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Low temperature relaxation of residual stress in Ti–6Al–4V

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Abstract

Isothermal tests were performed on Ti–6Al–4V to observe time-dependent changes in the lattice parameter of the α (hexagonal close-packed) and β (body-centered cubic) phases. Synchrotron-based experiments show contraction in β at temperatures below 550 °C, and expansion at higher temperatures. Contraction results from annealing of preexisting residual stresses, while expansion results from V diffusion when the α → β phase transformation becomes kinetically active.
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1. Introduction

Microstructural changes that occur during heat treating and welding of Ti–6Al–4V affect the grain size, β content, β distribution, and ultimately the mechanical properties of the alloy [1,2]. These changes have recently been investigated using synchrotron-based in situ X-ray diffraction (XRD) to develop a better understanding of the microstructural changes that occur during welding [3,4], and constant heating rate experiments [5]. Measurements of the lattice parameters and phase fractions made during these experiments provided details about the individual response of the α and β phases during the α → β transformation. Results showed that the lattice expansion of the β phase is strongly affected by both thermal and chemical effects during the α → β transformation. In particular, the partitioning of V to the β phase during the α → β transformation resulted in a significant increase in its lattice parameter above that expected by thermal effects alone.

While performing the constant heating rate experiments, an unexpected dip in the lattice parameter of the β phase was observed just before the α → β transformation initiated [5]. This dip is studied here in more detail using isothermal experiments and in situ synchrotron-based X-ray diffraction performed at the advanced photon source (APS). The results of these experiments confirm the contraction of the β lattice parameter at temperatures below 550 °C.

2. Experimental procedures

2.1. Materials

Ti–6Al–4V ELI was obtained in 100 mm diameter bar stock that had been mill annealed at 705 °C for 2 h then air cooled to room temperature. Chemical analysis showed that the alloy contained 6.0Al, 4.2V, 0.11O, 0.17Fe, 0.0028H, 0.014C, 0.009N, <0.03Si, bal. Ti, by wt.%. Fig. 1 shows the microstructure of the starting material, which was polished and chemically etched using a modified Kroll's solution containing 5 ml HF, 10 ml HNO_3, 30 ml lactic acid for approximately 30 s.

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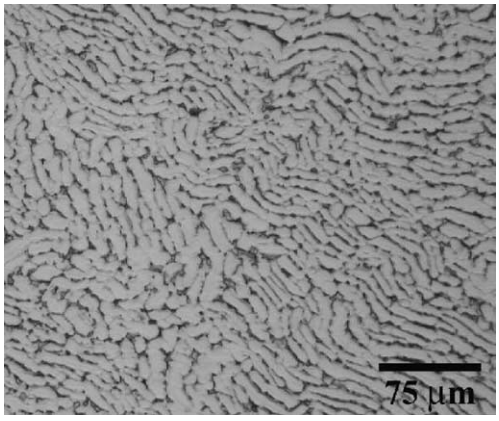

Fig. 1. Light optical micrograph showing the microstructure of the Ti–6Al–4V base metal. The dark etching phase is β.

The microstructure contains a small amount of β distributed intergranularly around the elongated α grains. Micro-chemical compositions of the starting α and β phases were determined using microprobe analysis. These results showed that the initial microstructure contains 12.1% β and 87.9% α phase [5], that V and Fe partition to the β phase while Al partitions to the α phase [5].

2.2. In situ X-ray diffraction experiments

The X-ray diffraction measurements were performed on the UNICAT beam line BM-33-C at APS using a 30 keV X-ray beam from a ring current of 100 mA. After passing through a water cooled Si(1 1 1) monochromator, and the beam was focused and sized to dimensions of 1 mm wide by 0.25 mm high using a dynamically bent Si crystal and collimator slits. The CCD X-ray detector was manufactured by Roper Scientific (A99k401, RS/Photometrics) and uses 2 in. square array of 1024×1024 pixels spaced 60 μm apart to capture the diffraction patterns produced on a scintillating screen. Typical diffraction patterns and additional details about the diffraction setup are provided elsewhere [5].

In situ lattice expansion was measured from shifts in Bragg angles relative to their initial room temperature values. The initial lattice parameters ($a = 2.930$, $c = 4.677$ for hexagonal close-packed (hcp), $a_0 = 3.224$ for body-centered cubic (bcc)) were measured on the Ti–6Al–4V base metal alloy using a conventional CuKα XRD instrument. During the in situ experiments, the average lattice expansion of the bcc (1 1 0) and (2 0 0) peaks was used for the β phase, and the average lattice expansion of the hcp (0 0 2), (1 0 1), (1 0 2), and (1 1 0) peaks was used for the α phase.

Changes in the lattice parameter were determined from changes in the 2θ peak positions during the experiments. The 2θ peak positions contain small systematic errors caused by movement of the synchrotron beam relative to the location of the X-ray detector, which was placed 30 cm away from the sample. The estimated vertical beam motion of 0.25 mm corresponds to an angular deviation of approximately 0.0043° on the CCD camera. This deviation results in a relative variation in d-spacing of 0.00048 Å, which further corresponds to 0.0012 Å in the bcc lattice parameter and 0.0011 Å in the hcp lattice parameter.

Ti–6Al–4V test coupons measured 100 mm long by 4.75 mm wide by 2 mm thick and were heated by passing electrical current through them while they were clamped into a water-cooled copper fixture. This direct resistance heating setup allowed the samples to be heated at rates up to 100 °C/s, while water cooled grips allowed the sample to be rapidly cooled at similar rates. The isothermal experiments were performed by rapidly heating the samples to a predetermined temperature, holding at this temperature for a predetermined time, and then rapidly cooling the samples to room temperatures. Additional details about the experimental procedures can be found elsewhere [5]. To minimize oxidation of the samples during the high temperature runs, all of the experiments were performed in an environmentally controlled chamber which was pumped down to a minimum of 10 mTorr and backfilled with high purity helium gas prior to heating the sample.

3. Results

At low temperatures, before the $\alpha \rightarrow \beta$ transformation begins, the crystal lattice expands during heating due to thermal expansion effect only. The thermal expansion coefficient for Ti–6Al–4V is approximately a weighted average of that of the α and β phases and was measured to be 9.5×10^{-6} °C^{-1} up to a temperature of approximately 500 °C for this alloy [5]. Above 500 °C, the expansion coefficient dips slightly under the constant heating rate conditions then increases to 12.8×10^{-6} °C^{-1} during the $\alpha \rightarrow \beta$ transformation [5]. The dip in the expansion coefficient that was observed at approximately 500 °C was not expected since it occurred before any significant transformation took place, and because 500 °C was too low a temperature to expect that diffusion of alloying elements (V, Al) would occur under the moderately high heating rates examined.

In order to investigate the dip in the expansion of the lattice parameter of the β phase, isothermal holds were performed here at six different temperatures, ranging from 400 °C to 650 °C, in 50 °C increments. Fig. 2a shows the results of two of the isothermal experiments, comparing the lattice parameter of the β phase as a function of isothermal holding time at 450 °C and 600 °C. The lattice parameter of β initially increased from its room temperature value to higher values during heating

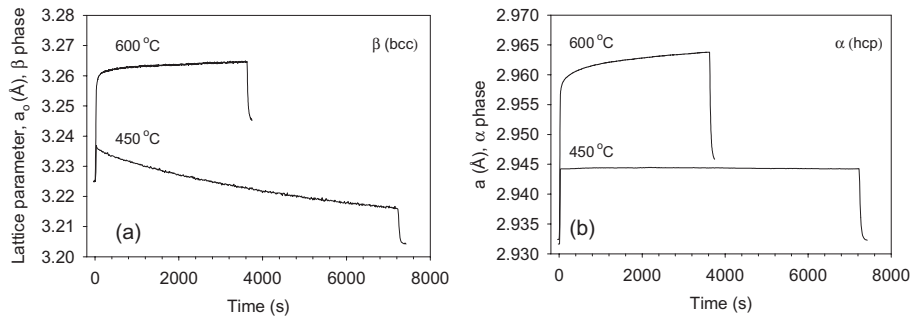

Fig. 2. Lattice parameters as a function of isothermal hold time at 450 °C and 600 °C for (a) the β phase and (b) the α phase.

to the isothermal temperature. In these experiments, the samples required less than 30 s to reach the isothermal holding temperature at a controlled heating rate of 25 °C/s. The samples were held at their respective elevated temperatures for a duration of 1 h, except for samples held at the two lowest temperatures which were held for 2 h. The additional hold time at the lowest temperature allowed more complete contraction of the lattice parameters. Although the majority of the transformation occurred within the first hour at low temperatures, it appears as though the changes had not quite completed at the end of the 2 h hold periods. This incomplete contraction of the sample is believed to cause only a minor quantitative difference, and will not affect the overall trend of the results reported here.

Fig. 2a shows the results of the isothermal hold at 450 °C where the lattice parameter of the β phase continually decreased from its initial value of 3.2350 Å at 450 °C, to 3.2163 Å. In comparison, at 600 °C the lattice parameter of the β phase increased from its initial value of 3.2495 to 3.2646Å. In both cases the lattice parameter of the β phase decreased rapidly when the alloys were cooled to room temperature, but did not return to their starting values. After the tests were completed, the final β lattice parameter for the 450 °C hold was smaller than its initial room temperature value, while that of the 600 °C hold was larger than its initial room temperature value. Fig. 2b plots the corresponding changes in the lattice parameter of the α phase as a function of holding time at the same two temperatures. For the α phase, the lattice parameter increased during both the 450 °C and 600 °C isothermal holds, suggesting different behaviors of the α and β phases, particularly at low temperatures.

The low temperature behavior (lattice contraction during the isothermal hold) of the β phase was observed at 400, 450, 500 and 550 °C, whereas the high temperature behavior (lattice expansion during the isothermal hold) was observed at 600 and 650 °C. This data is summarized in Table 1 for the six tests, which gives the lattice parameters for both phases during the different stages of the isothermal experiment. In this table, the lattice parameters are measured before heating (initial), immediately after achieving the isothermal temperature (start isothermal), immediately before cooling the sample (end isothermal), and immediately after the sample had completely cooled to room temperature (final).

Fig. 3 compares the change in the lattice parameters during the isothermal hold with the total change in the lattice parameters at room temperature before and after the test. In Fig. 3a the change in the β lattice parameter is shown as a function of temperature, indicating a clear trend whereby the lattice parameter decreases during the isothermal hold (circles) at temperatures of 550 °C and

Table 1
Summary of the bcc and hcp lattice parameters, both during and after the isothermal hold experiments

	2 h		1 h			
	400 °C	450 °C	500 °C	550 °C	600 °C	650 °C
bcc, a_0, (Å)						
Initial	3.2250	3.2250	3.2250	3.2250	3.2250	3.2250
Start isothermal	3.2313	3.2350	3.2407	3.2442	3.2495	3.2535
End isothermal	3.2191	3.2163	3.2259	3.2363	3.2646	3.2711
Final	3.2073	3.2043	3.2101	3.2192	3.2452	3.2498
hcp, a, (Å)						
Initial	2.9320	2.9320	2.9329	2.9320	2.9320	2.9320
Start isothermal	2.9440	2.9447	2.9472	2.9488	2.9563	2.9579
End isothermal	2.9441	2.9450	2.9478	2.9498	2.9634	2.9664
Final	2.9329	2.9331	2.9328	2.9338	2.9454	2.9459

The test duration was lengthened from 1 to 2 h at the two lowest temperatures to allow more time for the stresses to relax.

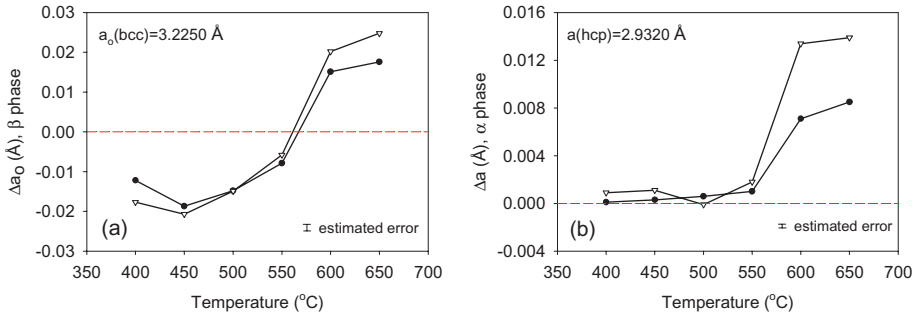

Fig. 3. Changes in the lattice parameters for (a) the β phase, and (b) the α phase. The circles symbols correspond to the net change during the isothermal hold; the triangles correspond to the net change at room temperature before and after the experiment. The estimated error for the bcc lattice parameter is 0.0012 Å and that of the hcp phase is 0.0011 Å.

below. This amount of decrease in the lattice parameter matches the difference in lattice parameters before and after the test (triangles) at these temperatures, indicating that the change occurs during the isothermal hold. Between 550 °C and 600 °C, the lattice parameter of the β phase increase to positive values, indicating that the lattice is expanding during the isothermal hold. In addition, a net difference in the lattice parameter before and after the test occurs, such that the lattice parameter after the test is larger than the increase in the lattice parameter that occurred during the isothermal hold.

Fig. 3b plots the change in the lattice parameter of the α phase as a function of temperature, showing that the lattice parameter of the α phase increases slightly during the isothermal hold for temperatures up to 550 °C. At temperatures above this level, the increase becomes much larger. As with the β phase, the change in the lattice parameter of the α phase before and after the test matches that observed during the isothermal hold for temperatures up to 550 °C. At higher temperatures, the overall change in lattice parameter is larger than the amount of increase that occurred during the isothermal hold.

4. Discussion

The elevated temperature isothermal hold can affect the lattice parameters of the α and β phases in several ways. At high temperatures, the α phase will transform to the β phase according to the thermodynamics of the system [5]. Since V concentrates almost entirely in the β phase, the concentration of V in the β phase will depend on its volume fraction. As the fraction β increases during the transformation, its V concentration will decrease, which will result in an overall increase in the lattice parameter of the β phase [3–5]. This effect has been measured, showing that the lattice parameter of β will increase from 3.21 Å to 3.26 Å as the V concentration decreases from 19 to 11 wt.% [6]. Paradoxically, the V content of the α phase decreases with increasing temperature above 750 °C due to the phase transformation and thermodynamics of the system [5], and the α lattice parameter should show a corresponding increase as its V content decreases [5]. During cooling, the β → α reverse transformation would result in the opposite effect. Since these transformation are diffusion controlled [5,7–9], the temperature needs to be high enough to enable the redistribution of alloying elements.

At low temperatures, where long range diffusion of alloying elements is too slow to induce significant phase transformations, changes in lattice parameters would more likely be related to stress relaxation. The sources of the residual stress in the as-received alloy are differential thermal expansion of the α and β phases and phase transformations resulting from the thermomechanical processing of the base metal alloy. During cooling to room temperature from 705 °C (the mill annealing temperature of this alloy), the atomic volume of the β phase decreases more than that of the α phase [5,10,11]. The decrease in atomic volume of the β phase relative to the α phase generates tensile stresses in β up to its the yield stress as the alloy cools. During reheating, as in the experiments performed here, the tensile stresses in the β phase relieve, causing its lattice parameter to decrease. The maximum recoverable strain during stress relaxation would be related to the elastic modulus of the β phase and its yield stress. Here, the macroscopic values for the elastic modulus and yield stress of Ti–6Al–4V of 106 GPa and 1100 MPa, respectively [12], were used to represent those of the β phase. The maximum strain that would be recovered during stress relaxation can be approximated by the ratio of the yield stress to the elastic modulus which is 1.04%.

The results shown in Table 1 indicate that the decrease in the β lattice parameter varied with isothermal temperature. The maximum change was observed during the 450 °C isothermal hold, where the lattice parameter decreased from 3.2350 to 3.2163 Å. This change corresponds to a decrease in the β lattice parameter of 0.57%, which is approximately half of the total strain

that could be relaxed during annealing if the residual stress level was at or near the yield stress at room temperature. Longer hold times at 450 °C would probably increase the amount of measured relaxation since the plot shown in Fig. 2a does not appear to have reached its minimum level at the end of the 2 h run. Thus, the observed change in the lattice parameter of theβ phase seems reasonable if the change is due to stress relaxation. At 400 °C, a smaller decrease in the lattice parameter of β was observed during the isothermal hold, presumably due to the fact that the relaxation was occurring at a slower rate and the stress relaxation was less complete than at 450 °C. Longer hold times would most likely have increased the amount of stress relaxation in this sample.

At temperatures above 550 °C, the β lattice parameter increases during the isothermal hold rather than decreases. This behavior cannot be explained by stress relaxation, and indicates that another mechanism is contributing to the changes in lattice parameters at these temperatures. Here, it is likely that diffusion of alloying elements is occurring, which allows a partial $\alpha \rightarrow \beta$ transformation to take place. This would explain an increase in the β lattice parameter due to the redistribution of V as previously mentioned. In addition, a phase transformation contribution to the lattice parameter may or may not be recovered on cooling to room temperature, depending on the cooling rate. The results presented in Fig. 3 show that the β lattice parameter increased after the alloy has cooled to room temperature for isothermal holds at 600 °C or higher. These results support the fact that diffusion is occurring during the isothermal hold and indicate that the cooling rate was fast enough to retain some of the β phase that was created at high temperatures.

Fig. 3b plots the corresponding changes in the α lattice parameter, which also shows a transition in behaviors between 550 °C and 600 °C. At 550 °C and below, the α lattice parameter increases slightly during the isothermal hold, and retains this same amount of change after the alloy has cooled back to room temperature. This slight increase in the α lattice parameter is consistent with the decrease in the β lattice parameter during stress relaxation of the two phase mixture. The smaller change in the α phase compared to the β phase can be explained by their relative volume fractions in the microstructure whereby the minority β phase is strained more severely than the surrounding α matrix. The α phase shows a lattice expansion at high temperatures, and exhibits a room temperature lattice parameter that is larger than that before the experiment. Above 600 °C, the change in the room temperature α lattice parameter is larger than the amount that occurs during the isothermal hold, indicating again that some $\alpha \rightarrow \beta$ phase transformation occurred during the thermal cycle.

5. Conclusions

(1) Two different types of lattice parameter changes were observed in the β phase using in situ X-ray diffraction. At temperatures below 550 °C, the β lattice parameter decreased with holding time, showing a contraction of 0.57% after 2 h at 450 °C. Above 550 °C, the β lattice parameter increased with holding time.

(2) The time-dependent decrease in the β lattice parameter at temperatures 550 °C and below is caused by relaxation of pre-existing residual stresses that were created in the two phase alloy as it cooled from its original high temperature processing cycle. During stress relaxation, the β lattice parameter changes less than α because of the large difference in the relative amounts of each phase in the microstructure.

(3) The time-dependent increase in the β lattice parameter at temperatures above 550 °C is caused by the $\alpha \rightarrow \beta$ phase transformation, which becomes kinetically active at these temperatures. The resulting increase in the lattice parameter is the result of the partial $\alpha \rightarrow \beta$ transformation and redistribution of V within the growing β phase.

(4) The lattice parameter of α also changes during the isothermal hold. At temperatures 550 °C and below, its lattice parameter increases slightly to compensate for the corresponding decrease in the β lattice parameter as the residual stresses relax. At temperatures above 550 °C, the α lattice parameter shows an additional increase after cooling to room temperature due to the effects of the $\alpha \rightarrow \beta$ phase transformation.

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