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Controlling the thermal gradient during in situ transmission electron microscope heating experiments

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Recent advances in in situ transmission electron microscopy (TEM) technology enable routine high-resolution observation of materials under conditions close to their application environment, thereby providing profound insights into the correlations between materials microstructures and properties at elevated temperatures. Nowadays, microelectromechanical system (MEMS)-type microheaters enable very accurate control of local temperature control during the in situ TEM experiment. So far, however, these experiments can only be carried out with a homogeneous temperature distribution over the area of interest.

Here, we report a method to introduce thermal gradients in in situ experiments, relevant, e.g. to study metal materials under far-from-equilibrium process conditions, by modifying a commercially available MEMS-based microheater. Using a focused ion beam (FIB), the sample is placed over a specially cut window not on top but near the heater [1].

COMSOL simulations are performed to study the effect of window geometry on the established thermal gradient and to tune and control the thermal gradients across the TEM sample. To verify the introduction of the thermal gradient on a Si FIB lamella, the Ag sublimation approach [2] is applied for profiling of the temperature across the specimen. The measured gradient was found to be ~6.3×10^6 K/m [3]. Further simulations indicate that the thermal gradients can reach up to ~10^7 K/m at the setting temperature of 1400 K. The good agreement between simulation and experiment encourages further detailed investigations of microstructures under controlled thermal gradients.

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