Application Note

Fusion™

Thermal Drift and Settle Times Measured for Different Temperature Excursions in the TEM

Introduction

Thermal drift and settle times during heating experiments in the electron microscope present a crucial problem for *in situ* imaging and analysis. When a traditional bulk heater is used during temperature ramp, the area of interest can drift significantly. This requires lower microscope magnifications to enable the operator to visualize the amount of drift and adjust the position of the stage to account for it. Even small temperature changes can cause areas of interest to quickly drift out of view, making it difficult to obtain images and analytical information from the same area.

The Fusion heating and electrical biasing platform is designed to significantly reduce thermal drift and settle time. Fusion uses semiconductor devices called E-chips, which have a monolithic ceramic membrane that acts as the heater and sample support. At just 700 x 700 µm, the membrane has a very small thermal budget, allowing for heating rates of 1,000 °C per ms and up to 1200 °C with cooling at roughly the same rate.

The membrane is composed of materials that have matched coefficients of thermal expansion (CTEs), which help minimize drift. Fusion's heating capability is attractive for *in situ* heating experiments where low drift and high stability is required for image acquisition and analytical analysis of the same areas at atomic resolution. The fast ramp rates and settle times enable the user to acquire data faster and more accurately, saving time without compromising data quality.

Experiment

To quantify the drift and settle time characteristics of Fusion, two sets of experiments were performed. In the first set, drift and settle time were measured as the temperature was stepped. Using a double tilt holder, the temperature was stepped from room temperature (RT) to 500 °C, RT to 1200 °C and 500 °C to 1000 °C at 40 kX magnification in a JEOL 2010F operating at 200 kV. This magnification was chosen so the area of interest would remain in the field of view over the entire temperature excursion.

The settle time is defined as the instant the temperature was applied to when the sample was observed to stop drifting. The following table shows the amount of drift and settle time measured for each temperature excursion. Figure 1 shows images at RT (top), and 350 °C (bottom). The top image was taken after the sample was observed to stop drifting at ~1 minute.

Table 1: Fusion displacement as a function of time and temperature

Temperature	Displacement	Settle Time
RT to 500 °C	180 nm	1 minute
RT to 1200 °C	1100 nm	2 minutes
500 °C to 1200	980 nm	1 minute

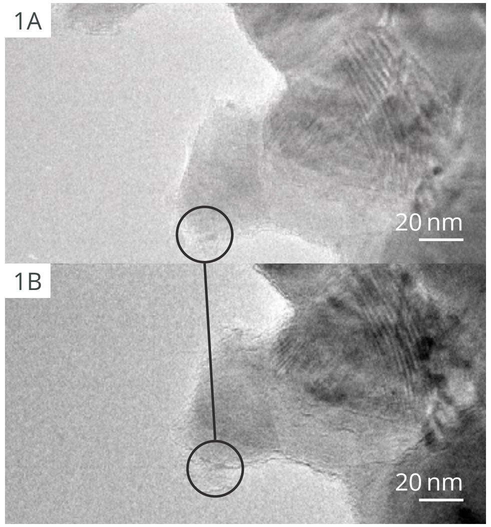

Figure 1A-B: A represents bright-field TEM image at RT. **B** represents bright-field TEM images at 350 °C

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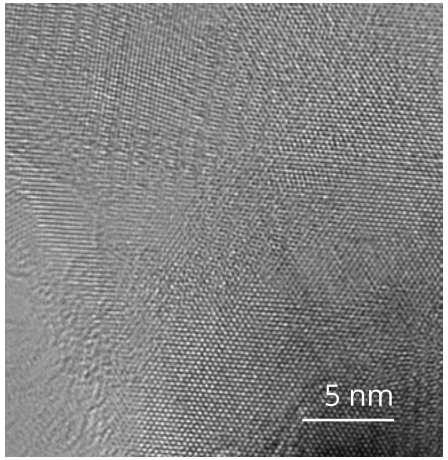
In the second set of experiments, HRTEM imaging capabilities of Fusion at elevated temperatures is illustrated. HRTM images were obtained while the temperature was ramped to 900 °C in a JEOL 2010F. upon instantaneous ramping to 350 °C, resolutions below 1.2 Å were consistently achieved. Figure 2 shows a high-resolution image immediately after a 4 °C/second ramp. The FFT of this image (inset) indicates 1.2 Å resolution.



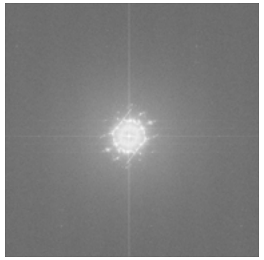


Figure 2: HRTEM image immediately after 4 °C/sec ramp

Discussion

Fusion's heating membrane can change temperature very rapidly (millisecond timescale). The rapid temperature change causes drift to primarily occur after the temperature change is completed. Figure 2 shows high resolution after temperature was instantly changed from RT to 350 °C.

These images were taken to show how quickly the heating membrane settles after a step in temperature. The sample drift is equal in magnitude and opposite in direction for heating and cooling. The same area of interest is imaged before and after the temperature excursion without changing the position of the microscope stage.

Fusion can maintain high-resolution while heating. In addition to stepping immediately to a specific temperature, Fusion is capable of ramping at user defined rates. If the ramp rate, or temperature jump, is kept at reasonable rates high-resolution is not compromised.

When the membrane heats, it expands. As it expands the membrane will bow, the 2D projection of bowing is reflected as drift. Sample type can also play a role in the drift and settle time characteristics.

Applications

Thermal drift and settle time is applicable to all heating experiments in the electron microscope. By minimizing drift and settle time during and after temperature changes, images and analytical information can be acquired faster and more accurately. Contact us to discuss the full range of capabilities of Fusion with Thermal E-chip™ sample supports. We can be reached at (919) 377-0800 or contact@protochips.com.

Reference: The temperature step experiments were done at Sandia National Laboratories in Livermore, CA. The temperature ramp experiments were done at the Karlsruhe Institute of Technology in Karlsruhe, Germany.