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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 DENSsolutions, 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_x 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 DENSsolutions WildfireTM 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).

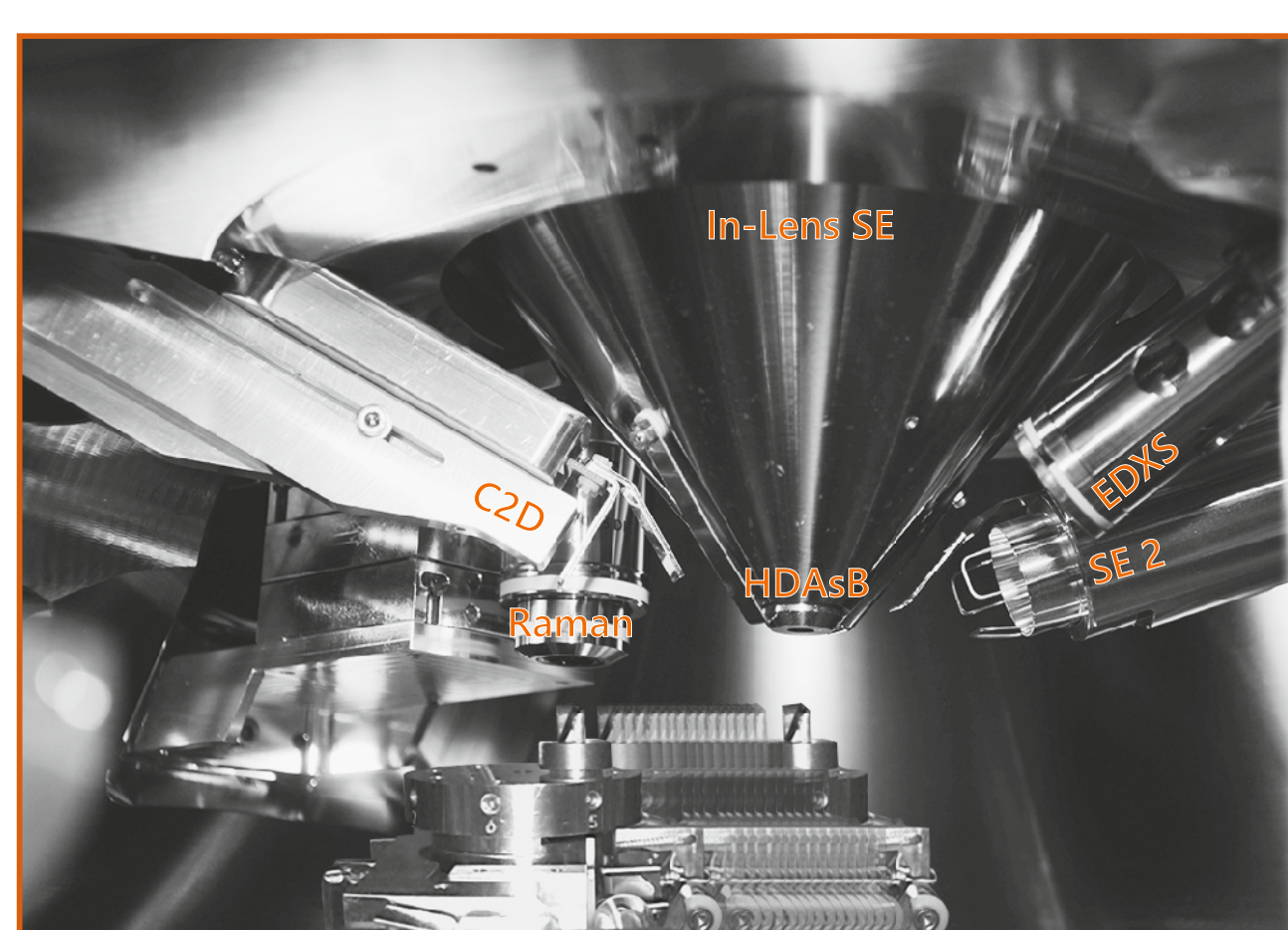


Figure 2. Specimen chamber of the Zeiss Sigma 300 VP (“variable pressure”) SEM installed at FELMI-ZFE, which features a WITec Raman confocal microscope alongside other detectors.

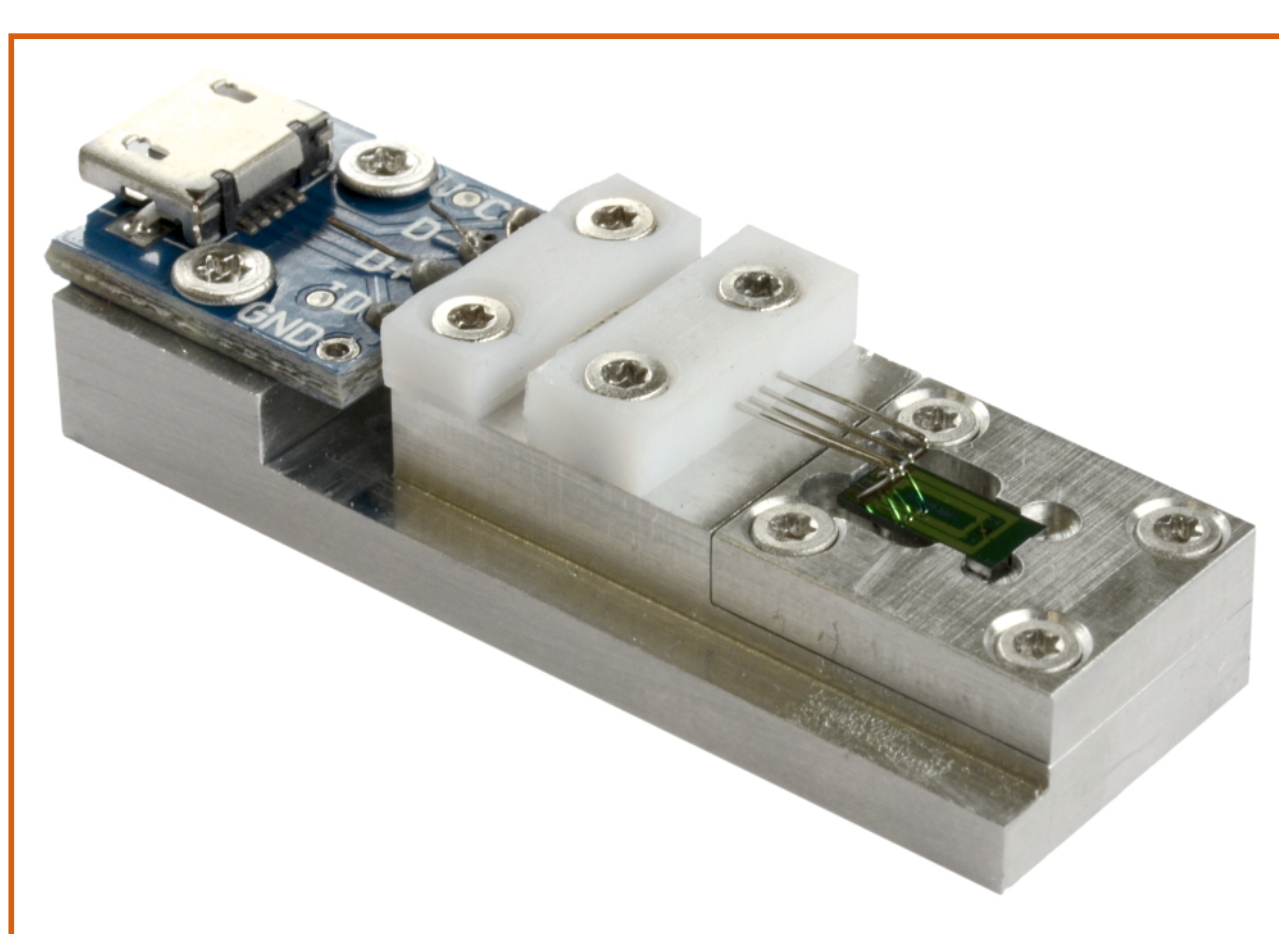


Figure 3. Custom built holder to use TEM heater chips in (Raman-)SEM, FEBID, and AFM applications and for correlative microscopy, here combined with a DENSsolutions WildfireTM S3 used in our experiments.

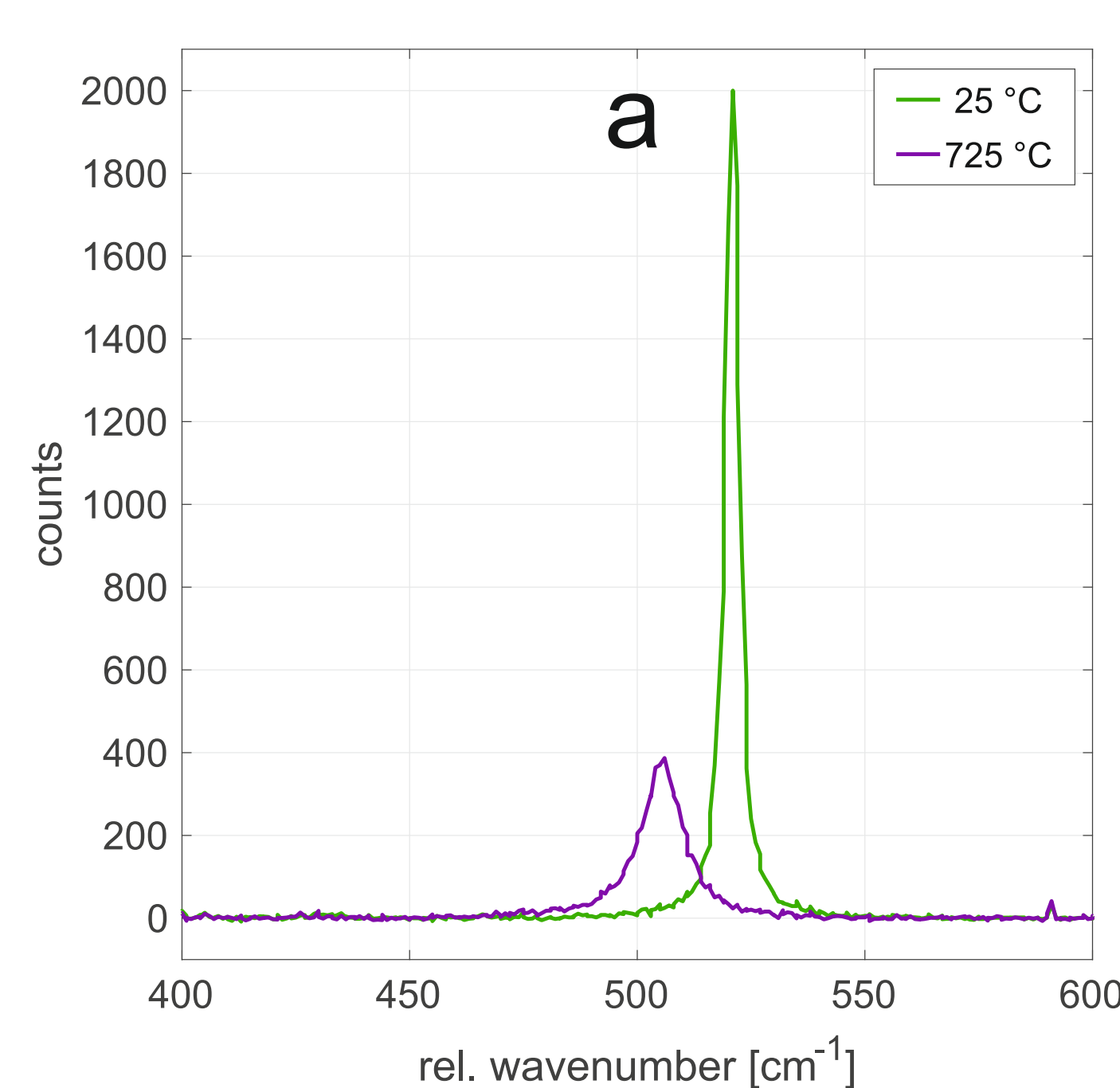
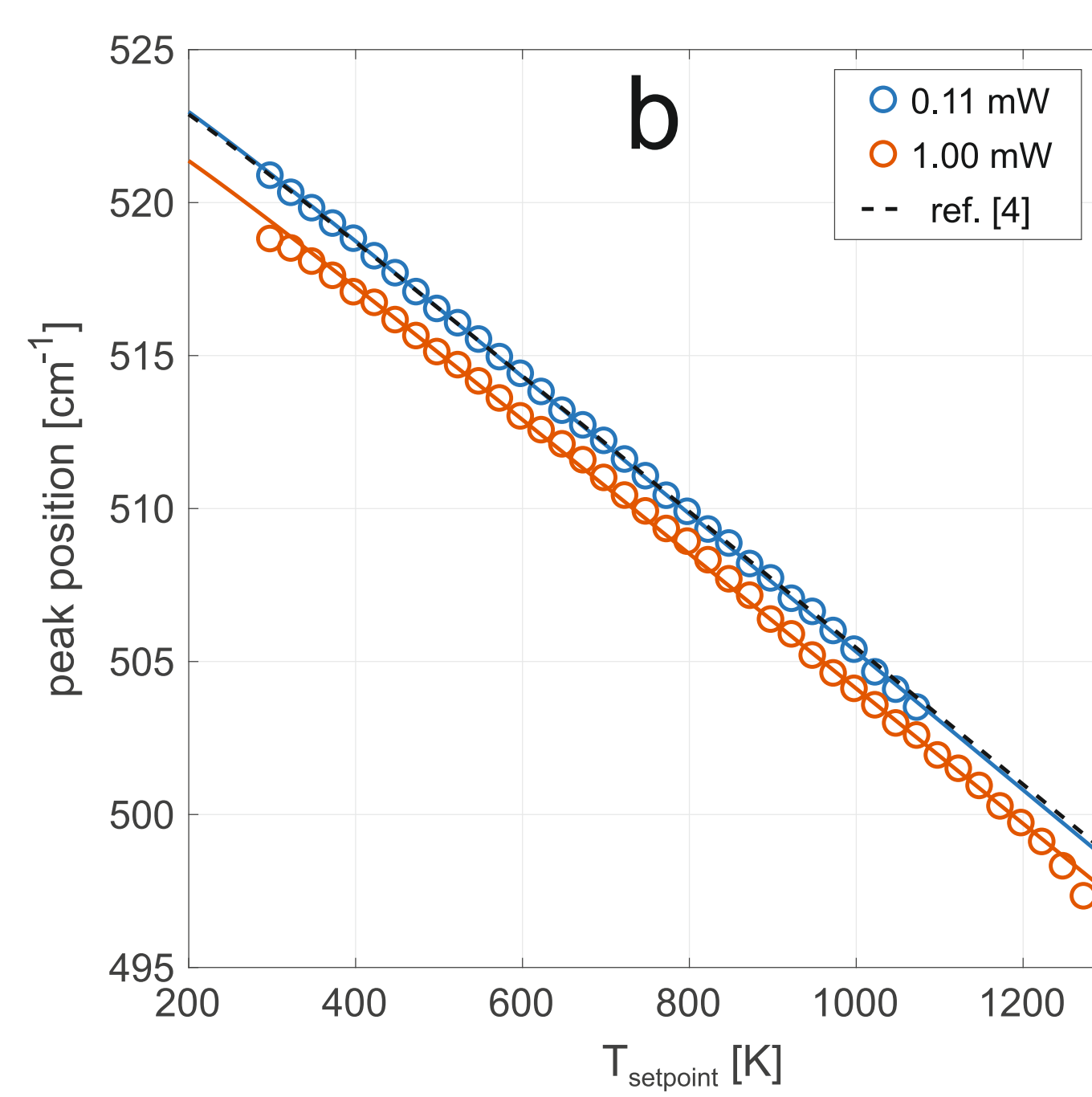


Figure 1. a) Measured shift of the Raman active LO mode in Si between 25 °C and 725 °C. **b)** Calibration measurements for different laser powers. The dashed line shows reference data from [4] for three-phonon scattering.



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, systematic 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.

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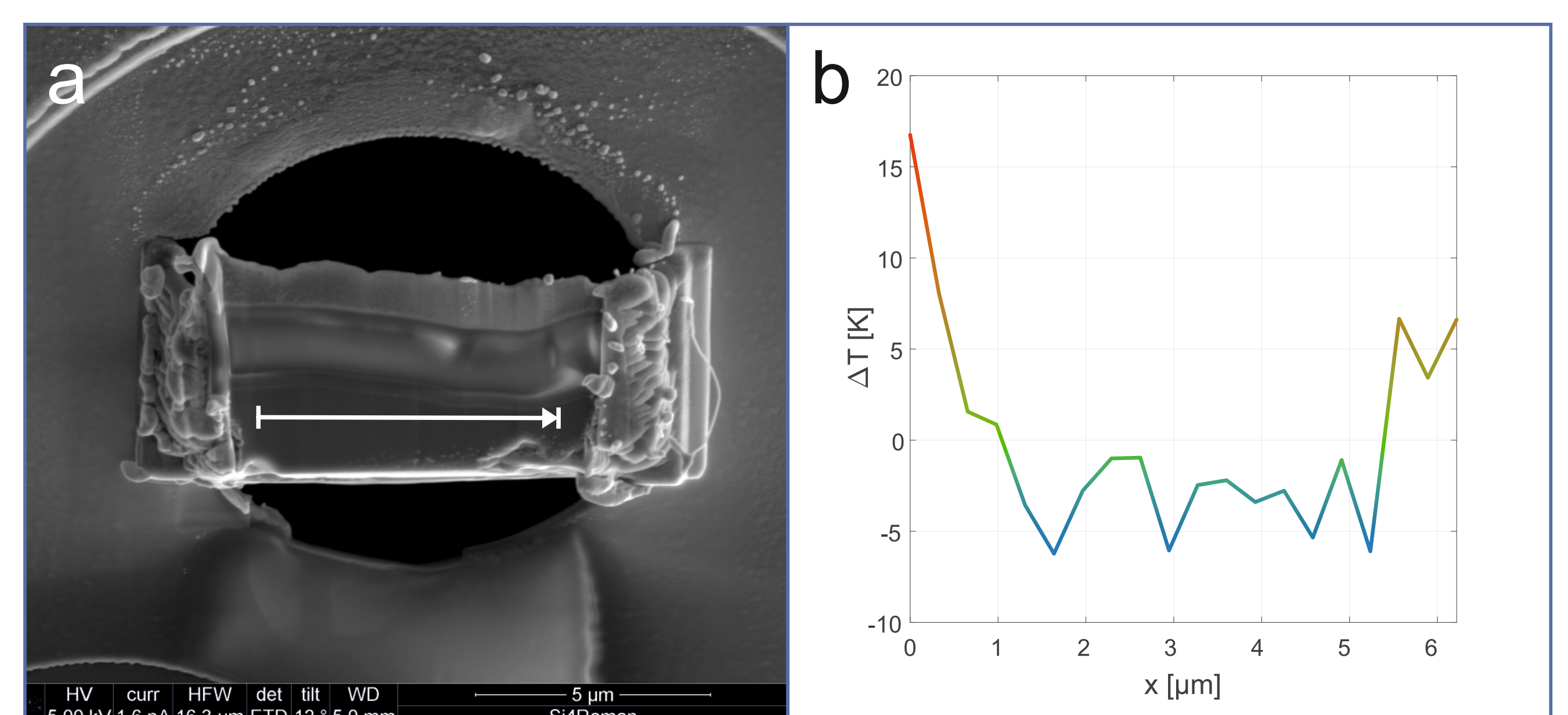


Figure 4. a) FIB lamella (Si, oxide layer, Pt) on a DENSsolutions WildfireTM S3 heater chip (tilted view) after measurements. Raman line scan in Si along the indicated distance. **b)** Temperature homogeneity across the FIB lamella based on the calibration model and the Raman mode shift at a nominal set point of 800 °C.

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

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