Temperature Calibration of a MEMS-based Heating Holder by Isothermal Sublimation of Silver Nanocubes

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Over the last decade, the introduction of micro electro-mechanical system (MEMS) based heating holders has led to a renewed interest in *in situ*-TEM investigations of thermally activated processes. These miniaturized micro-heater devices have superior thermal stability and minimal specimen drift, which allows for the observation of dynamic processes at high spatial resolution [1]. Despite these advances, the reliable interpretation of results from *in situ* heating experiments in the TEM is still limited by difficulties in measuring the specimen temperature accurately under *operando* conditions. Moreover, the greatly reduced thermal mass of the specimens used in MEMS chip micro-heater experiments exacerbates this problem, and so an *in situ* measurement technique with both high temperature sensitivity and high spatial resolution is required. Previously, several different approaches have been considered such as; direct observations of well-defined isothermal transformations in standard samples [2], pyrometry-based measurements [1], and measurement of temperature-dependent shifts in spectrometric [3] or diffraction data [4]. Recently, Asoro et al. [5] investigated the sublimation kinetics of Ag nanoparticles during *in situ* heating. It was shown that the sublimation temperature was dependent on the nanoparticle size in a manner consistent with that expected on the basis of the Kelvin equation. Asoro et al. proposed that this could form the basis for a new method both to measure the specimen temperature during *in situ* heating experiments and to evaluate the effects of such factors as electron beam heating during such experiments.

In this study, we have explored the potential of this method to measure the temperature of the membrane on a MEMS-based heating chip. Monodisperse cuboidal polyvinyl pyrolidone (PVP) capped Ag nanocubes were used in systematic isothermal sublimation experiments, and the data (Figure 1) obtained were analyzed to measure the temperature across the surface of the micro-heater device and to investigate the various sources of error affecting the temperature measurements. It is shown that for an appropriate choice of experimental parameters (nanoparticle size, loading, ramp to the holding temperature, and illumination conditions) the sublimation of Ag nano-cubes can be used to measure the specimen temperature to an accuracy of ± 5 ˚C, over the range 700 – 850 ˚C. The measurements are reproducible from area to area on the same MEMS chip, and from chip to chip of the same type. The values of specimen temperature obtained are consistently lower than the calibrated MEMS heater plate temperatures, and it is shown that this cannot be explained on the basis of random errors in the experimental measurements or systematic errors in the materials parameters used in the Kelvin equation analysis. It is proposed that this is instead due to the low thermal conductivity of the electron-transparent amorphous silicon nitride support membrane on the chip.

References

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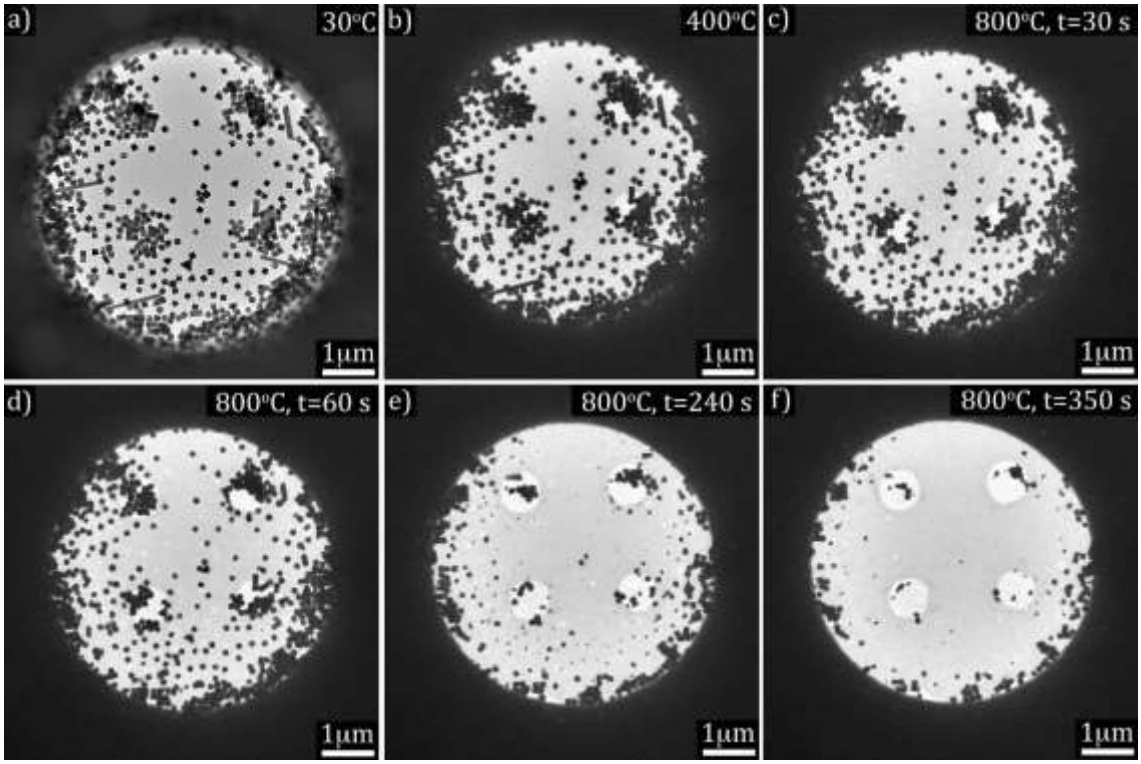

Figure 1: Sequence of BF TEM images obtained from an isothermal sublimation experiment performed at a set-point temperature of 800˚C with continuous exposure to the electron beam: (a) initial configuration of Ag NCs before the experiment, (b) during the intermediate hold at 400 ˚C, (c-f) during the hold at the set-point temperature.