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Focused Ion Beam Sample Preparation for High Temperature In-situ Transmission Electron Microscopy Experiments: Use Carbon for Now

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The dual-beam focused ion beam (FIB) is routinely used for preparing thin sections of bulk specimens for transmission electron microscopy (TEM) experiments. For standard TEM work, the goal of this preparation technique to produce a thin (site-specific) specimen that remains representative of the bulk sample, while minimizing any sample-preparation induced artefacts. This complex procedure involves depositing (usually via ion-beam-assisted deposition) a protection layer, followed by the process of lift-out, attachment, and thinning. However, for a high-temperature in-situ TEM experiment, the behavior of the heated FIB-prepared specimen needs to recapitulate that of an unmodified bulk sample as much as possible; any observed physical changes must not be an artefact of the sample preparation method. Furthermore, the specimen needs to remain mechanically stable under heating, without change of shape, position or, at worst, complete detachment, and any residual FIB-deposited protective material must not interfere.

This work presents an approach to FIB preparation for samples that were heated in an electron microscope, demonstrates its viability, and emphasizes important problems with the most common (Pt and W) adhesion materials. Adhesion of the lamella is usually achieved via local ion-beam modification and deposition of a vapor-injected precursor; the poor thermal stability of most commonly used FIB-deposited materials at high temperatures presents a significant challenge for high temperature TEM imaging which needs to be addressed [1-3]. We will present the findings of our studies of the temperature stability of three commonly used FIB-deposited materials: Pt, W and C. Our initial studies were performed on FIB-deposited lines and blocks on SiN membranes, imaged over a temperature range from room temperature to approximately 1000 C heated using a furnace-style heating holder. These preliminary experiments yielded important information about the evolution and disintegration of these materials as a function of the applied temperature. We will also show observations of silicon lamellae, each welded to a SiN substrate via a different FIB-deposited material, during heating to high temperatures. The final state of each lamella at 1000 C is shown in Figure 1. We will describe how each lamella is compromised by the choice of weld and/or protection material, with macroscopic tungsten mobility, Pt/Ga crystal formation, and the expulsion of Ga droplets from C. Of the three tested FIB-chemistries for specimen protection and adhesion, we will argue that for many situations C is the most suitable; data to support this argument, acquired using a MEMS heating holder, will be shown (Figure 2) in which this preparation approach is found to generate fewer contaminants; we will also demonstrate that the weld remains intact, with minimal lateral dimensional change. However, we will show evidence of Ga “weeping” from the FIB-deposited material, which we will quantify as a function of temperature.

Taken together, these results show the need for care in the choice of FIB-deposited materials during in-situ heating (and related) experiments, and the pressing need for an investigation of other ion beam and precursor chemistry combinations, to allow the full range of materials to be studied in in-situ TEM without contamination or modification of their behaviors due to sample preparation.

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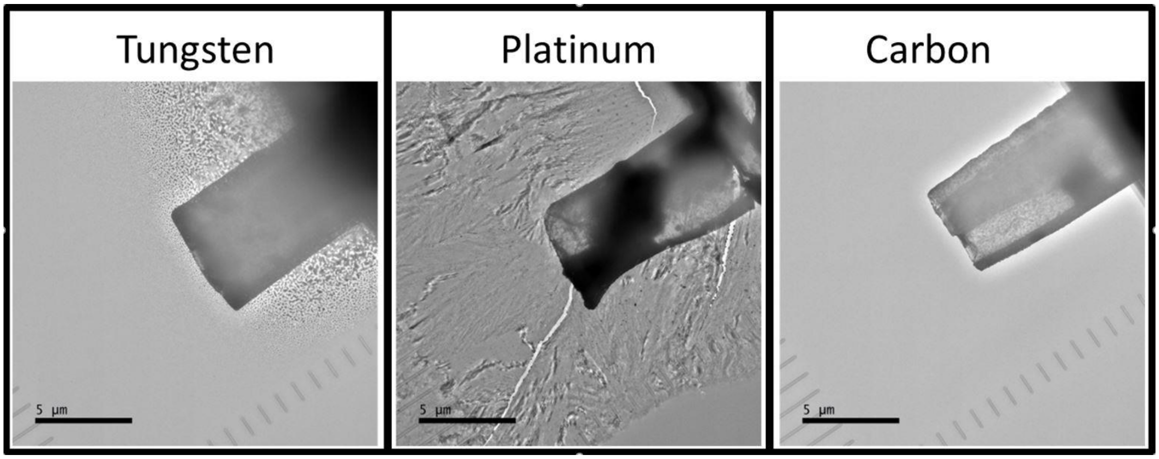

Figure 1. TEM images of Si lamellae welded to a SiN grid after heating to a nominal temperature of 1000 C. The weld material for each lamella is indicated above each image.

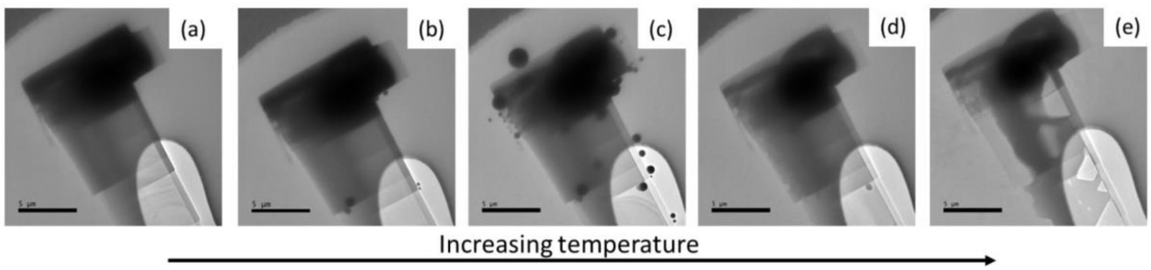

Figure 2. TEM images of a silicon lamella welded to a MEMS heating chip at successively increasing temperatures showing (a) room temperature, (b) the onset of Ga weeping and bead formation, (c) peak Ga bead generation, (d) the removal of Ga beads at higher temperature, and (e) the retention of the lamella and weld at elevated temperatures at which holes are observed in silicon. No significant deflection or translation of the lamella is observed, despite the significant loss of Ga from the weld material.

References

[1] C. Flathmann, H. Spende, T. Meyer, P. Peretzki and M. Seibt *Phys. Status Solidi A* (2019) 216, 1900308

[2] R. Staubinger, A. Beyer and K. Volz *Microsc. Microanal.* (2016) 22, 515-519

[3] M. H. Fawey, V. S. K. Chakravadhanula, M. A. Reddy, C. Rongeat, T. Scherer, H. Hahn, M. Fichtner and C. Kubel *Microsc. Res. Tech.* (2016) 79, 615-624

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