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High-Temperature BSE and EBAC Electronics for ESEM

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Observation of microscopic surface dynamics in SEM plays an essential role in the study of functional materials, however instrumentation requirements for such experiments are very challenging because of the combination of high temperature and environmental conditions. For example, characterization of collective atomic motion in gas-solid interactions requires temperatures of up to 1,000 °C and injections of gases [1,2]. FEG ESEM instruments provide a good platform for such experiments, however the common SEM detectors are not compatible with such demanding requirements. This work presents a novel BackScattered Electron (BSE) detector, as well as novel Electron Beam Absorbed Current (EBAC) electronics developed specially for high temperatures and reactive atmospheres.

Standard BSE detectors are not compatible with high temperature because they are sensitive to the infrared light emitted by the hot sample, which masks the signal induced by the BSE electrons. A suitable light-blind alternative is provided by replacing the sensing elements from semiconductor diodes to conductive absorption pads, and thus switching from induced to absorbed electron detection (AED). Semiconductor diodes are preferred at room temperature, because they provide fast intrinsic amplification. This advantage is lost with absorption pads, and thus the electronics design must be optimized for much lower signals, which results in a slower minimum dwell time in the range of a few µs/pixel. For this purpose, a double-sided ceramic board was designed to provide the absorption pads and AED electronics, respectively.

As the removal of diodes allows for greater flexibility in layout, a four-quadrant (4Q) detector geometry was readily designed and manufactured, showing sufficient performance for operation under normal electron beam currents. The 4Q BSE also provides the necessary simultaneous signals for quantitative topographic reconstruction [3], and thus the hot surfaces can be inspected in a 3D view for the first time. A by-product of the AED electronics is excellent sensitivity to very low energy electrons, in principle down to a few eV, much exceeding the performance of room-temperature BSE detectors in this regard.

A further limitation is imposed by the dynamic insertion of gasses into the ESEM, which itself alters the cascade process that is relevant for the signal of the Large Field Detector (LFD). It thus become unclear if a change of LFD signal originates from changes on the sample surface, or changes of the imaging gas and detector response. A suitable alternative to LFD is provided by EBAC, which provides a signal independent from gas composition and pressure. EBAC requires electrical connection to the sample, which is provided by the thermocouple used for temperature measurements. A combined *in situ* board was thus designed and manufactured, combining temperature and EBAC measurements. As expected, EBAC measurements at high temperature show spatial resolution similar to that of in-lens detectors at room temperature, whilst independent of working distance and acceleration voltage.

Additionally, a time-series acquisition plugin has been developed for the DISS5 software to allow for acquisition of dynamic experiments and assembly of 4D animations of high temperature experiments.

References:

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(a) (b) (c) (d) (e) (f)

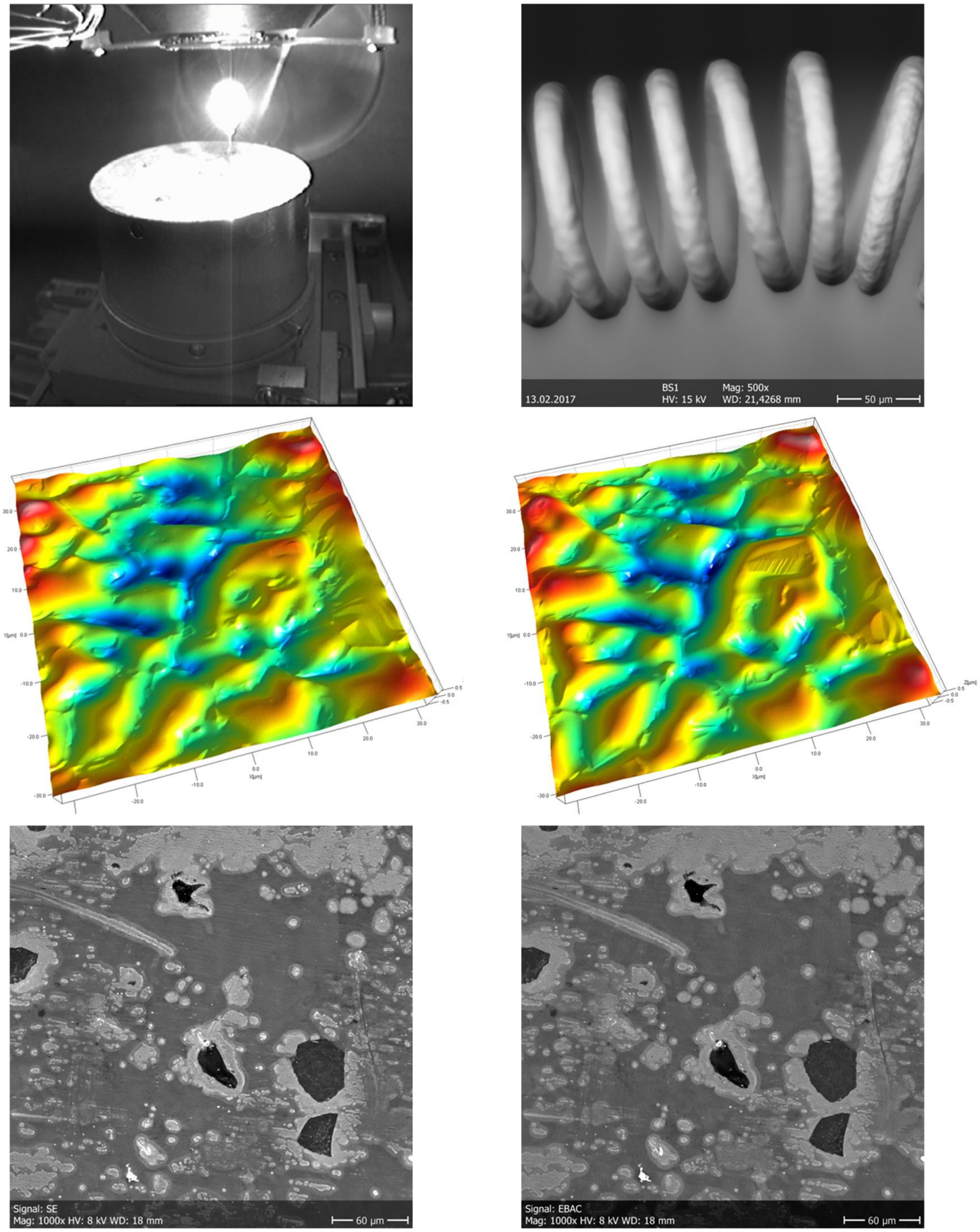

Figure 1. IR chamberscope view of heated W filament **(a)** and corresponding HT BSE image **(b)**. Extracts from a time series topographic reconstruction of Cu catalyst during hydrogen oxidation **(c, d)**. Large field detector **(e)** and Electron Beam Absorbed images **(f)** of graphene on Pt at 400 °C high temperature.

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