

Direct Real Time Microstructure Evolution Observation of Ti-6Al-4V Alloy by *In-Situ* EBSD during Cycling Heating and Cooling at Elevated Temperatures using μ heater in SEM/FIB Systems

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As the most widely applied industrial materials in aerospace and medicine fields, titanium alloy Ti-6Al-4V has drawn ongoing research interests regarding the control and optimization of its microstructure in order to achieve desired properties. Numerous experimental and modelling efforts have been made to understand the microstructures, phases and phase transformation mechanism in this dual phase alloy in the past decades[1]. However, none of the previous work has achieved direct real time observation of the microstructure evolution of Ti-6Al-4V alloy at desired elevated temperatures (above β transus temperature approximately 995°C) due to technical limitations. Nevertheless, state-of-the-art microelectromechanical system (MEMS)-based micro heating device (μ Heater) brings unique opportunities for convenient, fast and reliable *in-situ* heating (rapid heating/cooling rate in the order of 10^4 K per second up to 1200°C) under high vacuum conditions in SEM/FIB systems[2] and is compatible with all types of detectors such as EBSD, EDS and STEM[3].

In this contribution we present a comprehensive study of *in-situ* EBSD characterizations on deformed dual phase ($\alpha+\beta$) Ti-6Al-4V alloy during cycling heating and cooling treatments between 850°C and 1200°C using μ Heater in a ThermoFisher Scientific DualBeam™ SEM/FIB Helios™ G4 work station equipped with a standard EBSD detector. Chunk sample of size 50x40x20 μm^3 was fabricated using FIB milling from the bulk, subsequently *in-situ* lifted out and welded onto the MEMS chip by an in-chamber micromanipulator (see Figure 1). The sample surface was then cleaned on the MEMS chip with low energy ion to improve EBSD pattern quality. The metallic chunk was first heated at a rate of 10^3 K per second to 850°C, subsequently treated in a cycling heating and cooling process between 850°C and 1200°C using the same heating rate. *In-situ* EBSD maps were acquired at room temperature and during each isothermal annealing step. The average hit rate (indexing percentage) for the maps are above 95% and did not decrease during the heating. It is also shown that high quality EBSD patterns can be collected even at 1200°C attributing to the small thermal mass of the μ Heater. Real time microstructure evolution such as dynamic recrystallization, grain boundary migration (see Figure 2) and phase transformation (Ti $\alpha\leftrightarrow\beta$) (see Figure 3) was observed during the continuous heat treatment sequence. All analytical work was conducted at 20kV and 11nA with a step size of 300nm. In addition, bulk microstructures of both the as-received and post mortem state have been investigated by extracting TEM lamellae from the chunk sample and checking with STEM-in-SEM technique in the same work station.

References:

- [1]C. Kenel *et al.*, Sci. Rep. **7** (2017), p. 1.
- [2]L. Mele *et al.*, Microsc. Res. Tech. **79** (2016), p. 239.
- [3]L. Novák *et al.*, Microsc. Microanal., **23 S1** (2017), p. 928.

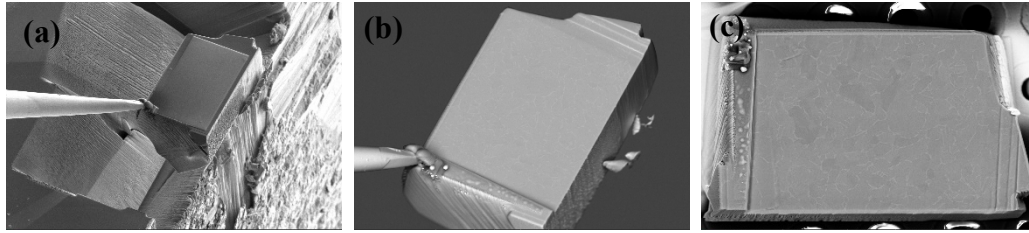


Figure 1. The Ti-6Al-4V chunk sample (a) fabricated in bulk and *in-situ* lifted out by FEI ‘Easylift’ micromanipulator, (b) surface polished and (c) attached to a MEMS heating chip. The chunk surface size is 50µm x 40µm.

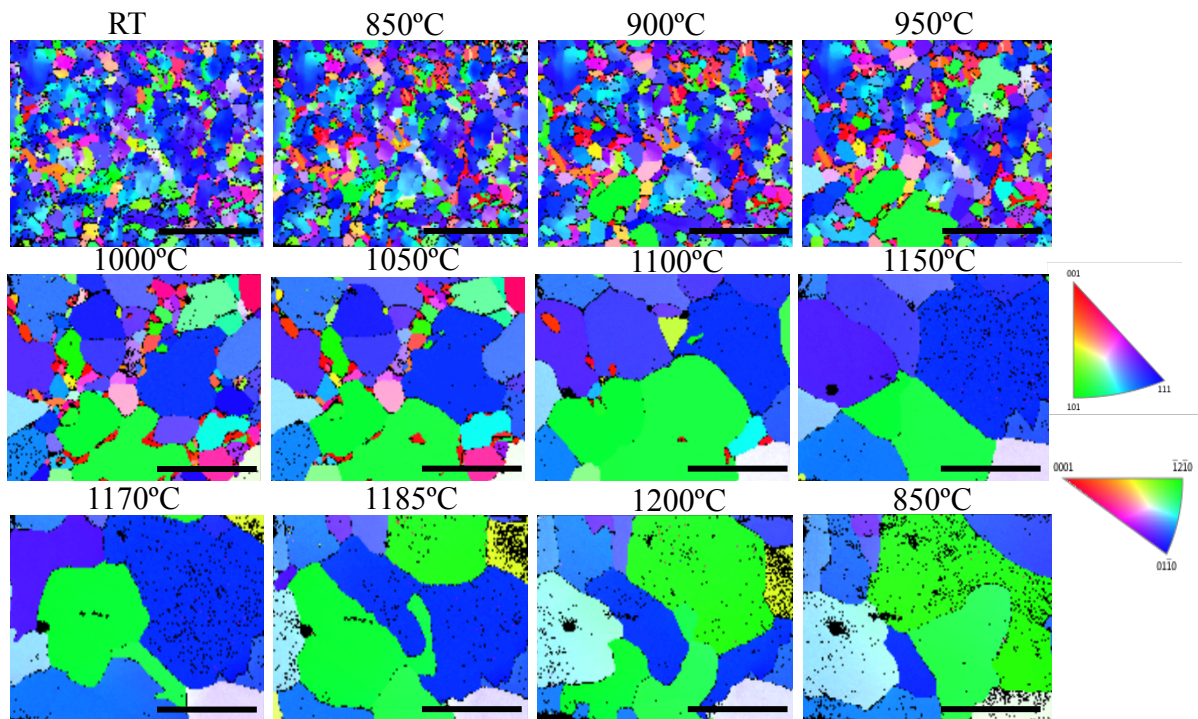


Figure 2. Original EBSD inverted pole figure maps of Ti-6Al-4V alloy collected at room temperature and at each isothermal step during the heating and cooling cycle. Scale bar in each map is 20µm. Each map was acquired in approximately 7 minutes. Sequential IPFZ maps show a distinct recrystallization and grain growth process at elevated temperatures.

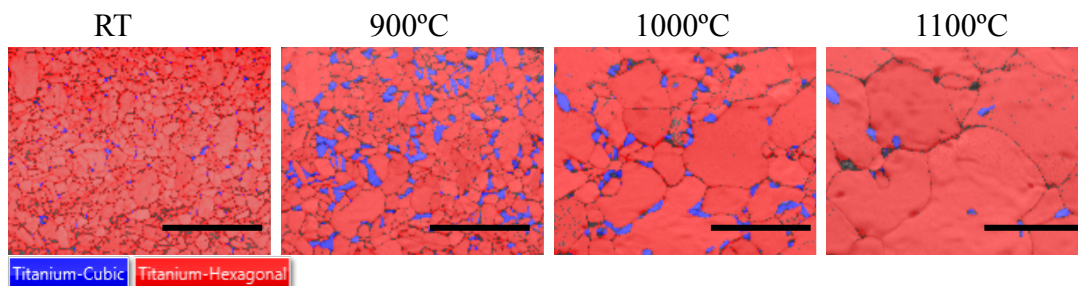


Figure 3. Overlaid phase maps with band contrast maps of Ti-6Al-4V alloy acquired at room temperature, 900°C, 1000°C and 1100°C show a transformation cycle from initial Ti-α(hcp) to Ti-β(bcc) to final Ti-α(hcp) in the *in-situ* heating sequence. Scale bar in each map is 20µm.