GRAIN GROWTH KINETICS IN BETA PHASE OF Ti-6Al-4V ALLOY.

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Abstract

The grain growth kinetics of a ß Ti-6Al-4V alloy has been determined by measuring different grain size parameters (perimeter, area, minimum and maximum diameter) as well as the ratio of the grain boundary area per unit volume for different heat treatment temperatures and times. The growth order and activation energy have been also evaluated.

Introduction

It is well established that the driving force for grain growth is the reduction in the energy associated with the decrease in grain boundary area , and it has been proposed that the rate of boundary migration is inversely proportional to the boundary radius of curvature (1). Grain growth takes place due to diffusion when thermal energy is given to the material. This means that the ratio number of grains/unit volume decreases, the size of the grains increases, and both the ratio of grain boundary area/unit volume, and the ratio of stored energy/unit volume, decrease. Consequently a state of higher thermodynamic stability is reached.

This grain growth kinetics at constant temperature and for normal grain growth of single phase metals follows the Arrhenius equation (2-4):

$$D - D_0 = Kt^n \qquad [1]$$

where D is the size of the grain at a certain time, D_0 is the initial grain size, t is the heat treatment time, and K and n are constants which depend on the metal composition and the temperature, but are independent of grain size.

Moreover, if atomic diffusion across the grain boundary is a simple activated process, it can then be shown that K can be written as (2-3):

$$K = K_0 \exp(-E_a/RT) \qquad [2]$$

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where E_a is the activation energy for the process, T is the temperature in Kelvin and R is the universal gas constant. Therefore, the law of grain growth can be written in terms of both temperature and time:

$$D - D_0 = K_0 \exp(-E_a/RT)t^n \qquad [3]$$

In the present work, the experimental investigation of the grain growth kinetics at different temperatures and times of heat treatment for Ti-6Al-4V alloy in ß phase has been carried out . One of the reasons, usually stated, for avoiding annealing treatments and hot working of the Ti-6Al-4V alloy at temperatures over ß transus is the large grain growth exhibited at such temperatures. The aim of the present work is to quantify the grain growth kinetics when the alloy is heat treated in its ß phase.

Experimental Procedure

Grain growth kinetics have been studied in a Ti-6Al-4V alloy kindly donated by Industrias Quirúrgicas de Levante S.A., which came in cylindrical rods of 13 mm in diameter and 11 cm in length. Discs 6 mm high were cut and 30 specimens were prepared. The chemical composition of this alloys is shown in Table I. The microstructure of the as received material corresponded to the "mill annealed", obtained by forging at 950°C, annealing at 700°C for 2 hours and cooling in air. Fig.1 shows a tranverse cut of the rod. Both the chemical composition and microstructure are in good agreement with the ASTM F136-84 standard (5) for wrought Ti-6Al-4V for surgical applications.

Table I. Chemical composition of the Ti-6Al-4V ELI.

Al	V	Fe	C	O_2	N_2	H_2	Ti
6.1	4.0	0.11	0.021	0.09	0.010	0.0012	Balance

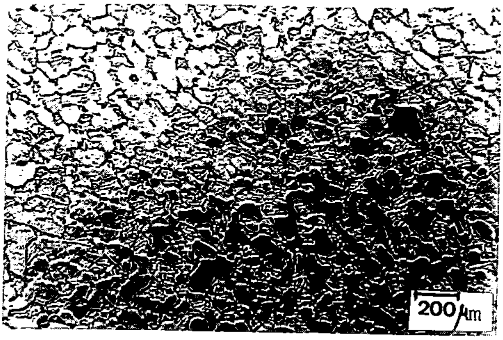

Fig.1. "Mill-annealed" microstructure corresponding to a transverse cut of the rod.

Two specimens were used as reference samples, whilst the others 28 were subjected to different heat treatments above the ß transus temperature of the material. The temperatures used were 1050, 1100, 1150 and 1200°C and the heat treatment times were 5, 10, 15, 20, 30, 60 and 120 minutes at each temperature.

A set of specimens was placed in a tubular furnace with argon atmosphere at a fixed temperature for each experiment and then removed from the furnace according to the time sequence and rapidly quenched in water at 20°C.

The specimens were then metallographically polished and etched with a mixture of HNO_3 and HF acids diluted in water. Metallographic observation was carried out and grain size paramenters (perimeter, area, minimum and maximum diameter) were obtained by Image Analysis with a MIP (Microm Image Processing) equipment. The samples were studied transversally and longitudinally. Fig.2 shows the microstructure of a sample heat treated at 1050°C for 5 minutes.

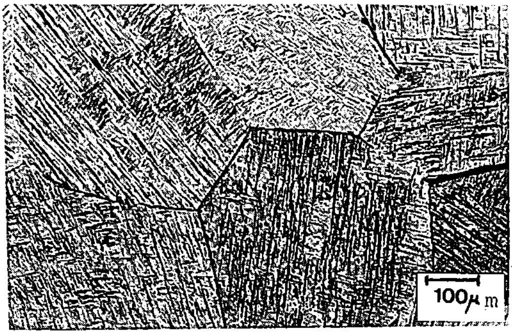

Fig.2. Martensitic microstructure of a sample heat treated at 1050°C during 5 min.

Experimental Results

The grain size values obtained (perimeter, area, minimum and maximum diameter) are shown in relation to heat treatment time and at each temperature in Figs. 3, 4, 5 and 6 for each growth parameter. The grain size increases as both the heat treatment temperature and the heat treatment time increase. Taking into account that the initial average grain size of the alloy has an area of 24000 μm^2, the final average grain size after 120 minutes of heat treatment increases by a factor of 19 at 1050°C, by a factor of 31 at 1100°C, by a factor of 45 at 1150°C and by a factor of 58 at 1200°C.

These graphs show that rapid grain growth occurs during the first 15 to 20 minutes of heat treatment, but subsequently the grain growth rate decreases.

The grain growth kinetics of the alloy ß Ti-6Al-4V follows the Hillert distribution (6), since the maximum radius is smaller than 1.8 times the value of the average radius. This means that growth takes place in a uniform way in the whole material and that the distribution of sizes obeys an asymptotic law, typical of equilibrium states. Such kinetic behaviour agrees with equation [1], since after taking logarithms, linear equations are obtained with correlation coefficients greater than 0.97. The slope of such straight lines is the growth order n, shown in Table II.

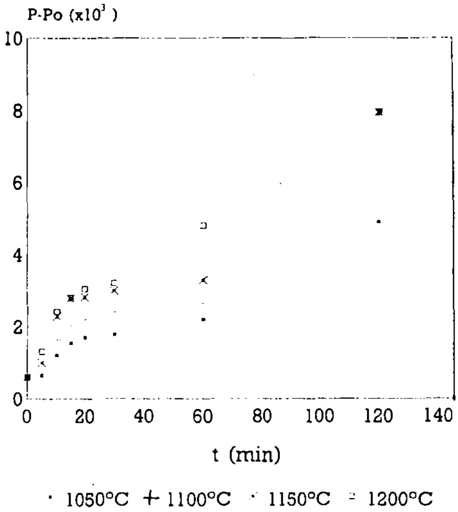

Fig.3. Perimeter (μm) in relation to temperatures and times of heat treatment (min).

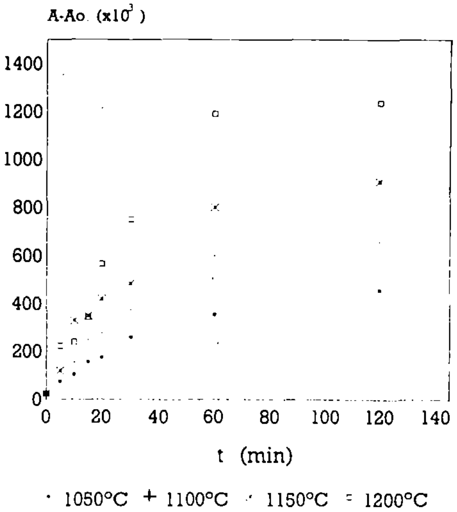

Fig.4. Area (μm^2) in relation to temperatures and times of heat treatment (min).

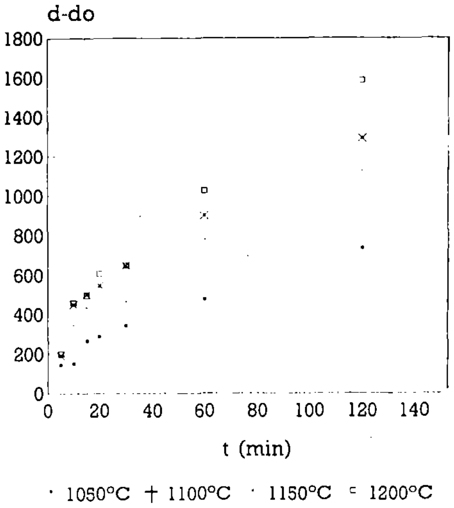

Fig.5. Minimum diameter (μm) in relation to temperatures and times of heat treatment (min).

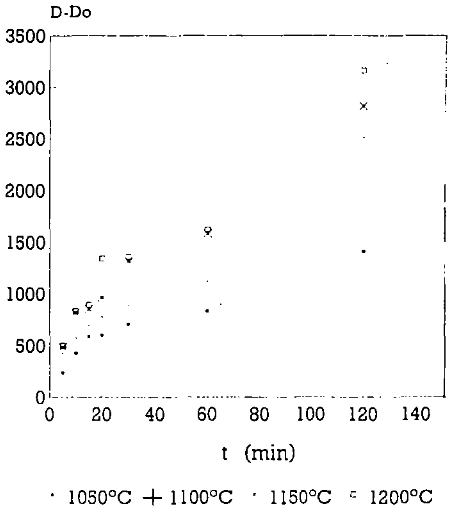

Fig.6. Maximum diameter (μm) in relation to temperatures and times of heat treatment (min).

The activation energy for grain growth, using the different growth parameters, has been calculated from equation [2]. Table III shows the corresponding average values for the different heat treatment times. Such results have been obtained by taking logarithms to equation [3], considering the growth order constant with temperature for growth parameter. The activation energies range between 91 KJ/mol for the maximum diameter and 98 KJ/mol for the perimeter. The fact that the values are all very similar show that grain growth can be described by a simple activation law.

Table II. Growth order and different heat treatment temperatures for the measured parameters. (r is the lineal correlation coefficient).

T(°C)	Equation	Growth exponent
Perimeter		
1050	$P-P_0 = 2.49t^{0.55}$	0.55 (r=0.99)
1100	$P-P_0 = 2.57t^{0.55}$	0.55 (r=0.97)
1150	$P-P_0 = 2.75t^{0.52}$	0.52 (r=0.98)
1200	$P-P_0 = 2.84t^{0.49}$	0.49 (r=0.98)
Area		
1050	$A-A_0 = 4.46t^{0.59}$	0.59 (r=0.99)
1100	$A-A_0 = 4.67t^{0.59}$	0.59 (r=0.99)
1150	$A-A_0 = 4.75t^{0.63}$	0.63 (r=0.97)
1200	$A-A_0 = 4.82t^{0.65}$	0.65 (r=0.97)
Minimum diameter		
1050	$d-d_0 = 1.75t^{0.53}$	0.53 (r=0.98)
1100	$d-d_0 = 1.97t^{0.52}$	0.52 (r=0.98)
1150	$d-d_0 = 2.02t^{0.54}$	0.54 (r=0.97)
1200	$d-d_0 = 1.97t^{0.59}$	0.59 (r=0.99)
Maximum diameter		
1050	$D-D_0 = 2.11t^{0.50}$	0.50 (r=0.98)
1100	$D-D_0 = 2.20t^{0.55}$	0.55 (r=0.98)
1150	$D-D_0 = 2.33t^{0.53}$	0.53 (r=0.99)
1200	$D-D_0 = 2.28t^{0.60}$	0.60 (r=0.99)

Table III. Activation energies for the different grain size parameters.

Parameter	Equation	Activation energies (KJ/mol)
Perimeter	$K= 10^{7.00}\exp(-98/RT)$	98
Area	$K=10^{10.01}\exp(-97/RT)$	97
Minimum diameter	$K= 10^{6.04}\exp(-94/RT)$	94
Maximum diameter	$K= 10^{6.24}\exp(-91/RT)$	91

The ratio of the grain boundary area per unit volume (Gv) has been determined at each temperature and for each heat treatment time. Assuming that the grains have the ideal shape of a tetrakai-decahedron, the value of Gv can be determined from the mean grain boundary area on a random section from (7):

$$Gv = 3.059/A^{1/2} \qquad [4]$$

Fig.7 shows a steep decrease of Gv with time up to 15 minutes. For longer times a limiting grain size is asymptotically approached.

When the samples were heat treated at higher temperatures (1300°C) or during longer times (more of 120 minutes at 1200°C), abnormal grain growth was observed, as shown in Fig.8. This is a common effect in other metals and alloys. Burke (8) suggested that when a secondary grain has become considerably larger than its inmediate neighbours, it can grow at their expense, at a level of normal grain growth inhibition which may still prevent the growth of smaller grains since the boundary tension forces at the edge of an exceptionally large grain will be further from equilibrium, and will therefore provide a greater driving force for growth by successive straightening and recurving, than is the case for the (inhibited) smaller grains.

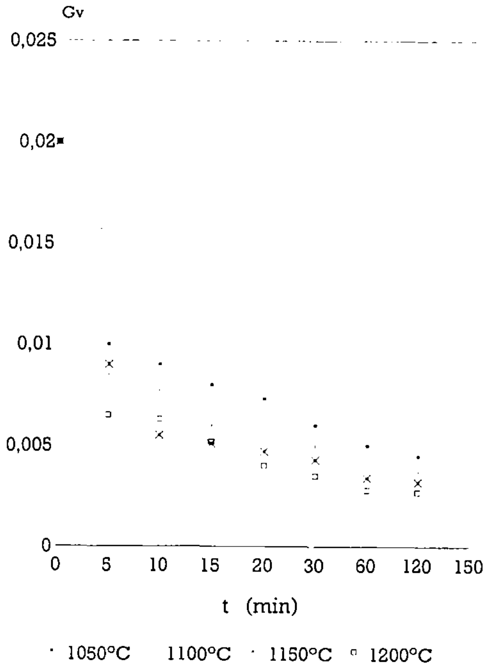

Fig.7. Ratio of the grain boundary area per unit volume at each temperature and for each heat treatment time (min).

Fig.8. Microstructure of a sample heat treated at 1200°C during 120 min.

Discussion

The Figs. 3, 4, 5 and 6 show very rapid grain growth up to 15-20 minutes of heat treatment time, followed by a decreasing grain growth rate. Such decrease can be interpreted in terms of the grain size increase which produces a decrease in the grain boundary area per unit volume ratio. This means that the grain boundary interfacial energy per unit volume decreases and therefore, the driving force for grain growth is lower. The final result is a slower growth kinetics which will eventually decrease to zero when the grain boundary area will not be able to introduce further growth at such heat treatment temperature.

The average value of the growth exponent is 0.56, which is high compared to that of other metals and alloys. Aluminium (99.99%) shows a growth order of 0.1 at 400°C, which increases to 0.3 at 600°C (9); for the austenitic grain of carbon steels (0.8%C), it ranges between 0.1 at 760°C and 0.23 at 980°C (10) and for cartridge brass (70%Cu, 30%Zn) it is 0.2 in the range of 500-700°C (10). In the case of b.c.c. alloys, where the structure is more open and prone to diffusion, the growth order is higher and for α-Fe the values increase from 0.2 at 600°C to nearly 0.5 at 800°C (10) whilst for the ß-brasses the values range from 0.35 at 500°C to 0.60 at 850°C (9, 11).

From the results shown in Table II, it can be noticed that the growth exponent increases with temperature for the area, minimum diameter and maximum diameter parameters, whilst it decreases for the perimeter. Such behaviour seems to indicate that the grains tend to become spheroidized. This means that as the radius of curvature of the grain boundaries increases, the growth rate of the grains decreases since the atomic transfer through the grain boundary varies inversely with the curvature radius (1). In fact when the shape factor of the grains is calculated (ratio between the ideal spherical area calculated with the average diameter and the area experimentally determined), as shown in Table IV, it can be noticed that it tends to increase with both heat treatment temperature and time. Ideally, the shape factor would be one for an equiaxed spheroidized grain structure. Such behaviour confirms that the grains tend to become spherical by reducing their perimeter and by increasing their area and their maximum and minimum diameters in the two dimensional experimental data.

Table IV. Shape factor of the grains.

t/T	1050°C	1100°C	1150°C	1200°C
5	0.68	0.68	0.70	0.70
10	0.69	0.70	0.70	0.70
15	0.70	0.70	0.70	0.72
20	0.71	0.72	0.73	0.74
30	0.71	0.72	0.73	0.75
60	0.73	0.73	0.74	0.76
120	0.73	0.74	0.75	0.77

In general, the maximum value of the growth exponent in the kinetics of normal growth is 0.5. Departures from n=0.5 to higher values have been currently interpreted in terms of solute drag or the effect of texture (12). Although texture is normal in this titanium alloy, it does not seem to play a leading role when normal grain growth is observed. It can be noticed from the results shown in Table II that the growth exponent for the ß Ti-6Al-4V alloy is slightly higher than 0.5. The physical explanation of such behaviour lies in the diffusion of solute atoms which induces the grain boundary migration. In fact, in the initial mill annealed structure, the solute atoms of aluminium and vanadium are not evenly distributed in the two phases and the transformation to ß phase produces a drag effect at the grain boundaries. The diffusion process which leads to solute depletion at the grain boundaries induces an increase in the rate of growth (13).

The activation energies for grain growth are virtually independent of the grain size parameters used in their determination. The actual values obtained are small when compared to other metallic systems like α-Fe in alpha phase where the value is 239 KJ/mol, or in γ-Fe which is 270 KJ/mol for Cu in Al where it is 136 KJ/mol and for Zn in Cu where it is 171 KJ/mol (10). Therefore, it seems that for Ti-6Al-4V the diffusion through grain boundaries is easier than in the others alloys and consequently, the grain growth will take place faster.

Acknowledgements

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