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Optimized High-Temperature *In-Situ* Transmission Electron Microscopy Double-Tilt Sample Heating Platform

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The most common method for controlling structure through processing is through simple thermal excursions, as temperature causes atomic diffusion, and thus allows structural re-ordering. The primary experimental method used to determine the local internal structure of materials is that of transmission electron microscopy (TEM). Therefore, the most common dynamic *in situ* microscopy experiments revolve around the relatively simple act of heating a sample.

Several *in-situ* sample heating TEM holders have been available over the last decades [1]. Thin-film technology has allowed much more local heating of the specimen in the TEM, enabling more stable imaging during *in-situ* TEM heating experiments. More recently direct temperature measurements on these thin-film heating systems have made temperature measurements at the sample become closer to becoming reality [2]. Although double-tilt TEM heating holders have been available, they suffer from some of the same limitations as standard double-tilt TEM holders do. Specifically, backlash in the tilting mechanism and lack of repeatability of the tilt makes it difficult to get the sample in exactly the right orientation and to know the exact angle of rotation. In this work we will use an optimized double-tilt thin film heating platform that minimizes mechanical artefacts in the tilting mechanism and provides very stable image performance when heated to temperatures >1000°C. Outside of standard temperature characterization of these thin-film heating TEM systems using high resolution optical thermal imaging, we also used melting standards to confirm accurate temperature response of the on-chip temperature sensor to indicate the temperature at the sample. We can identify very local and discrete melting events and correlate this to the sensor response (Figure 1), where local melting happens at very discrete moments (typically within a few frames), and surface diffusion occurs in matters of seconds following that.

In addition to this, we used model-systems to evaluate the *in-situ* TEM sample heating system performance and studied the behavior of gold nanoparticles and nanorods on SiN_x substrates. Starting with characterizing of particle motion and stability under the beam and then the phase transformation and (rapid) shape change at the melting point. Figure 2 shows a rapid frame-to-frame phase transformation process of gold nanorods as we ramp the temperature to 1000°C and show that we can use the system to track phenomena in nanostructures over the full high temperature range of the TEM heating system with good image stability. These results illustrate the optimized overall mechanical stability and temperature performance of this *in-situ* TEM double-tilt heating platform.

References:

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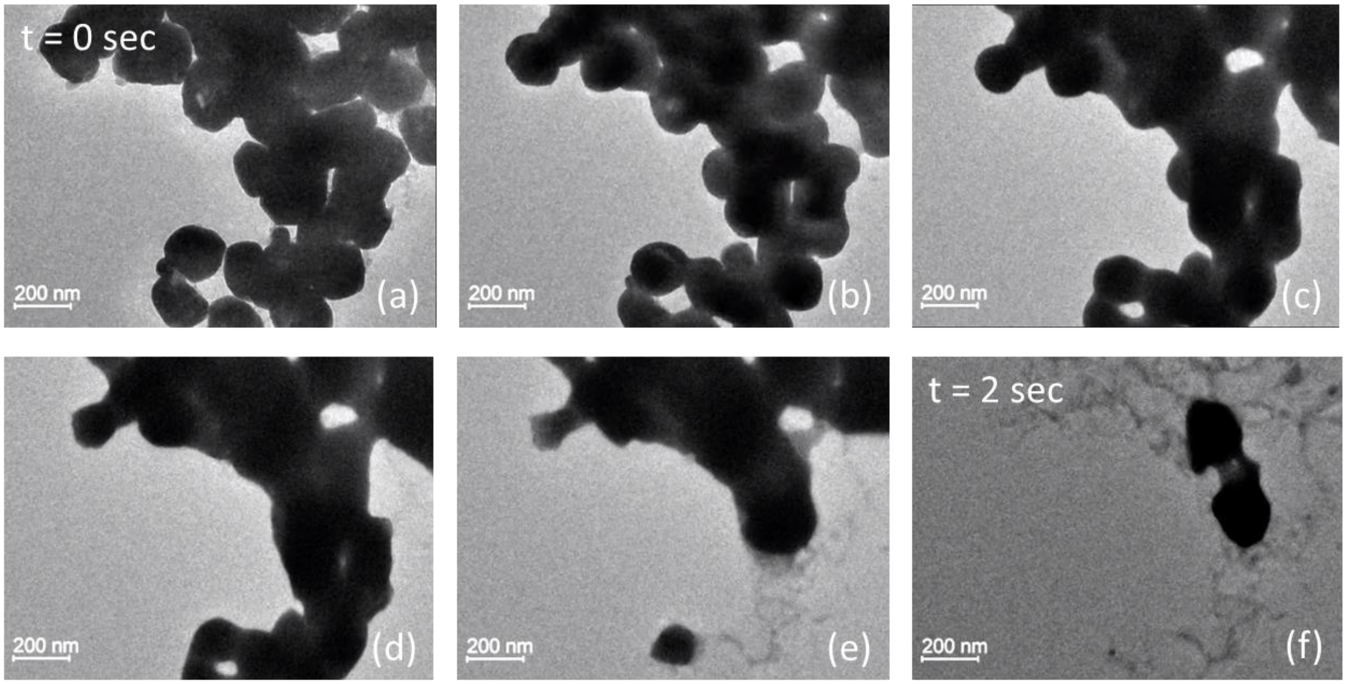

Figure 1. (a-f) Melting-event of silver nanoparticles at ~950°C; ~200nm nanoparticles with a known melting point were melt locally on the electron-transparent membrane of the sample support membrane to confirm sensor temperature response in the *in-situ* TEM heating system. Note that although the process of melting and diffusion is fast, it is still possible to observe the several stages in this process.

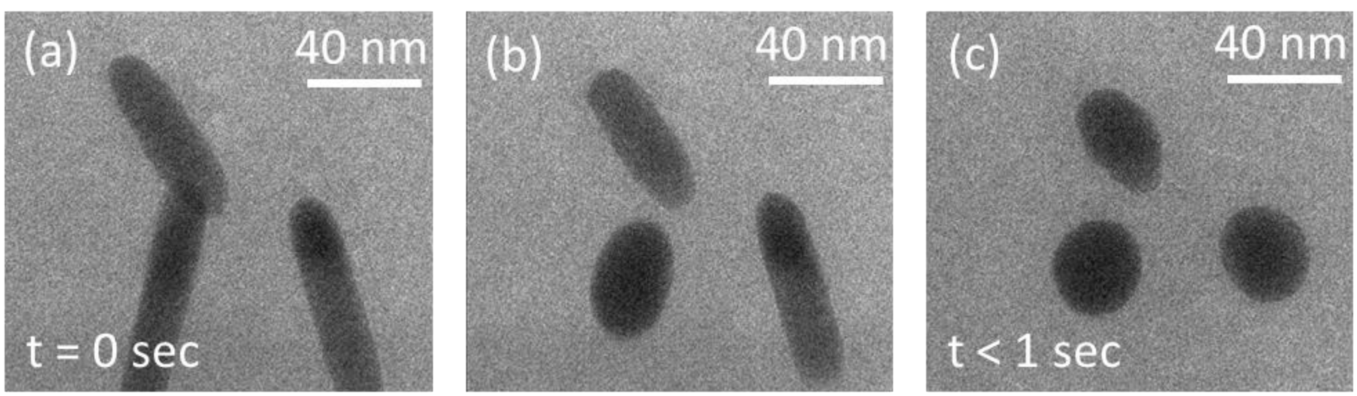

Figure 2. (a-c) High temperature phase-transformation event in gold nanorods during temperature ramp showing fast shape change within a few frames, as well as the tracking ability in the high temperature range during an *in-situ* TEM temperature ramp.