Recent trends in in-situ heating in SEM and FIB/SEM systems.

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Significant innovations in the area of sample heating have opened up new application possibilities and improved ease of use of the in-situ SEM. Scientists now have the unique opportunity to choose a heating module to perfectly match the sample type and experiment design and hence to significantly improve the quality of acquired data or to achieve unique results that were unreachable in the past. We will present in-situ experiments in the SEM and FIB/SEM using several types of heating stages under gaseous or high vacuum environment. Processes taking place at high temperatures will be characterized by means of secondary (SE), backscattered (BSE) and transmitted electrons. Energy dispersive spectroscopy (EDS) and electrons backscatter diffraction (EBSD) will be employed to monitor heat induced changes in elemental composition and crystallography.

Exploring the dynamic response of a sample to the applied temperature under a gaseous environment in the ESEM is a method that has proven in the past decades. Its main advantage is a homogeneous distribution of gas in the specimen chamber and hence around the sample allowing studying reactions such as oxidation or reduction using water vapor or forming gas (H_2/N_2). Another approach is to inject a reactive gas into the proximity of the area of interest while the chamber stays in high vacuum. This is possible with the newly developed High Vacuum Heating Stage. The module ensures cleanliness of the experiment and benefits from using standard detectors including in-lens. Moreover, its design enables one to tilt up to 70° for in-situ phase transformation and recrystallization studies by EBSD as illustrated in Figure 1. Further extension of the application space towards rapid changes of temperature, full detector compatibility and ultra-high resolution imaging requires change of the heater technology. The micro heating plate device called Heater based on MEMS technology provides extraordinary heating rates in the order of 10^4 kelvin per second with a maximal temperature of 1200°C. Typical Heater samples are either particles with a size ranging from nanometers to tens of microns or FIB prepared blocks transferred from a bulk sample to the heated area in-situ in the FIB/SEM. Thanks to the Heater design, STEM imaging (Figure 2) and EDS analysis of heated samples with minimized influence of thermal radiation become possible on top of SE, BSE and EBSD.

In conclusion, the application potential of in-situ SEM has been significantly broadened by the development of new types of heating stages. Choosing the appropriate heating module with respect to the sample type and design of the experiment significantly improves the quality of experimental data. Capabilities in signal detection and ultra-high resolution imaging have been greatly increased. Last but not least, the integration of all heating modules into the SEM User Interface sharing identical control elements and philosophy has very much improved the ease of use of in-situ SEM.

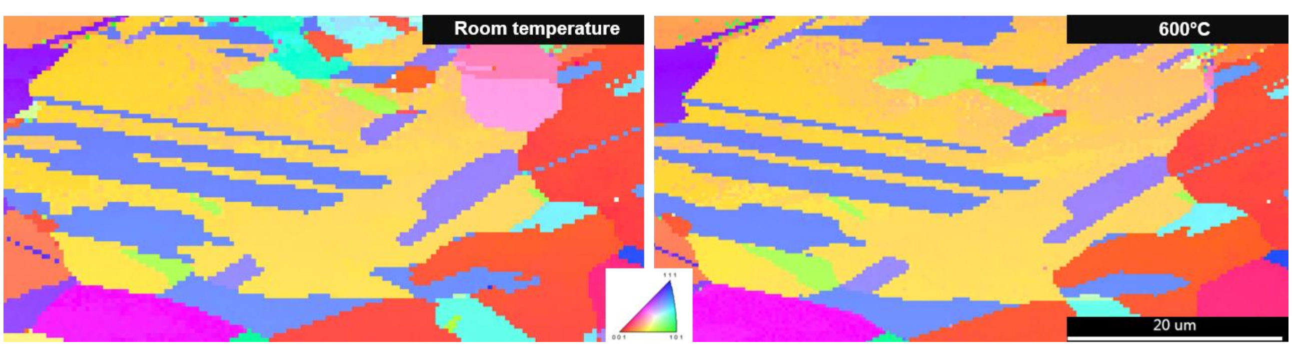

Figure 1. IPF orientation map showing recrystallization of copper when heating from room temperature to 600°C.

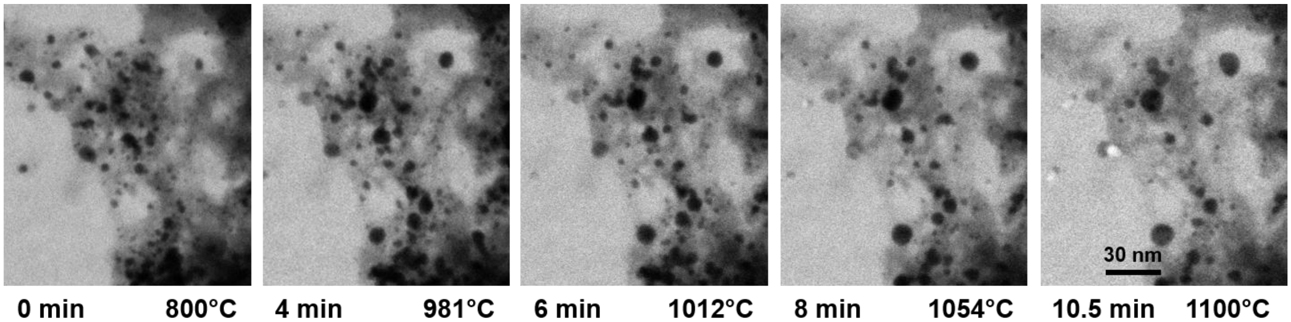

Figure 2. Bright-field STEM images of a coalescence of Pd nanoparticles on carbon while heating from 800 to 1100°C by the Heater.

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