Measuring the Temperature Homogeneity Across FIB Lamellae for In Situ TEM Experiments via Raman Scattering in Crystalline Silicon

Robert Krisper\(^1\), Harald Fitzek\(^1\), Evelin Fissithaler\(^1\), Werner Grogger\(^1\)

1. Graz Centre for Electron Microscopy, Steyrergasse 17, 8010 Graz, Austria
2. Institute of Electron Microscopy and Nanoanalysis, Graz University of Technology, Steyrergasse 17, 8010 Graz, Austria

Introduction

In all in situ heating experiments, an accurate reading of the applied temperature as driving force for effects like phase transitions or precipitation is the basis for retrieving interpretable and reliable results. For the MEMS heater chip manufactured by DENSiSolutions, a four-point probe resistive measurement of the heating spiral tunes the temperature with an accuracy of more than 95% \(^1\). However, as the thermal contact between heater chip and specimen is usually not well defined, methods to measure the actual sample temperature locally (e.g. in a TEM) are the focal point of ongoing research. These include recording alterations in parallel beam electron diffraction patterns of Au nanoparticles \(2\), plasmon peak shifts in EELS for Al nanoparticles \(3\) or applying Raman spectroscopy to Si particles \(1\).

Methods

Unlike distinct particles adhering to an electron transparent SiN membrane, a mounted FIB lamella is in better thermal contact with bulkier parts of the heater chip membrane. With this study, we take an approach towards measuring the temperature distribution across such a lamella via recording the shift of the Raman active LO mode in Si, figure 1.a, using the theoretical model suggested by Balkanski et al\(^4\).

We carried out the measurements using a Zeiss Sigma 300 SEM with an integrated WITec Raman confocal microscope, figure 2, to simulate vacuum conditions inside a TEM. The spectrometer laser features a wavelength of 532 nm and adjustable output power settings, which we set to 0.11 mW to obtain a reasonable signal while not excessively contributing to the heating of the chip (as seen with 1.0 mW). A DENSiSolutions Wildfire\(^{TM}\) heater chip in combination with a custom-built holder for contacting the former in SEM, FIB and AFM applications provided the primary temperature stimulus (figure 3).

Results

We recorded the Raman peak shift over a temperature series from 25 °C to 800 °C at 0.11 mW laser power and up to 1000 °C at 1.0 mW respectively, and fitted the data (figure 1.b) for calibration. The stability of the spectrometer, the quality of the fit and the small standard deviation obtained from time series measurements result in a statistical uncertainty of \(\Delta T = \pm 2°C\). However, systematical errors arise due to parameters which shift Raman peaks but are challenging to control during a heating experiment (mechanical stress within the lamella \(5\)) or during the manufacturing of the specimen (nanocrystallinity of the sample \(6\)). Figure 4.b therefore depicts the relative temperature homogeneity at a nominal temperature set point of 800 °C across a 6.2 μm long scan line in Si on a FIB lamella (figure 4.a) instead of an absolute temperature value.

Conclusion

- Repeatability of measurements and precision of the used WITec Raman microscope inside the Zeiss Sigma 300 VP allow for accurate temperature readings, based on Raman active mode shifts in Si.
- Temperature calibration is in good agreement with models for three-phonon interactions in Si at low laser power.
- Due to the way a FIB lamella is mounted on a heating chip, mechanical stress may corrupt absolute temperature measurements.
- A line scan across a FIB lamella reveals that temperatures in the center of the lamella are rather homogeneous, but 10 K to 20 K lower than close to the fixation points on the heater chip.

References


Contact

robert.krisper@felm-zfe.at
harald.fitzek@felm-zfe.at
www.felm-zfe.at