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# Assessing the Precision of Local Temperature Measurement by Plasmon Energy in In-Situ Heating Electron Microscopy

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## Background incl. aims

In-situ transmission electron microscopy (TEM) has become increasingly important in the characterization of microstructures in functional and structural materials when real-time observation under application conditions is required to reveal underlying structure-property relationships [1]. With the advancement of microelectromechanical systems (MEMS) technology for the application of stimuli on the materials, the high sample stability and precise control of the stimuli, especially in elevating temperature with a local accuracy within 4% [2], enable atomic-scale resolution in such in situ TEM experiments. However, uncertainty remains regarding the exact temperature profile across a TEM sample itself, which limits the interpretation of observed structural phenomena in in-situ TEM heating experiments.

## Methods

Here, we exploited plasmon energy expansion thermometry (PEET), detecting temperature-dependence of the bulk plasmon peak in an energy electron loss spectrum (EELS) [3] and testing its validity and accuracy using tungsten (W). The reasons to assign W as the model material come from the high melting temperature which is above 3000 °C and the sharp plasmon resonance in EELS, as shown in figure(a). These advantages benefit PEET through improved precise EELS peak determination over a wide implementation temperature range.

For sample preparation, W lamellas with varying sample thickness of 30 to 70 nm were prepared using a ThermoFischer Hydra Plasma FIB (Xe plasma at 30 kV) and were mounted on a DENS Wildfire heating chip [2] using W-gas assisted deposition. Two types of FIB-cut W lamellas were prepared: One is placed at the center of the spiral heater where we expect a homogeneous temperature distribution, as shown in figure(b). The other one is placed at an off-central position where we expect a thermal gradient [4].

Ex-situ temperature measurements were conducted using Raman spectroscopy with a 532 nm laser of beam size less than 1 µm. Si particles of 45 nm diameter were drop-casted on the heating chips. Exploiting the temperature dependence of the bonding vibration, the local temperature can be obtained by the Raman frequency of the Si-Si bond, which shifts with temperature at approximately 0.0232 cm-1 per °C.

For the in-situ heating experiments, both a ThermoFischer Themis (at EMAT) and Spectra (at DTU) S/TEM equipped with X-FEG mono have been operated at an accelerating voltage of 300 kV. For our PEET experiments, the monochromator was excited to achieve an energy resolution of 0.12eV. The effect of the STEM convergence semi-angle (3.6 to 18 mrad) as well as of the EELS collection semi-angle (6 to 70 mrad) was investigated in the PEET mapping experiments. The bulk plasmon peak was collected using Quantum 966 Gatan Image Filter with a dispersion of 0.01 eV per pixel and dwell time of 0.1 second per pixel.

## Results

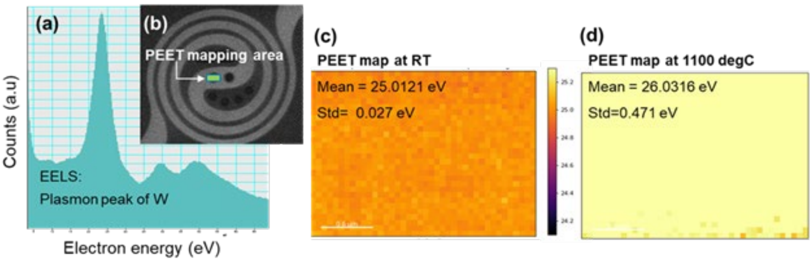
PEET mapping on the W lamella at the center of the spiral heater (figure(a)) reveals a temperature-dependent plasmon energy shift of 1.8 eV as the temperature progressively increases from room temperature (figure(c)) to 1100 °C (figure(d)). Correspondingly, the standard deviation when measuring at multiple positions also considerably increases, from 0.027 eV at room temperature (figure(c)) to 0.471 eV at 1100 °C (figure(d)), respectively. To understand this variation in plasmon peak energy, we attempt to correlate the PEET mapping and the local estimated sample thickness (using the log-ratio method in EELS) and find a noticeable correlation as one factor responsible for this large deviation.

Leveraging these findings as calibration data, we further extended the application of PEET for detecting the temperature difference over space. The profound temperature gradient of the off-central W lamella was predicted by COMSOL simulation and verified by ex-situ Raman measurements. The results show that the thermal gradient can reach approximately 106 °C /m (from 667 to 772 °C within a 10 µm length) at a set temperature of 1000 °C. Interestingly, on the PEET map of the off-central W lamella at the same set temperature of 1000 °C, the plasmon energy shifts to higher values as the position moves farther away from the spiral center. By using the calibration data for correlating the plasmon energy to the local temperature, the data from the PEET map closely matched the thermal gradient observed in the ex-situ Raman measurement and simulation results.

## Conclusion

Our study demonstrates that PEET is capable of obtaining quantitative temperature measurement results with the consideration of the sample thickness. Moreover, it effectively identifies pronounced thermal gradients within the sample. The improvement of the temperature determination in PEET provides a more reliable analysis of structure-properties correlation under various thermal conditions.

## Graphic:



## Keywords:

plasmon energy expansion thermometry

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