SEM Insights: Sample Temperature Evolution during EBSD

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Background incl. aims
Electron Backscatter Diffraction (EBSD), with application in geology, ceramics, semiconductor, and metals research, enables a comprehensive analysis of the microstructure in materials. By mapping crystallographic orientation it aids in understanding phenomena, such as character of grain boundaries, etc., for the investigation of crystal/grain growth, phase transformations, as well as the detailed analysis of material deformations and strain.

Despite its widespread use, still many challenges remain, such as the effect of sample surface roughness, sample preparation artifacts, sample drift during data acquisition, image distortions due to the sample positioning and drift, effect of surface contaminations, as well as simply the complexities in EBSD data interpretation. This is stimulating ongoing research and requests further improvements.

Furthermore, potential effects of the electron beam itself, particularly beam-induced heating, remain an unexplored concern . Such effects could affect the microstructure of examined samples or lead to inaccuracies in assumed temperature conditions during experiments, in particular in in-situ heating experiments. Here, we aim to investigate these effects to better understand their impact on samples and discuss potential associated drawbacks.

Methods
In-situ heating holders based on micro-electro-mechanical systems (MEMS) were initially developed for stable and accurate transmission electron microscopy (TEM) investigations, but have since also found application in in-situ SEM experiments. Equipped with a four-point measurement system on their heating spiral, these holders provide real-time temperature feedback based on the measured resistivity of the heating spiral material, enabling precise temperature control during experiments as well as a direct feedback on additional sample heating effects.

With this approach, in our study we want to investigate the effects of beam exposure on sample temperature of various materials, as well as significant data acquisition parameters (i.e. SEM acceleration voltage, beam current, dwell times) as they occur during EBSD measurements of bulk-like samples. By utilizing COMSOL, a finite element analysis tool, we simulate the Joule Heating of the MEMS device to predict the temperature distribution in our sample caused by the Electron Beam, thereby improving our understanding of the complex electron beam-sample interaction.

Results
Our study provides insights into the evolution of sample temperature under EBSD-relevant microscope conditions. We observed an increase in the sample temperature of approximately 40K in dependency of the acceleration voltage used for an Iron Sample. These findings we compare with our COMSOL modeling results.
Thereby, we will emphasize the significance of temperature effects, particularly in critical cases like beam-sensitive samples, where beam-induced sample heating could affect interpretation of microstructural analysis results.

Additionally, we highlight the effects of beam-induced heat during in-situ heating experiments in SEM as well as the importance of incorporating an active temperature control feedback loop in such setups.

Conclusion

Here we present a novel approach to real-time temperature monitoring of samples, particularly under EBSD-relevant measurement conditions. This will provide important insights into the dynamic thermal behavior of specimens during examination in a scanning electron microscope

Keywords:

SEM, EBSD, Beam Heating