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Investigation of Solid-state Transformations Under Extreme Thermal Transients Using *In-situ* TEM Heating Experiments

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Micro-electro-mechanical systems (MEMS) based heating stages have revolutionized in-situ transmission electron microscopy (TEM) experiments and are widely used to observe thermally activated processes at high spatial resolution [1]. TEM heating experiments are routinely performed to observe phenomena such as sublimation and melting of metal nanoparticles, phase transformations in complex alloys, and de-wetting of thin films, to name a few. These MEMS-based micro-heaters are ideal to observe solid-state transformations in materials at the atomic scale, because of their reduced sample drift, small thermal mass, fast heater response times, and high temperature homogeneity across the heater surface [2,3].

In metal additive manufacturing (AM) processes, an electron (EBM) or laser heat source melts powder particles/wires that are deposited in a ‘layer-by-layer’ process to build an AM part [4]. The most recent deposited layer experiences rapid melting & solidification, while previously deposited layers undergo complex and repeated thermal cycling processes with rapid (re)heating and (re)cooling causing intricate alloy phase transformations. The unidirectional heat flow and complex thermal cycles results in AM parts with anisotropic microstructures that consists of columnar grains growing along the build direction. Non-uniform properties resulting from these anisotropic microstructures have limited the qualification of AM parts for critical load bearing applications. Major efforts are being directed towards developing AM parts with equiaxed microstructures. However, these efforts are limited by the lack of understanding on the mechanisms that govern solid-to-solid transformations in AM-like processes [5]. AM parts experience large thermal gradients (10^4-10^7 K/m) and high cooling rates (10^3-10^5 K/s) that are drastically different to parameters in conventional manufacturing processes. In order to understand the mechanisms that govern these solid state transformations in AM under extreme thermal transients, we demonstrate the use of a new in-situ MEMS based micro-heater that can simulate AM process conditions inside a TEM.

In this report, we describe the methodology to generate a thermal gradient across a TEM sample using a MEMS-based heater. Subsequently, this device is used to investigate the influence of thermal gradients on solid-solid interfaces in EBM AM Ti-6Al-4V (Ti64). A commercially available MEMS-based micro heater (DENS solutions) is modified using a dual beam focused ion beam scanning electron microscope, Helios-600, Thermo Fisher Scientific (FIB-SEM). A window with dimensions (25 µm X 30 µm) is milled through the silicon nitride membrane next to the metallic spiral heating element in the FIB (Figure 1(a)). Infra-red thermal camera was used to measure the temperature distribution across the MEMS heater surface (Figure 1(b)). The thermal gradient (dT/dx) was measured from the heater to the edge of the window and found to be ~ 10^6 K/m, which is within the range of thermal gradients observed in AM processes. The FIB-cut lamella obtained from a Ti64 gas atomized powder particle was placed [6] across the modified window with one end on the heater and the other end on the silicon nitride membrane that acts as the sink (Figure 2(a-c)). Finally, *in situ* TEM heating experiments were performed to study solid-solid interface stability under AM like conditions [7].

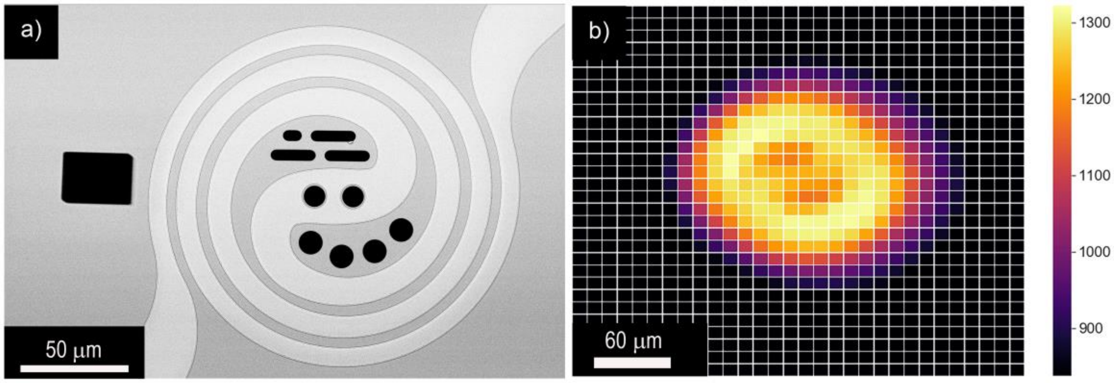

Figure 1. (a) MEMS micro heater with a modified window next to the spiral heating element, (b) Infra-red thermal image of the temperature distribution across the MEMS microheater device at 1300°C

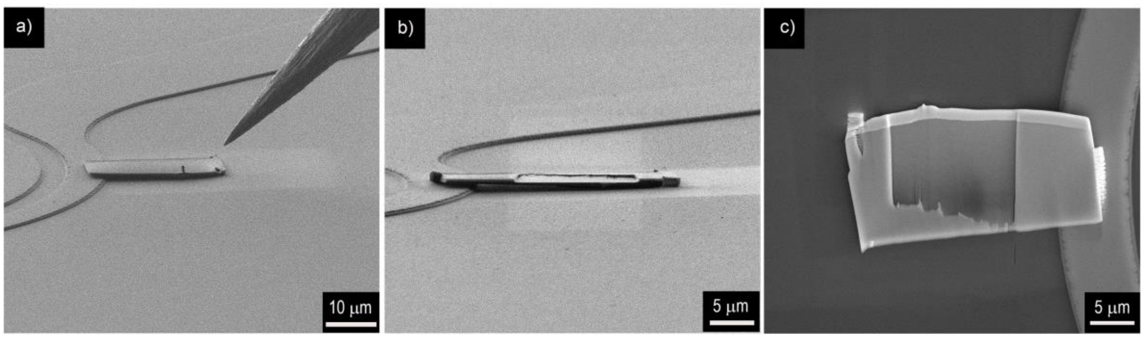

Figure 2. Secondary ion beam (a-b) and secondary electron (c) SEM image of a Ti64 TEM lamella placed over the modified window of the MEMS micro heater. The right side of the lamella is attached to the heating element (heater) and the left end is attached to the silicon nitride membrane (sink).

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