

The Effect of ω - and α -Phase Precipitation on the β -Phase Lattice Parameters During 400°C aging in Ti-11Cr(at.%)

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β -stabilizing elements, which reduce the β -transus temperature, are added to Ti alloys to assist in the retention of the β phase after quenching from above the β -transus temperature. The lattice parameter of the bcc β phase is affected by the addition of such elements. For example, the β -phase lattice parameter increases with increasing Nb content [1] and decreases with increasing Mo or Cr content [2, 3]. Additionally, heat treatments are used to induce the β -to- ω and β -to- α phase transformations in β -Ti alloys. During the precipitation and growth of the ω and α phases, β -stabilizers diffuse out of the ω and α precipitates and into the surrounding β matrix [4, 5]. This diffusion changes the β -phase composition, which affects the β -phase lattice parameter.

While the β -phase lattice parameters of Ti-Cr based alloys have been investigated as a function of alloy composition [3], the lattice parameters of the β phase have not been tracked during the phase transformations. In this work, atom probe tomography (APT) and high-temperature X-ray diffraction (HTXRD) were used to investigate the relationship between the β -phase lattice parameter and phase composition during the β -to- ω and β -to- α phase transformations.

A Ti-11Cr (at%) alloy was levitation melted in a 2kg, 90Dx80L LEV levitation induction furnace and hot forged at approximately 1050°C into 25 x 60 x 250-mm³ blocks, then homogenized using a 900°C anneal for 1 h in vacuum and ice-water quenched at an estimated cooling rate of 34.7°C/s. A 400°C heat treatment was chosen to induce the ω - and α -phase transformations.

Samples for APT sample preparation were cut from the forged alloy blocks using a diamond saw, aged at 400°C for 0.75 h in vacuum followed by air quenching, then metallographically polished to a mirror finish according to [6]. APT needle specimens were prepared from those samples by the FIB-based lift-out and annular milling method described in [7] using a Thermo Fisher Quanta 3D field emission gun (FEG) dual beam FIB/scanning electron microscope (SEM) equipped with an Omniprobe nanomanipulator. A CAMECA local electrode atom probe (LEAP) 4000X HR system, in pulsed-voltage mode with a 200 kHz pulse frequency, 50 K specimen temperature, pulse fraction of 0.2, and detection rate of 0.5%, was used for all APT data collection.

Samples for HTXRD were cut using an electrodischarge machine and polished using 320 grit silicon carbide paper and water to remove any macroscopic surface defects or oxide. Final sample dimensions were approximately 17mm x 17mm x 1.1mm. HTXRD was performed using a Bruker-AXS (Madison,

WI) D8 diffractometer with an automatic sample changer, a Vantec linear position-sensitive detector, Cu-K α radiation, and an Anton-Paar HTK1200 furnace with ultra-high-purity nitrogen gas. The room temperature scan of the initial β -homogenized condition exhibited only the (101) β , (200) β , and (211) β peaks, confirming the fully β -phase microstructure. The heating rate to 400°C was 30°C/min. Data was collected in-situ over a 2θ range of 25° to 75° every 0.5 h during the 12 h 400°C exposure. The Rietveld refinement and lattice parameter calculations were accomplished using software suite PDXL version 2 [8].

HTXRD scans indicated that the ω and α phases form between 0 and 0.5 h at 400°C. ω - and α -phase peaks were present throughout the 12 h 400°C treatment. The β -phase peaks shifted to a higher angle with time, shown in Figure 1(a). Rietveld analysis was used to determine the lattice parameter of the β phase for each HTXRD scan, see Figure 1(b). The β phase decreased from 3.235Å at 0.5 h to 3.188Å at 12 h. The β -phase lattice parameters were fit to a logarithmic curve with an R^2 of approx. 0.91. The weighted-profile residual (Rwp) for each Rietveld analysis was between 4.87% and 7.85%.

APT was used to collect detailed, high-resolution compositional data of the phases. Because APT experiments indicated that the ω and α phases are Ti-rich and β -stabilizer poor [4, 5], a Cr isosurface corresponding to 8 at.% Cr was plotted on all samples, see Figures 2(a) and (c). These isosurfaces illustrated the interfaces between the precipitates and the β phase, with the β phase being Cr-rich. The Cr isosurfaces were used to create proximity histograms in all samples and the average composition of each phase was determined. A representative example is shown in Figure 2(b). Plotting the average β -phase composition from all tips, see Figure 2(c), shows that the β phase decreased in Ti content and increased in Cr content with time, see Figure 2(d).

During the transformation, the Cr diffused from the α and ω phases into the β phase and the Ti diffused from the β phase into the α and ω phases [4, 5]. The increase in Cr and decrease in Ti in the β phase was greater between 0.75 and 3 h than between 3 h and 12 h. The β -phase lattice parameter also decreased more rapidly during the first 4 h at 400°C than between 4 h and 12 h. To determine the relationship between composition and the lattice parameter of the β phase, lattice parameters for 0.75, 1.5, 3, 6, and 12 h were calculated from the logarithmic trendline in Figure 1(b). Plotting those calculated lattice parameters against the average concentrations of the β phase for each time shows a linear relationship between Ti concentration, see Figure 2(e), Cr concentration, see Figure 2(f), and lattice parameter.

HTXRD and APT were used to determine the relationship between composition and lattice parameter for the β phase in Ti-11Cr. HTXRD and Rietveld analysis allowed the evolution of the lattice parameters of the β phase at 400°C to be observed. The β -phase lattice parameter decreased with an increase in Cr and decrease in Ti concentration as the ω and α phases precipitated and grew. This changing lattice parameter could affect the strain between the β matrix and the precipitates as they form in this and other Ti-Cr alloys, influencing further phase transformations and mechanical properties [9].

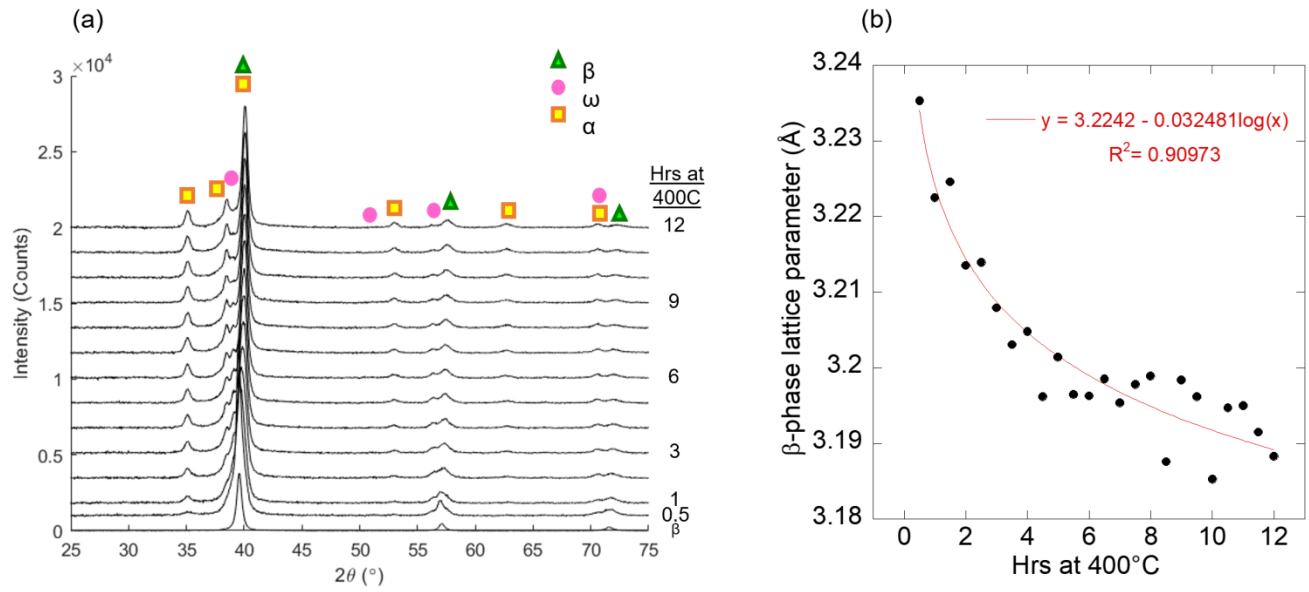


Figure 1. HTXRD intensity versus 2θ plot (a) showing the evolution of the β -, α -, and ω -phase peaks of Ti-11Cr at 400°C for different time intervals. The β -phase lattice parameters as a function of time at 400°C (b) calculated from the HTXRD scans using Rietveld analysis.

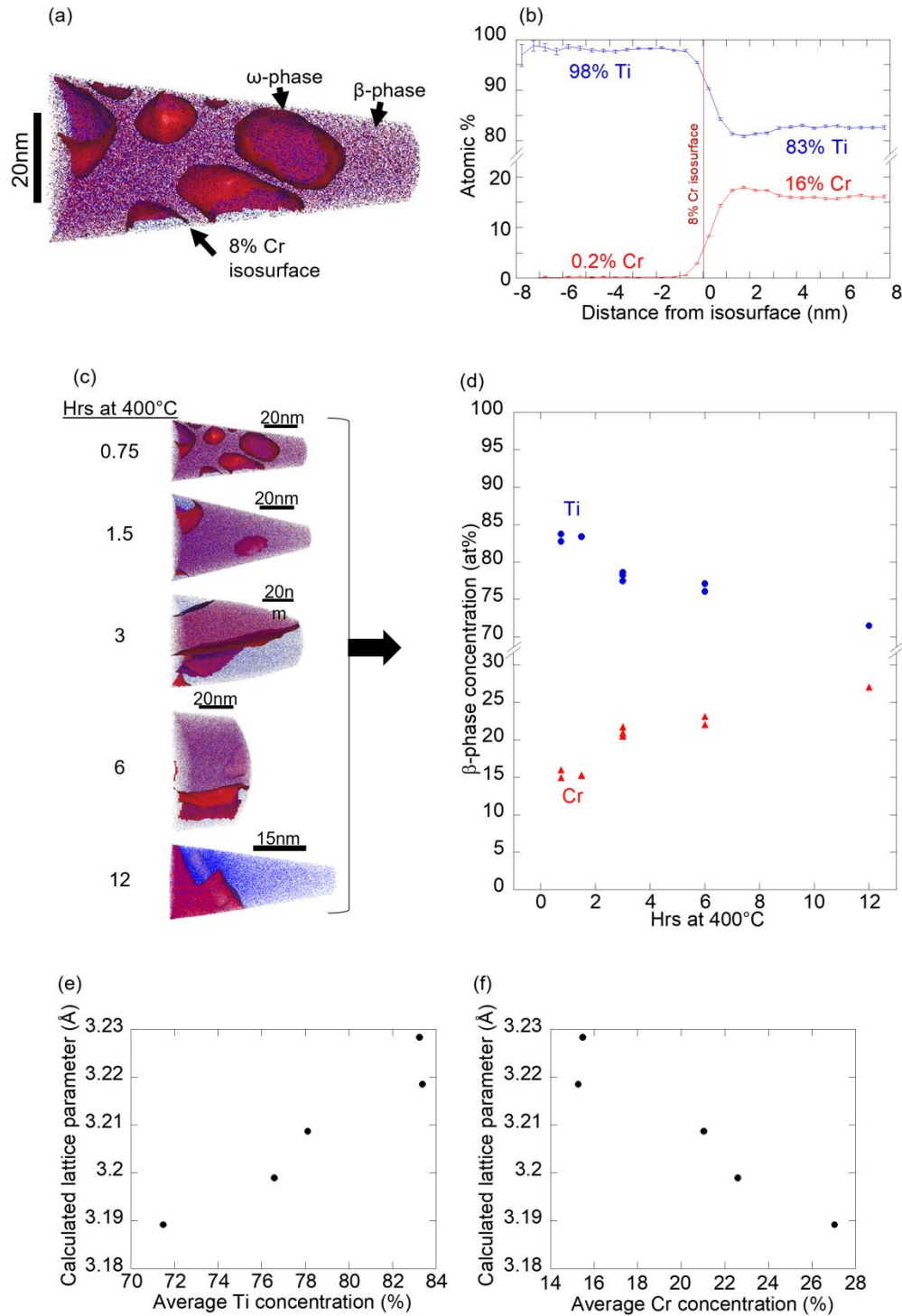


Figure 2. APT tip reconstruction of 0.75 h 400°C Ti-11Cr (a) with corresponding proximity histogram (b) showing the β phase as Cr-rich. Representative APT tip reconstructions (c) used to find the average β -phase compositions as a function of time at 400°C (d). The calculated β -phase lattice parameters as a function of Ti concentration (e) and Cr concentration (f).

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