High-Temperature BSE and EBAC electronics for ESEM

Wolfgang Joachimi [1], **Matthias Hemmleb** [1], **Uwe Grauel** [1], **Zhu-Jun Wang** [2], **Marc Willinger** [2,3] **and Grigore Moldovan** [1]

[1] point electronic GmbH, Weinberg Campus, Halle (Saale), Saxony-Anhalt, Germany
[2] Department of Inorganic Chemistry, Fritz Haber Institute of the Max Planck Society, Berlin, Germany
[3] Scientific Center for Optical and Electron Microscopy - ScopeM, ETH Zürich, Zürich, Switzerland

Abstract

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. 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.

Technology

A first limitation to electron detection is imposed by the high temperature conditions, which leads to emission of infrared light from the hot sample. A suitable light-blind solution is provided by replacing the diode sensors with conductive pads, and thus switching from induced to Absorbed Electron Detection (AED).

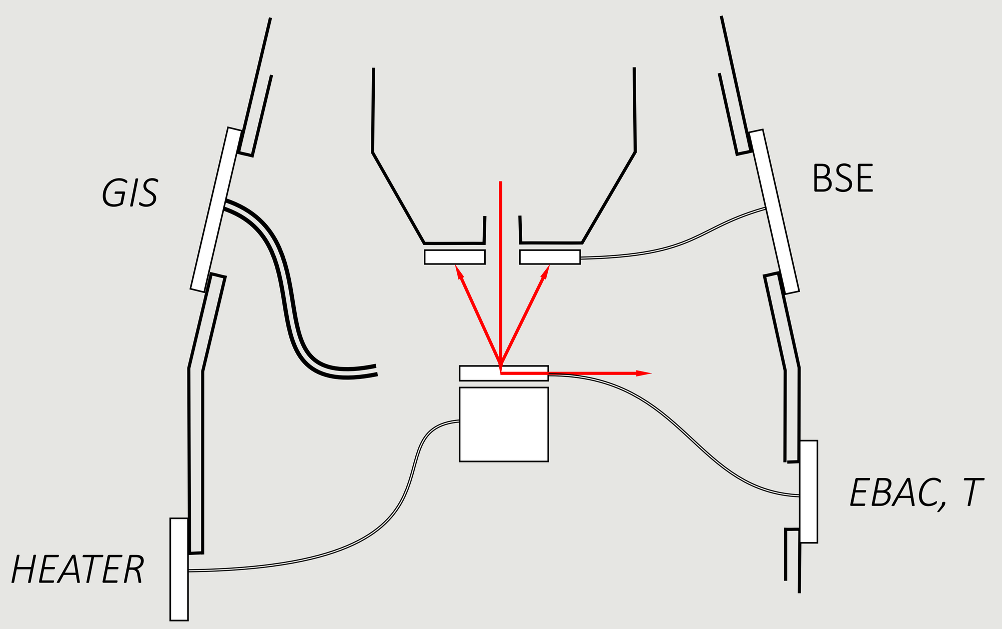

Figure 1: Diagram of HT ESEM set-up illustrating use of BackScattered Electron (BSE) and Electron Beam Absorbed Current (EBAC) signals

A second limitation to electron detection is imposed by the dynamic insertion of gasses into the ESEM, which itself alters the cascade process of Large Field Detectors (LFD). It thus become unclear if a change of LFD signal originates from changes on the sample surface, or changes of the imaging gas. A suitable alternative is provided by Electron Beam Absorbed Current (EBAC) imaging.

High-Temperature BSE

AED must amplify BSE signals in the range of 100fA, and thus the electronics must be optimized for high gain with low noise. BSE also requires imaging speed in the range of 10µs/pixel. These requirements are satisfied with *in situ* pre-amplifications on a double-sided ceramic board, which also provides the absorption pads.

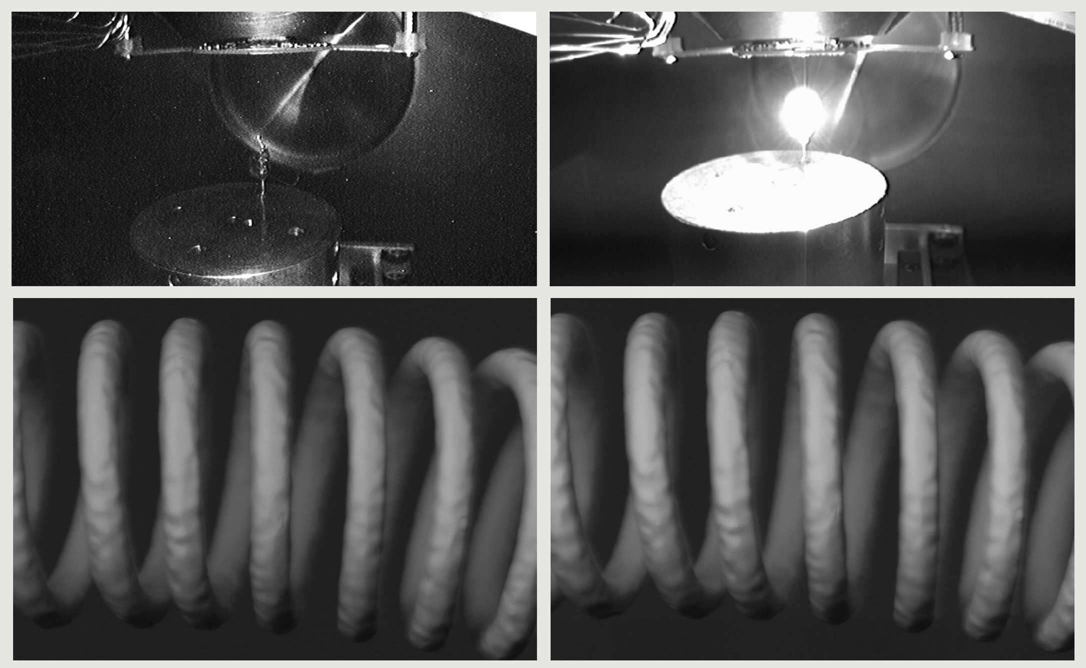

Figure 2: IR chamberscope views and BSE images of cold and heated W filament

High-Temperature SEM Topography

A four-quadrant (4Q) BSE geometry was designed, showing sufficient performance for operation under normal conditions. 4Q provides the necessary signals for topographic reconstruction, and thus hot surfaces can now be inspected in 3D for the first time.

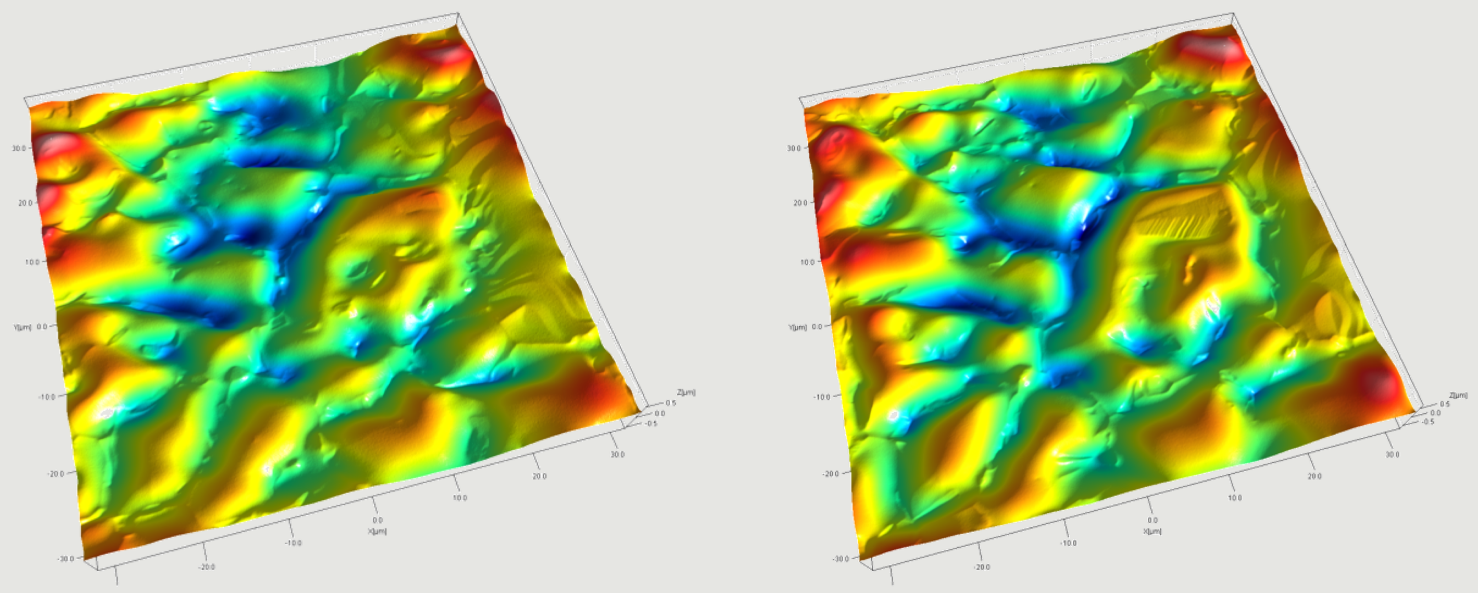

Figure 3: Extracts from a time series topographic reconstruction of Cu catalyst during hydrogen oxidation

High-Temperature EBAC

A custom solution has been designed to amplify the EBAC signal and measure temperature using the thermocouple connection to the heated sample.

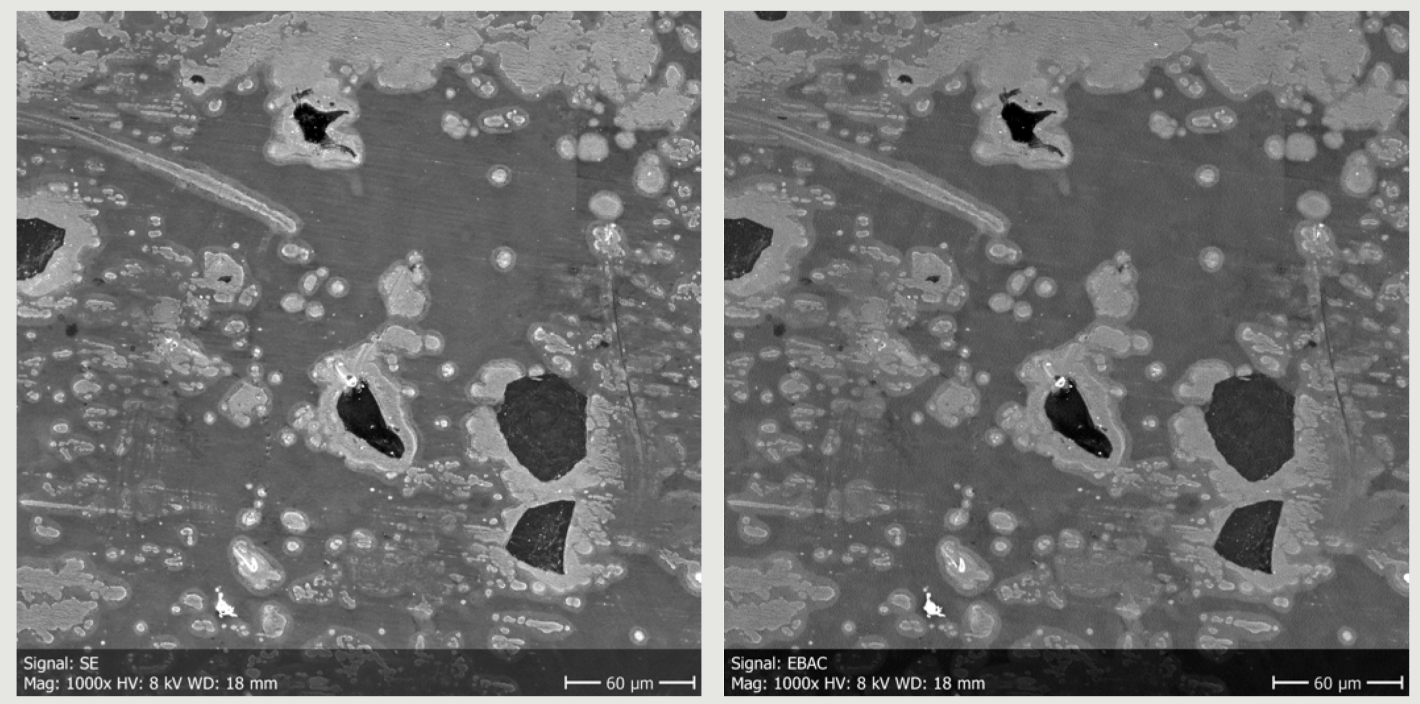

Figure 4: Simultaneous Large field detector (left) and Electron Beam Absorbed images (right) of graphene on Pt at 400 °C.

The *in situ* electronics have two calibrated outputs

- Temperature, as measured from the thermocouple
- EBAC, as collected from the sample

By comparison with LFD, EBAC signal collection is independent of species and pressure of the environmental gas, whilst retaining the same high resolution. In addition, EBAC provides a quantitative signal, and thus image intensity can now be used for analysis of surface chemistry and layer thickness.

References

- Z.-J. Wang et al., ACS Nano 9 (2015), p. 1506
- S. Poitel et al., ECS Trans. 78, 1, (2017), p. 1615-1632
- M. Hemmleb et al., EMC Proc. (2016), p. 489-490

point electronic GmbH | Erich-Neuß-Weg 15 | 06120 Halle (Saale) | Germany
Tel. +49 345 1201190 | Fax. +49 345 1201223 | info@pointelectronic.de | www.pointelectronic.com