1844
doi:10.1017/S1431927622007255

Microsc. Microanal. 28 (Suppl 1), 2022

In situ TEM Observations of Thermally Activated Phenomena in Materials Under Far-From-Equilibrium Conditions

Sriram Vijayan[1*], Kaustubh Bawane[2], Fidelma Giulia Dilemma[2], Lingfeng He[2], Carolin Fink[1] and Joerg R Jinschek[3]

[1.] Department of Materials Science & Engineering, The Ohio State University, Columbus, Ohio, USA.
[2.] Idaho National Laboratory, Idaho Falls, Idaho, USA.
[3.] National Center for Nano Fabrication and Characterization (DTU Nanolab), Technical University of Denmark (DTU), Kgs. Lyngby, Denmark.
[*] Corresponding author: vijayan.13@osu.edu

Structural materials, that have been fabricated using additive manufacturing (AM) or have been welded using metal joining processes, experience varying spatial and temporal thermal transients due to a very local but high energy heat source. Similarly, components experience varying thermal transients when 'in service', e.g. in next generation nuclear reactor cores, gas turbine engines, re-entry space vehicles and solder joints in micro-electronic packages. These varying thermal transients (extreme thermal gradients (10^4 - 10^6 K/m) and/or rapid thermal cycling (10^2- 10^3 K/s)) cause microstructural changes due to solid-solid phase transformations under far-from-equilibrium conditions forming metastable phases with unknown properties.

In order to improve materials performance, dynamic microstructural evolution under such thermal processing or 'in service' conditions need to be better understood. Currently, this information can only be obtained through *post-mortem* characterization of parts. To study these dynamic processes at high spatial resolution and correlate them to material properties and performance, the development of new *in-situ* heating stages is needed. Previously, in-situ transmission electron microscopy (TEM) heating experiments have been used to study thermally activated phenomena such as defect-solute interactions, diffusion-controlled and interface-controlled phase transformations in structural materials [1-3]. Micro-electro-mechanical-system (MEMS)-based heating stages have the potential to enable such studies. Benefits of MEMS microheaters include fast heater response times, low thermal mass, and high temperature homogeneity [4]. These MEMS heating stages have enabled in-situ investigations of steady-state phenomena at high spatial resolution under isothermal conditions.

Recently, we have modified a commercially available MEMS microheater to enable the generation of a thermal gradient across a TEM sample [5]. Ex-situ infra-red thermography and the in-situ 'Ag nanocube sublimation' technique [4] were used to confirm the temperature distribution and reveal a large thermal gradient (10^6 K/m) across the area of interest [6]. Additionally, the rapid heating and cooling rates of up to 10^3 K/s of MEMS devices allows us to mimic processing and/or 'in service' like conditions for a wide range of applications, inside the TEM.

Here, we show that MEMS based heating stages can be used to replicate transient thermal conditions by adopting three simple strategies, as shown in Figure 1(a-c). *In situ* TEM case studies that mimic the processing and/or 'in service' thermal conditions for three critical applications are presented.

Ductility dip cracking (DDC) is a solid-state phenomenon that occurs in re-heated weld metal or heat affected zones of base metals [7]. DDC is a major problem in Ni-Cr alloy filler metals due to macroscopic stresses during welding and strain localization at the grain boundaries that leads to grain boundary sliding and cracking. The role of grain boundary precipitation on this cracking phenomena is controversial in the literature. In this case study, we investigate the effect of carbide precipitation along grain boundaries under rapid thermal cycling conditions (Figure 1(a)), in a modified Ni-Cr weld filler containing alloying additions of Mo and Ta, inside the TEM.

During nuclear fission, the *in reactor* thermal conditions result in large thermal gradients across the fuel pellet and cladding material. These extreme thermal gradients result in redistribution of constituent fuel elements and fission products along the thermal gradient [8]. Here, we study the effect of *in reactor* thermal gradients on the microstructural evolution in a U-Zr metallic fuel alloy using the modified MEMS heating stage. Additionally, the role of tantalum buffers (Figure 1(b)) to prevent interdiffusion of U and Si (from the silicon nitride membrane) during heating and its impact on the thermal gradient will be presented.

The repetitive melting of powder and remelting of previous layers during metal AM processing results in steep thermal gradients and rapid thermal cycling, which affects the microstructural evolution within AM builds [9]. Here we investigate the microstructural processes that contribute to the solid-state transformations under the influence of gradient heating and thermal cycling (Figure 1(c)) in Ti-6Al-4V alloy.

In summary, the key findings from the three case studies along with the limitations of this modified MEMS device will be discussed [10].

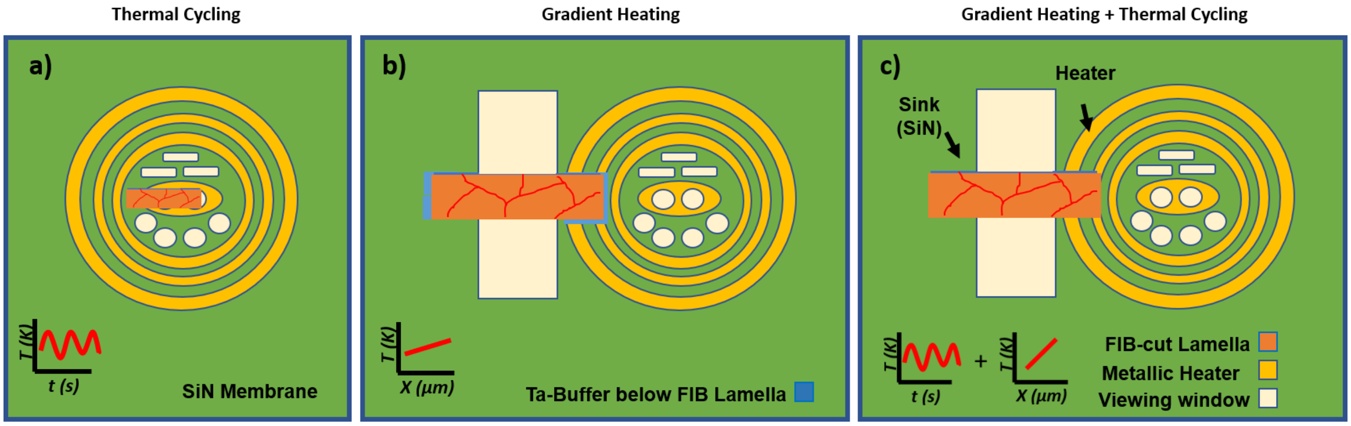

Figure 1. Schematic of (a) an unmodified device used to mimic thermal cycling, (b) the modified MEMS device to mimic *in reactor* thermal gradients across a lamella placed on Ta-buffers (to prevent interdiffusion during heating), and (c) the modified MEMS device used to mimic 'AM like' process conditions by combining gradient heating and thermal cycling

References:

[1] S Vijayan, PhD Theses, 2018, University of Connecticut.
[2] S Vijayan et al., J. Mater. Sci. **54** (2019), p. 9921.
[3] H Leonard et al., Mater. Charact. **178** (2021), p. 111239.
[4] S Vijayan and M Aindow, Ultramicroscopy **196** (2019), p. 142.

[5] S Vijayan et al., Microsc. Res. Techn. (2021) p. 1.
[6] S Vijayan et al., Microsc. & Microanal., **23** (2017), p. 708.
[7] GA Young et al., Welding Journal **87** (2008), p. 31.
[8] GL Hofman et al., J. Nucl. Mater., **227** (1996), p. 277.
[9] M Shao et al., Mater. & Des., **196** (2020), p. 109165.

[10] The authors acknowledge funding from Office of Naval Research (ONR), MURI grant 102359, and by the LDRD program at Idaho National Laboratory (INL)

https://doi.org/10.1017/S1431927622007255