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In situ S/TEM Heating Experiments to Study the Effects of Cyclic Thermal Gradients in Additive Manufacturing Build Processes

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Scanning/transmission electron microscopy (S/TEM) characterization methods have become routine techniques to investigate structures in nanomaterials based on unique abilities to analyze crystallographic structure, size, morphology, local defects as well as elemental distributions and electronic properties. However, the actual state and functional behavior of nanomaterials cannot always be inferred from examination under 'standard' imaging conditions. We need to characterize materials in their operating environment [1].

As an example, *in situ* S/TEM heating experiments allow us to understand structural stability and/or dynamics in materials - at temperatures relevant to the actual technical use. Over the past two decades, micro electro-mechanical systems (MEMS) with miniaturized heaters that have superior thermal stability compared to furnace-type heating stages [2,3] have enabled studies of thermally activated processes such as; growth/melting/sublimation of nanoparticles, phase tranformations and grain growth in metals, to name a few. Using these MEMS-based stages (Figure 1), heat can be applied in a precise way to perform controlled cyclic heating experiments at high spatial and temperature resolution [3]. The small thermal mass as well as the direct contact between microheater and specimen offer several benefits such as; fast heating-cooling cycles, rapid stabilization times at high operating temperatures (up to 1300 ^{o}C) and minimal thermal drift. Furthermore, the temperature of a specimen can now accurately be measured inside the S/TEM [4-6], which allows us to reliably interpret and correlate data. Recently, there is a need to develop fundamental understanding of complex thermal phenomena in additive manufacturing (AM) build processes, which demands (*in situ*) characterization tools that can generate temperature gradients across the specimen, in a controlled manner.

Metal-based AM techniques, such as the 'powder-bed fusion' methods, use a scanning electron/laser beam to selectively melt pre-alloyed powders placed over a bed in a layer-by-layer process, thereby manufacturing products of desired complex shapes. Steep thermal gradients in the build process, as well as thermal cycling (re-melting) of before deposited layers, result in unpredictable phase transformations causing inhomogeneties in the microstructure across the AM build. In order to eventually replace established technologies, it is necessary to understand the processing-structure-property relationship in AM builds. Ti-6Al-4V (Ti64) is a well-studied alloy used in the wrought form for critical aerospace and biomedical applications. AM Ti64 is natural candidate alloy system to replace conventionally processed Ti64 parts. Recent reports by *Kenel et al.* [7] investigated the phase transformation in AM Ti64 under laser melting and rapid cooling cycle using high-speed micro-X-ray diffraction (XRD). The focus of our ongoing research is to understand whether *in situ* S/TEM heating experiments could be applied to simulate thermal conditions present in metal-based AM build processes.

In this study, we present MEMS-based *in situ* S/TEM heating experiments, which simulate thermal AM build conditions (ramping up to 10^4K/sec with gradients up to 10^4K/m). Preliminary cyclic heating-cooling *in situ* experiments on Ti64 powder revealed a sequence that transforms hexagonal HCP α phase to form the body centered cubic β phase (on heating) and subsequently transforming the hexagonal α' martensite phase upon cooling to room temperature (see Figure 2) [8]. We will outline further concepts applying well-defined thermal gradients perpendicular and parallel to existing grain boundaries in Ti64. These results will provide further insights into the solid/solid phase transformations during fast heating and cooling cycles [9].

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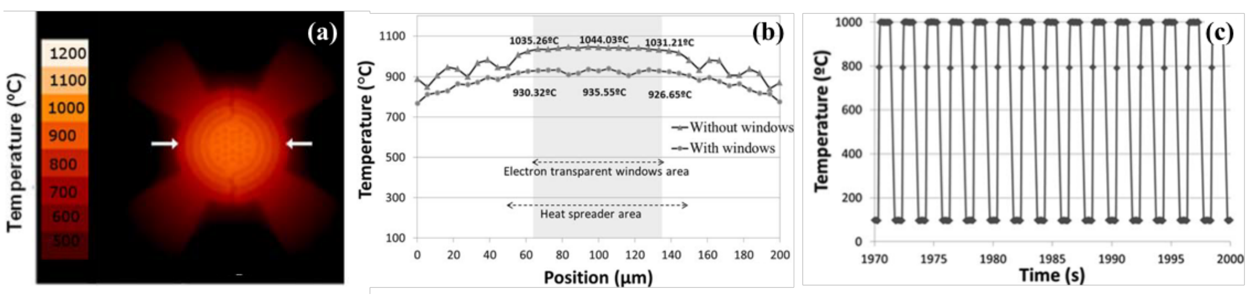

Figure 1. Taken from [3]: Thermal imaging of the MEMS microheater (a) and line scan along the direction indicated by the two arrows (b); (c) Closed-loop fast cycling (shown only the last 15 cycles out of the total of 1000) between 100°C and 1000°C: no variation in the calibration coefficients has been measured. (Re-use from own publication [3] with permission, Copyright © 2016, John Wiley and Sons)

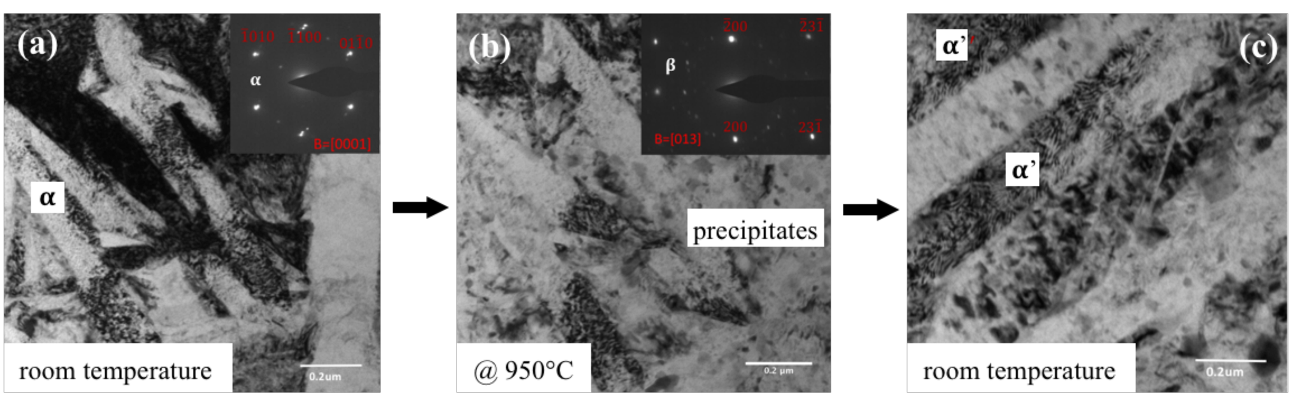

Figure 2. Conventional TEM images taken of AM Ti64 powder in an in-situ heating experiment, (a) BF image taken under zone axis [0001] showing hexagonal α phase at room temperature. (b) the same area taken under zone axis [013] indicating mainly cubic β phase at 950℃. (c) BF images after cooling back to room temperature showing needle-like α’ phase.

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