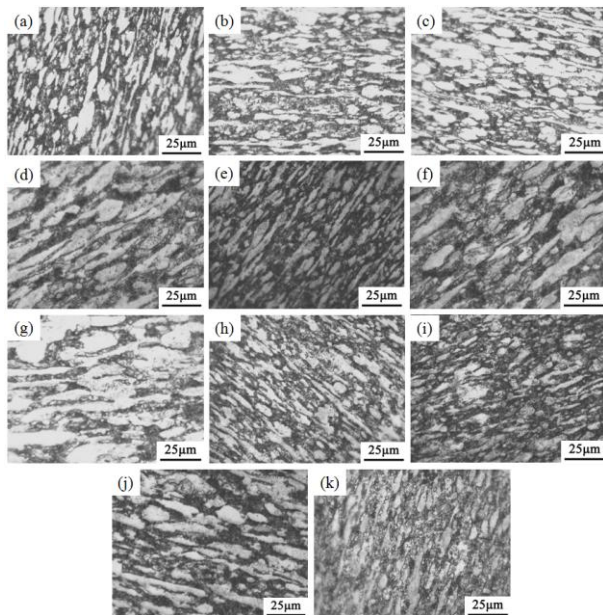


Fig. 4 Microstructural observation at 750°C temperature at different holding times- (a) 5 min, (b) 10min, (c) 20 min, (d) 30 min, (e) 45 min, (f) 60 min, (g) 90 min, (h) 120 min, (i) 150 min, (j) 180 min, (k) 210 min.

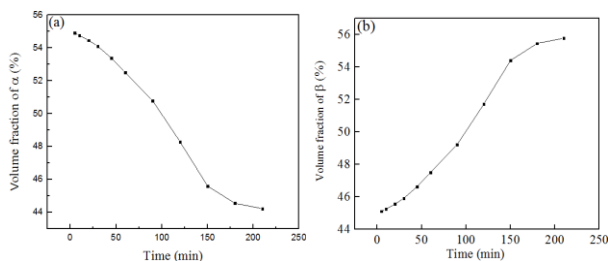


Fig. 5 Volume fraction of  $\alpha$  and  $\beta$  after heat treatment at 750°C temperature- (a)  $\alpha$  phase and (b)  $\beta$  phase.

Similarly isothermal phase transformation behavior was studied for 800°C, 850°C, 900°C and 950°C. Interestingly, with increasing the incubation temperature, the conversion rate of  $\alpha$  phase to  $\beta$  phase is rapid enough. For these higher temperatures  $\alpha$  phase converted to  $\beta$  phase in lower holding time and became steady. Significant phase transition happened in 150 min, 120 min, 90 min and 50 min for 800°C, 850°C, 900°C and 950°C respectively. In order to compare the phase transformation behavior of higher heat treated samples with lower heat treated samples, the details phase transformation of  $\alpha$  phase at 950°C will be discussed here. At 950°C temperature the microstructure of TC6 titanium alloy after different incubation time is shown in Fig. 6. It can be seen from the photomicrograph under various heat treatment times, with the holding time, where it is seen that  $\alpha$  volume fraction reduced and the rate of declination was very fast.

The  $\beta$  volume fraction is gradually increased. By comparing the effect of incubation temperature at 950°C with previous 750°C, 800°C, 850°C, 900°C,  $\alpha$  volume fraction showed the fastest decline, as can be seen from the photomicrograph images of Fig. 4 and Fig. 6. From Fig. 6 it is shown that, microstructure changes are also clearly visible for lower holding temperature. With the holding time, the microstructure of  $\alpha$  volume fraction decreased significantly for 20 min holding time as shown in Fig. 7(a). The content of  $\alpha$  phase become relatively small. When it was incubated at 50 min by applying heat, the microstructure had no longer  $\alpha$  phase. Until the holding time reached to 50 min, the microstructure of  $\alpha$  volume fraction was decreased and became steady after 50 min holding time. The relationship between the amount of  $\beta$  phase transformation of the TC6 titanium alloy at 950°C temperature and incubation time is shown in Fig. 6(b), which was obtained by using Image-pro Plus software. In conclusion, with higher incubation temperature, the rate of change of transformation ( $\alpha$  phase to  $\beta$  phase) is high enough and in lower holding time the phase change became saturated.

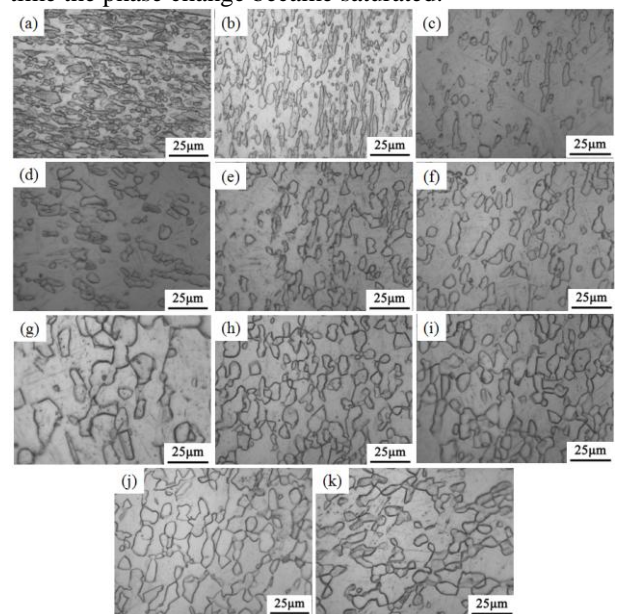


Fig. 6 Microstructural observation at 950°C temperature at different holding times- (a) 5 min, (b) 10 min, (c) 20 min, (d) 30 min, (e) 45 min, (f) 60 min, (g) 90 min, (h) 120 min, (i) 150 min, (j) 180 min, (k) 210 min.

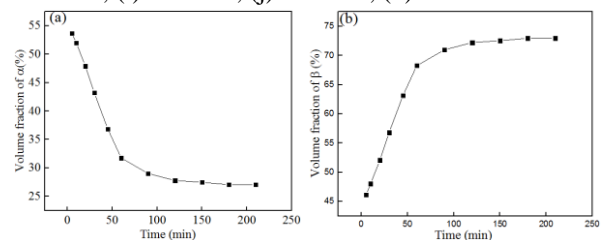


Fig. 7 Volume fraction of  $\alpha$  and  $\beta$  after heat treatment at 950°C temperature- (a)  $\alpha$  phase and (b)  $\beta$  phase.

### 3.4 Isothermal transformation kinetics model

The non-uniform temperature equal  $f-t$  the kinetic curves obtained at different temperatures replaces the coordinates drawn about coordinate system where the

S-curve plotted in change  $\lg \ln\left(\frac{1}{1-f}\right) - \lg t$  coordinates.

So to transform it into a straight line, linear fitting was done to slope of the line and intercept. The alloy obtained at different temperature for non-uniform temperature changing equal kinetic equation-

$$\lg \ln\left(\frac{1}{1-f}\right) = \lg k - n \lg t \quad (1)$$

Rewrite it as an exponent, known Avrami equation

$$f = 1 - \exp(-kt^n) \quad (2)$$

Where,

$f$  - The amount of change

$t$  - Time

$k$  - Rate constant

$n$  - Avrami exponent.

Avrami equation is theoretically applicable to non-uniform temperature change which was used to describe the phase transition kinetics equation quantitatively. When the value of  $k$  was larger the reaction rate was faster. At 750°C, the fitting of  $\lg k = -3.0485$ ,  $n = 1.65653$ , linear fit  $R^2 = 0.99643$ . Then at 750°C temperature  $n$  and  $k$  values were substituted into the Avrami's equation  $f = 1 - \exp(-kt^n)$  and following equation was obtained.

$$f = 1 - \exp(-3.0485 \times 10^{-4} t^{1.65653}) \quad (3)$$

Similarly, at 800°C, 850°C, 900°C, 950°C experimental and calculated values of kinetic equations are

$$f = 1 - \exp(-3.0404 \times 10^{-4} t^{1.79022}) \quad (4)$$

$$f = 1 - \exp(-2.9574 \times 10^{-4} t^{1.90616}) \quad (5)$$

$$f = 1 - \exp(-2.9087 \times 10^{-4} t^{2.1122}) \quad (6)$$

$$f = 1 - \exp(-2.8022 \times 10^{-4} t^{2.23909}) \quad (7)$$

For each temperature  $k$  value,  $n$  value and fitted a straight line for a comprehensive comparison was provided in table 2. With the heat treatment temperature, the rate of constant  $k$  value gradually increased, which also showed that with increasing temperature the rate of increase in the phase transition occurred. The increase rate of the constant  $k$  was not dependent on the increment rate of temperature. If the temperature was higher, the increase rate was greater. In 800°C, 850°C 900°C, 950°C under the rate constant  $k$  value increased respectively compared with an increase of temperature 750°C. The respective values were 1.93%, 23.34%, 37.95%, and 76.31%.

The values of  $\lg k$ ,  $k$ ,  $n$  of TC6 titanium alloy at each incubation temperature Avrami exponent  $n$  were about 2. The size of the  $n$ -value and phase transformation mechanisms relating to nuclear, ideally, homogeneous nucleation Avrami exponent  $n = 4$ . The value of  $n$  was more close to 4. The nucleation mechanism was closer to homogeneous nucleation. The value of  $n$  formed at different temperatures was seen with isothermal temperature. When the value of  $n$  increased that was the

lower the temperature, the greater the proportion of heterogeneous nucleation.

The alloy at 750°C, 800°C, 850°C, 900°C, 950°C temperature  $\alpha$  volume fraction versus time the quantitative relationship was drawn to the same Fig. 8. At each isothermal temperature, with the holding time,  $\alpha$ -phase volume fractions were first gradually decreased until a certain incubation time, the volume fractions tend to indicate value. A comprehensive comparison of various incubation temperatures at different holding time affected the relative volume fraction of  $\alpha$  of the alloy. It was seen that, the higher the isothermal temperature, the faster the phase transition, the shorter the time to reach the desired balance

Table 2:  $R^2$  according to temperature.

Temperature (°C)	$\lg k$	$k \times (10^{-4})$	N	$R^2$
750	-3.0485	8.9433	1.6565	0.99643
800	-3.0404	9.1116	1.7902	0.99645
850	-2.9574	11.0301	1.9061	0.99319
900	-2.9087	12.3375	2.1122	0.99758
950	-2.8022	15.7683	2.2390	0.99818

When the phase transition temperature reached equilibrium for 750°C, 800°C, 850°C, 900°C, 950°C under the insulation, the phase transition rate was slow with the holding time. At first the volume fraction of  $\alpha$  phase decreased and the decreasing rate was slow. After 2 hour the phase change balance, proportion relative to microstructure was not predictable. When the change occurred at this temperature of the TC6 titanium alloy the lower phase takes a longer time to change it. It was not conducive to energy conservation. Above 900°C temperature the volume fraction of  $\alpha$  phase to  $\beta$  phases inversion faster. The time required to reach the equilibrium shorter for 950°C. The above-mentioned five temperatures was the ideal heat treatment temperature

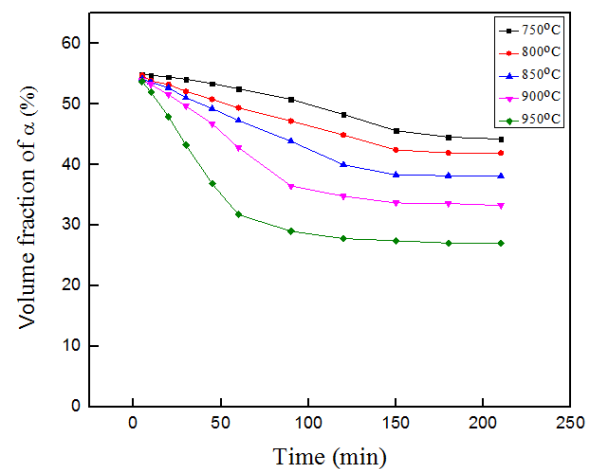


Fig. 8 Effect of incubation time on  $\alpha$  volume fraction at different temperatures.

It was found that for TC6 titanium alloy components the actual development of the heat treatment process is preferred above 950°C temperature. Heat treatment temperature and holding time was not less than

20 min, because, in this condition, the phase transition grew faster in the equilibrium phase transition and by holding time the control phase of alloy may form to gain the desired properties.

By the  $\alpha + \beta / \beta$  transformation point by applying different heating temperature, holding at different times, the changing in  $\beta$  phase volume fraction was determined at 750°C, 800°C, 850°C, 900°C, 950°C temperature changed the start time and the end time. The volume fraction of  $\beta$  phase transition in this research was selected as 1%. The  $\beta$  phase volume fraction is shown in Fig. 9 according to different holding times.

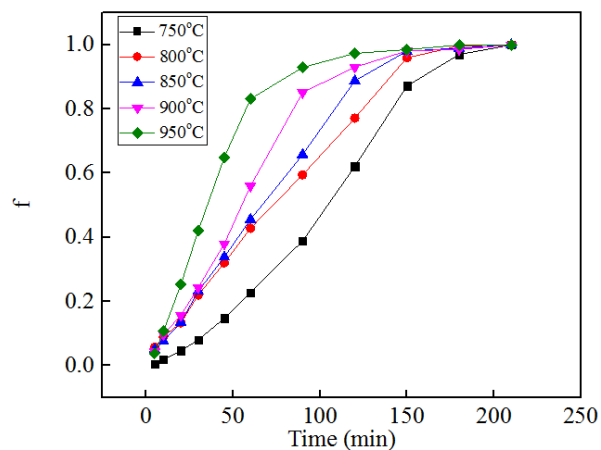


Fig. 8 Heterogeneous transition temperatures of each kinetic curve.

In order to clearly see the equal time which was carried out for  $\beta$  transformation temperature, at every temperature the transition starting time and ending time was plotted against time at different temperatures, which were shown in Fig. 8. The starting of transition point and end point were connected together to form a line which represents the isothermal transformation diagram of TC6 Ti alloy at quarter time, half time and total time which are shown in Fig. 9 (a), (b) and (c).

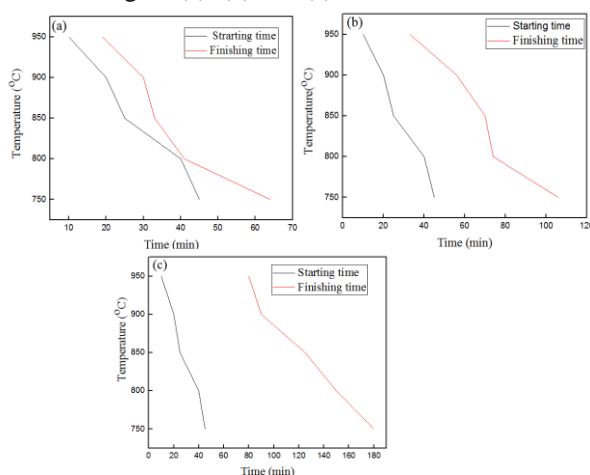


Fig. 9 TTT diagram of TC6 titanium alloy- (a) Quarter time, (b) Half time and (c) Entire time

### 3.5 Double heat treatment process

To improve the mechanical property of TC6 titanium alloy two stage heat treatment plays an important role to improve the mechanical or physical

property of the work material. For this heat treatment material geometry was not changed. Heat treatment involves various heating and cooling procedures performed to affect the micro-structural changes of a material. That's why it affects the mechanical property of the work part. From isothermal transformation kinetics and analysis of thermodynamics based on the heat treatment process design; the choice of the heat treatment temperature was not to be less than 950°C. The selected holding time was 20 min. On this basis, for the original alloy single heat treatment and double heat treatment process; the design cycle was selected not only to find the process parameters on the structural evolution of the law but also to get the influence of the heat treatment on the properties of the alloy and the corresponding optimization process, enabling technology, microstructure, performance and integration. The 1<sup>st</sup> stage of heat treatment controlled the shape of alpha phase and 2<sup>nd</sup> stage heat treatment controlled both the shape of primary alpha, secondary alpha and beta phase. The samples from the 1<sup>st</sup> stage were incubated again for 60 minutes and cooled by air and furnace respectively. The schematic of two stage heat treatment was shown in Fig. 1. After the heat treatment, the samples were metallographically prepared and microstructures were observed using optical microscopy.

At the beginning of two stage heat treatment process at first the samples were heated at 960°C temperature, where the holding time was 20 min and cooling process was water quenching. Then at the 2<sup>nd</sup> stage, the same samples were incubated again at 920°C, 930°C, 935°C, 940°C, 945°C, 950°C and 955°C temperatures respectively for 60 min and those heated samples were cooled by air and in the furnace. Fig. 10(a), (c), (e), (g), (i), (k), (m) represents the optical images of 2<sup>nd</sup> stage heat treated samples air for cooling. It can be said that the content of  $\alpha$  (both primary alpha and secondary alpha) was high when the applied temperature was 920°C and the amount of  $\beta$  content was lower than the  $\alpha$  phase. The amount of primary  $\alpha$  was also higher than the secondary  $\alpha$  phase. When the heating temperature was increasing gradually from 920°C to a higher temperature the content of  $\alpha$  phase also decreased and the amount of  $\beta$  phase content was increased with respect to temperature. From the above Fig. 11(a) it can be said that the volume fraction of  $\alpha$  phase content decreased while the temperature increased gradually. At lower temperature, the amount of  $\alpha$  phase was higher and with increasing temperature, at a certain stage it showed the equilibrium state. That means the changing rate was very slowly. From this graph it can be easily understood when the temperature was low; at 920°C, it decreased very quickly, but the decreasing rate was slow as the temperature increased gradually from 920°C to 950°C temperature. As the temperature increased again at higher stage 950°C to 955°C it showed the equilibrium curve. After plotting

volume fraction of  $\alpha$  phase with respect temperature it is appeared as “S-shape”. From Fig 11(b), it can be said that the volume fraction of  $\beta$  phase content increased while the temperature increased gradually. At lower temperature, the amount of  $\beta$  phase was lower and with increasing temperature at a certain stage it showed the equilibrium state. That means the changing rate was quick enough at higher temperature. At 920°C the amount of  $\beta$  phase was very little. But when the temperature increased to 930°C, it started to increase very quickly. But the increasing rate was fast as the temperature increased gradually from 920°C to 950°C temperature. As the temperature increased again at higher stage 950°C to 955°C it showed the equilibrium curve. After plotting volume fraction of  $\beta$  phase with respect temperature it was appeared as “S-shape” also.

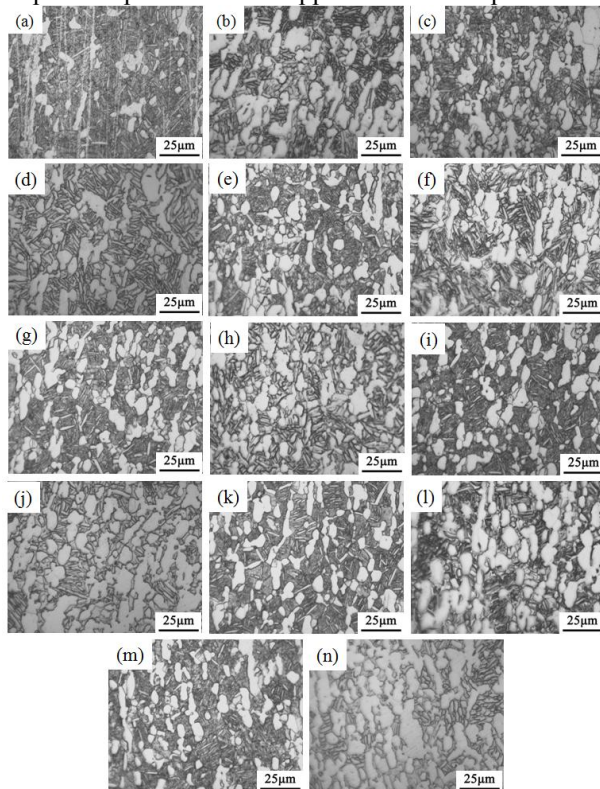


Fig. 10 Microstructures after 2<sup>nd</sup> stage heat treatment at 920°C 930°C 935°C 940°C 945°C 950°C 955°C temperature (a), (c), (e), (g), (i), (k), (m) by air cooling; and (b), (d), (f), (h), (j), (l), (n) by furnace cooling.

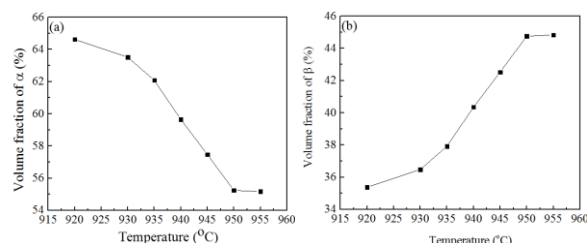


Fig. 11 Volume fraction of  $\alpha$  and  $\beta$  after air cooling (a)  $\alpha$  phase and (b)  $\beta$  phase.

Fig. 10 (b), (d), (f), (h), (j), (l), (n) represent the microstructures of furnace cooling specimens. It can be said that the content of  $\alpha$  (both primary alpha and secondary alpha) was high when the applied temperature

was 920°C and the amount of  $\beta$  content was lower than the  $\alpha$  phase. The amount of primary  $\alpha$  was also higher than the secondary  $\alpha$  phase. When the heating temperature was increasing gradually from 920°C to a higher temperature the content of  $\alpha$  phase also decreased with respect to time and the amount of  $\beta$  phase content was increasing with respect to temperature. But it is hard to calculate the exact percentage of  $\alpha$  and  $\beta$ .

Fig. 12(a) showed the volume fraction of  $\alpha$  phase content, which was obtained by Image-pro Plus software. The  $\alpha$  volume fraction was decreased while the temperature increased gradually. At lower temperature the amount of  $\alpha$  phase was high and with increasing temperature, at a certain stage it became equilibrium state. That means the rate of changing was very slow. From this figure it was easily understood when the temperature was low; at 920°C, it decreased very quickly, but the decreasing rate was slow as the temperature increased gradually from 920°C to 950°C temperature. As the temperature increased again at higher stage 950°C to 955°C it showed the equilibrium curve. after plotting volume fraction of  $\alpha$  phase with respect to temperature it was appeared as “S-shape”.

From Fig. 12(b) it can be said that the volume fraction of  $\beta$  phase content increased while the temperature increased gradually.  $\beta$  phase amount was low at low temperature and with increasing temperature, it showed the equilibrium state at a certain stage. When the temperature was low; i.e. at 920°C the amount of  $\beta$  phase was very little. But when the temperature increased to 940°C, it started to increase very quickly and the increasing rate was fast as the temperature reached to 950°C temperature. As the temperature increased again at higher stage 950°C to 955°C it showed the equilibrium curve. after plotting volume fraction of  $\beta$  phase with respect to temperature, it also appeared as “S-shape”. Similarly for 965°C and 970°C, in the 2<sup>nd</sup> stage heat treatment the phase transformation is little higher. The  $\alpha$  phase converted into  $\beta$  phase earlier than previous. Moreover, at higher temperature the equilibrium state comes earlier than before.

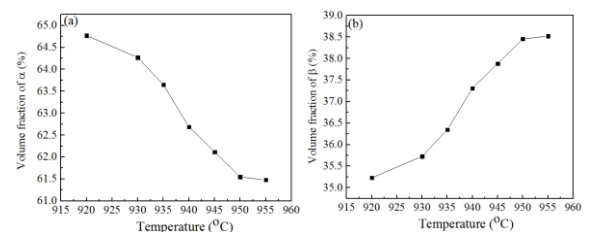


Fig. 12 Volume fraction of  $\alpha$  and  $\beta$  after furnace cooling (a)  $\alpha$  phase and (b)  $\beta$  phase.

In order to compare the trend of phase transformation of  $\alpha$  and  $\beta$  between lower temperature and higher temperature (1<sup>st</sup> stage heat treatment), at first TC6 samples were heated at 975°C and followed the 2<sup>nd</sup> stage heat treatment process, where the cooling processes are air cooling and furnace cooling. In the 2<sup>nd</sup> stage samples were incubated again at 920 °C, 930 °C, 935 °C, 940 °C, 945 °C, 950 °C and 955 °C temperatures respectively 60min and these were cooled by air and in the furnace. Fig. 13(a), (c), (e), (g), (i), (k) and (m) represent the air cooling specimens after 2<sup>nd</sup> stage heat treatment. The



content of  $\alpha$  (both primary alpha and secondary alpha) was high when the applied temperature was 920°C and the amount of  $\beta$  content was lower than the  $\alpha$  phase. The amount of primary  $\alpha$  was also higher than the secondary  $\alpha$  phase. When the heating temperature was increasing gradually from 920°C to a higher temperature the content of  $\alpha$  phase also decreased with respect to time and the amount of  $\beta$  phase content was increasing with respect to temperature. The volume fraction of  $\alpha$  phase content and  $\beta$  phase content followed the same phenomena as stated before, but the starting point of phase transformation occurred little earlier than before, as shown in Fig. 14(a) and (b). Moreover, the transformation process of  $\alpha$  phase into  $\beta$  phase completed earlier than before. Fig. 15(a) and (b) represents the change of volume fraction of  $\alpha$  phase and  $\beta$  phase respectively for furnace cooling. The  $\alpha$  content decreased while the temperature increased gradually, as shown in Fig. 15(a). At lower temperature, the amount of  $\alpha$  phase was higher and with increasing temperature, at a certain stage, it showed the equilibrium state. That means the changing rate was slow. Fig. 15(b) showed the change of volume fraction of  $\beta$  phase with increasing temperature. At lower temperature, the amount of  $\beta$  phase was lower and with increasing temperature at a certain stage it showed the equilibrium state. That means the changing rate was very quick. When the temperature was lower; at 920°C the amount of this phase was very little. But when the temperature increased to 930°C, it started to increase very quickly. But the increasing rate was fast as the temperature increased gradually from 920°C to 950°C temperature. As the temperature increased again at higher stage 945°C to 955°C it showed the equilibrium curve. After plotting volume fraction of  $\beta$  phase with respect to temperature it showed “S-shape” curve.

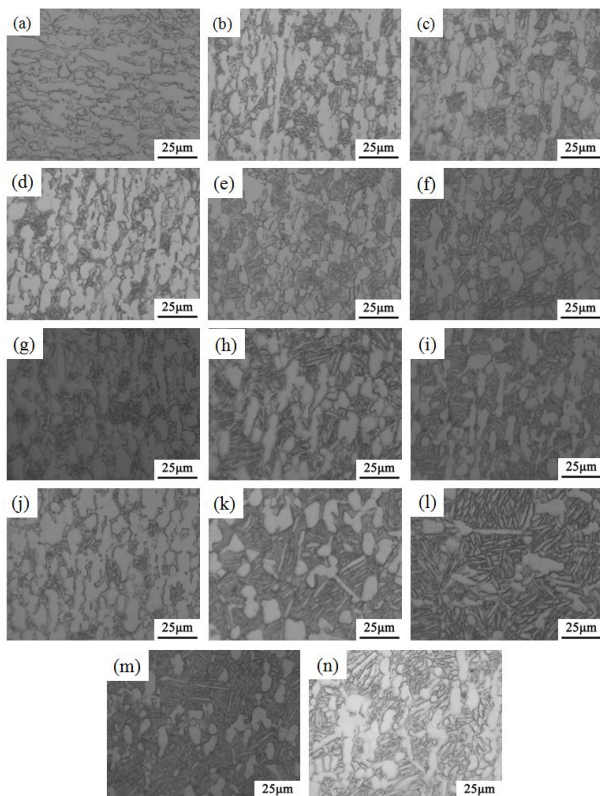


Fig. 13 Microstructures after 2<sup>nd</sup> stage heat treatment at (a) 920°C, (c) 930°C, (e) 935°C, (g) 940°C, (i) 945°C, (k) 950°C, (m) 955°C temperatures by air cooling; (b) 920°C, (d) 930°C, (f) 935°C, (h) 940°C, (j) 945°C, (l) 950°C, (n) 955°C temperatures by furnace cooling.

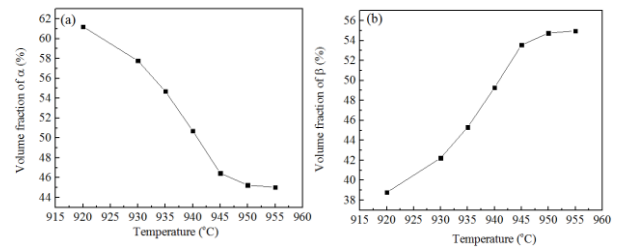


Fig. 14 Volume fraction of  $\alpha$  and  $\beta$  after air cooling (a)  $\alpha$  phase and (b)  $\beta$  phase.

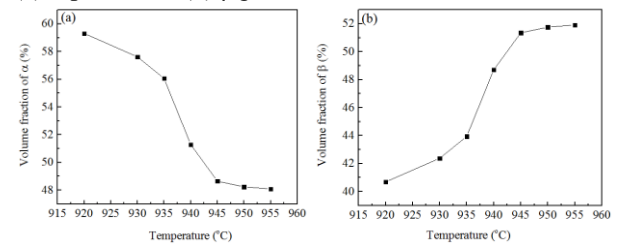


Fig. 15 Volume fraction of  $\alpha$  and  $\beta$  after air cooling (a)  $\alpha$  phase and (b)  $\beta$  phase.

After finishing first step heat treatment where the applying temperatures were 960°C, 965°C, 970°C, 975°C and cooling procedure was water quenching; the samples are reheated at 920°C and followed by the air and furnace cooling process.

#### 4. CONCLUSION

In this research the isothermal phase transformation behavior of TC6 titanium alloy was studied. According to microstructural observation, the  $\beta$  phase transition temperature was 985°C, which is similar to predicted simulation value. The phase transformation rate from  $\alpha$  to  $\beta$  phase was faster at 750°C temperature, which provided a reference for the development of the heat treatment process. The phase transformation behavior was studied below the  $\beta$  transition temperature. The holding temperature affects the phase transformation behavior. When the holding temperature is less, the transformation of  $\alpha$  phase to  $\beta$  phase is very slow. In contrast, the phase transformation took place earlier at higher holding temperatures and the volume fraction of  $\alpha$  and  $\beta$  became steady at higher temperatures. Isothermal transformation kinetics model was studied at below the  $\beta$  transformation temperature following Avrami equation. The considered cooling process for isothermal phase transformation and kinetics study was water quenching. Finally, two stage heat treatment processes was considered, where the samples were initially heated at certain temperature followed by water quenching and in the 2<sup>nd</sup> stage those heated samples were again reheated at different temperatures followed by air cooling and furnace cooling separately. For 2<sup>nd</sup> stage cooling processes for both air cooling and furnace cooling system when the temperature was increased, the primary  $\alpha$

phase (equiaxed  $\alpha$  phase) reduced and the secondary  $\alpha$  phase (lamellar  $\alpha$  phase) increased with increasing temperature. The  $\beta$  transformation rate also increased with increasing the temperature. In the two stage heat treatment process the transformation rate from equiaxed  $\alpha$  phase to lamellar  $\alpha$  phase was quick in air cooling process than furnace cooling. The two stage heat treatment process was studied in order to check the microstructural change for the development of mechanical properties of TC6 titanium alloy.

## 5. ACKNOWLEDGEMENT

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