White Paper

Electron backscatter diffraction (EBSD)

of nonconductive samples using in-situ charge compensation

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Electron backscatter diffraction (EBSD) is a powerful technique for structural characterization of micro or nano-crystalline samples in the scanning electron microscope (SEM). Reliable EBSD analysis of nonconductive samples so far required the use of conductive coatings or variable pressure (VP) SEM. The in-situ charge compensation (CC) system available with ZEISS SEM and CrossBeam instruments provides improved EBSD analysis of nonconductive samples without the drawbacks of these conventional methods.

EBSD of nonconductive samples

Diffraction patterns formed by electrons backscattered from a crystalline material in an SEM can be recorded with a dedicated EBSD detector that consists of a scintillator screen, a CCD camera and coupling optics. Characteristic dark and bright line pairs in the recorded diffraction patterns, so-called Kikuchi bands, relate to phase and orientation of the underlying crystal [1]. The analysis of these bands in multiple measuring points provides crystallographic orientation mapping, grain size mapping, grain boundary characterization, phase discrimination and distribution, and inter-phase orientation relationships with high lateral resolution in 2D, or, if combined with serial sectioning using a focused ion beam (FIB), in 3D [2]. Using in-situ sample modification techniques, EBSD can even be used to investigate the microstructural evolution of the sample under high temperature or mechanical strain.

Raw EBSD patterns usually do not show strong enough diffraction contrast to reliably perform Kikuchi band detection and indexing. They require extensive pre-processing, such as background removal and automatic pattern-by-pattern contrast optimization. Sophisticated and fast algorithms are available for pre-processing as well as the pattern analysis itself, but the quality of the raw data is fundamentally limiting the analysis result.

Nonconductive samples such as ceramics, polymers, and most biological samples typically charge up negatively during EBSD measurements. Accumulated surface charges act as a retarding field for the beam electrons. They will also deflect the backscattered electrons and, thus, degrade and shift the diffraction pattern. Without any countermeasures to mitigate sample charging, insulators do not provide sufficiently good diffraction patterns for consistent and reliable indexing.

In conventional SEMs, EBSD of nonconductive materials can only be done after deposition of a conductive coating of, e.g., a few nm of carbon or gold. However, it is difficult to adjust the correct coating thickness. If the layer is too thick, the signal to noise ratio of the diffraction patterns will decrease significantly, especially if low acceleration voltages are required. If it is too thin, the charge will not be sufficiently dissipated. Both will result in poor EBSD results.

In a VP-SEM, EBSD analysis of nonconductive samples can be done under low vacuum conditions. Here, the SEM chamber is flooded with water vapor or nitrogen. Electrons leaving the sample surface collide with gas molecules and ionise them. Positive ions are attracted to the negatively charged sample surface and balance out the charge. However, electrons produced during the ionization will interact with more gas molecules, so that a cascade of charged particles is produced. Collisions of the beam electrons with these particles in a large interaction volume will broaden the beam ('skirt effect'), and collisions of the electrons backscattered from the sample will degrade the diffraction pattern. Vapor pres-

sure, acceleration voltage, beam current and dwell times have to be iteratively adjusted on a given sample to balance the pattern degradation by low vacuum and the pattern improvement by surface charge reduction [3].

Both methods described above allow EBSD analysis of nonconductive samples only at the expense of decreased measurement sensitivity. Furthermore, both require tedious and time-consuming optimization of experimental parameters in order to achieve best results.

Solution

ZEISS FE-SEM and CrossBeam systems can be equipped with a gas injection system to provide a highly localized, adjustable nitrogen gas flow onto the sample surface. While the mechanism of charge neutralisation by gas ionisation is the same as in a VP-SEM, the low vacuum is limited to a small volume around the region of interest. Thus, detrimental interaction of beam and backscattered electrons with gas particles is drastically reduced compared to a VP system.
Since the rest of the chamber remains close to high vacuum conditions, no special VP detectors are needed. Image quality and resolution remain largely unchanged [4], and high vacuum conditions are restored within seconds after closing the gas flow. Pneumatic insertion and retraction of the device, as well as the gas flow are conveniently controlled from the microscopes software.

This unique CC system not only allows SEM imaging, but also EBSD analysis of nonconductive samples. Diffraction pattern degradation due to electron scattering in the gas is minimal and by far outweighed by the improvement in diffraction contrast. The CCs' standard gas injection nozzle can easily be replaced by one that is suitably shaped to be used with the very high specimen tilt angles necessary for EBSD.

Application example

Silicon nitrides have a high technological relevance as engineering ceramics for engine parts and bearings, high temperature parts and cutting tools, as well as in semiconductor technology for isolation and passivation. Textured, hexagonal β-Si_3N_4 is an excellent test specimen for our purpose, as it has a strongly anisotropic microstructure with highly oriented grains and a very high dielectric constant.

EBSD grain orientation maps were recorded from selected regions of such a sample with the CC gas flow switched on and off, but under otherwise identical conditions. The FE-SEM used was equipped with an Oxford Instruments Nordlys EBSD camera. Figure 1 shows a view of the SEM chamber

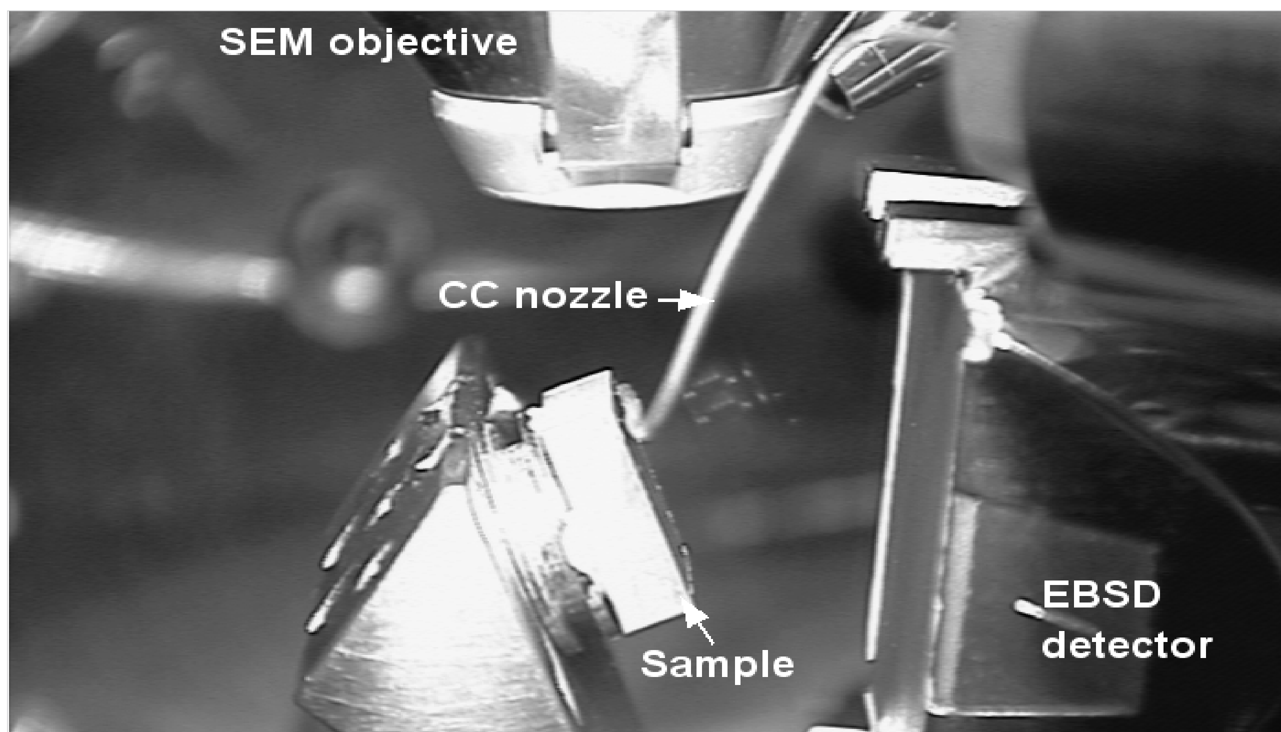

Fig 1 - *Photograph of the experimental setup inside the SEM chamber.*

with the experimental setup. The sample was tilted 70° to the electron beam, with the EBSD detector placed at a very short distance. The CC nozzle used had an S-shape to accommodate the high sample tilt and to avoid shadowing of the diffraction patterns towards the detector. The measurements were made at 5, 15 and 20 kV acceleration voltage, approximately 1500 pA beam current, a working distance of 11.7 mm, and a pixel dwell time of 100 ns. For data acquisition and processing, the Oxford Instruments Flamenco software was used.

In Figure 2, diffraction patterns recorded at 15 kV acceleration voltage from the sample with and without CC are compared. The pattern recorded with CC clearly shows higher contrast and better defined Kikuchi lines over a larger angular range than the pattern recorded without CC. With CC on, the experimental pattern is more consistent with the theoretical pattern.

The improvement achievable with CC becomes even more obvious when comparing grain orientation maps acquired with and without CC. SEM micrographs with overlayed EBSD orientation maps from selected regions are shown in Figures 3 (15 kV) and 4 (20 kV). Except for a few measurement points, the software was not able to find indexing solutions in the patterns recorded without CC. However, consistent orientation maps were obtained from the patterns recorded with CC.

Finally, Figure 5 presents an orientation map recorded at an acceleration voltage of only 5 kV using the CC. Due to the much smaller electron-sample interaction volume at this low voltage, this map shows a particularly high spatial resolution. It would not have been possible without using the CC system.

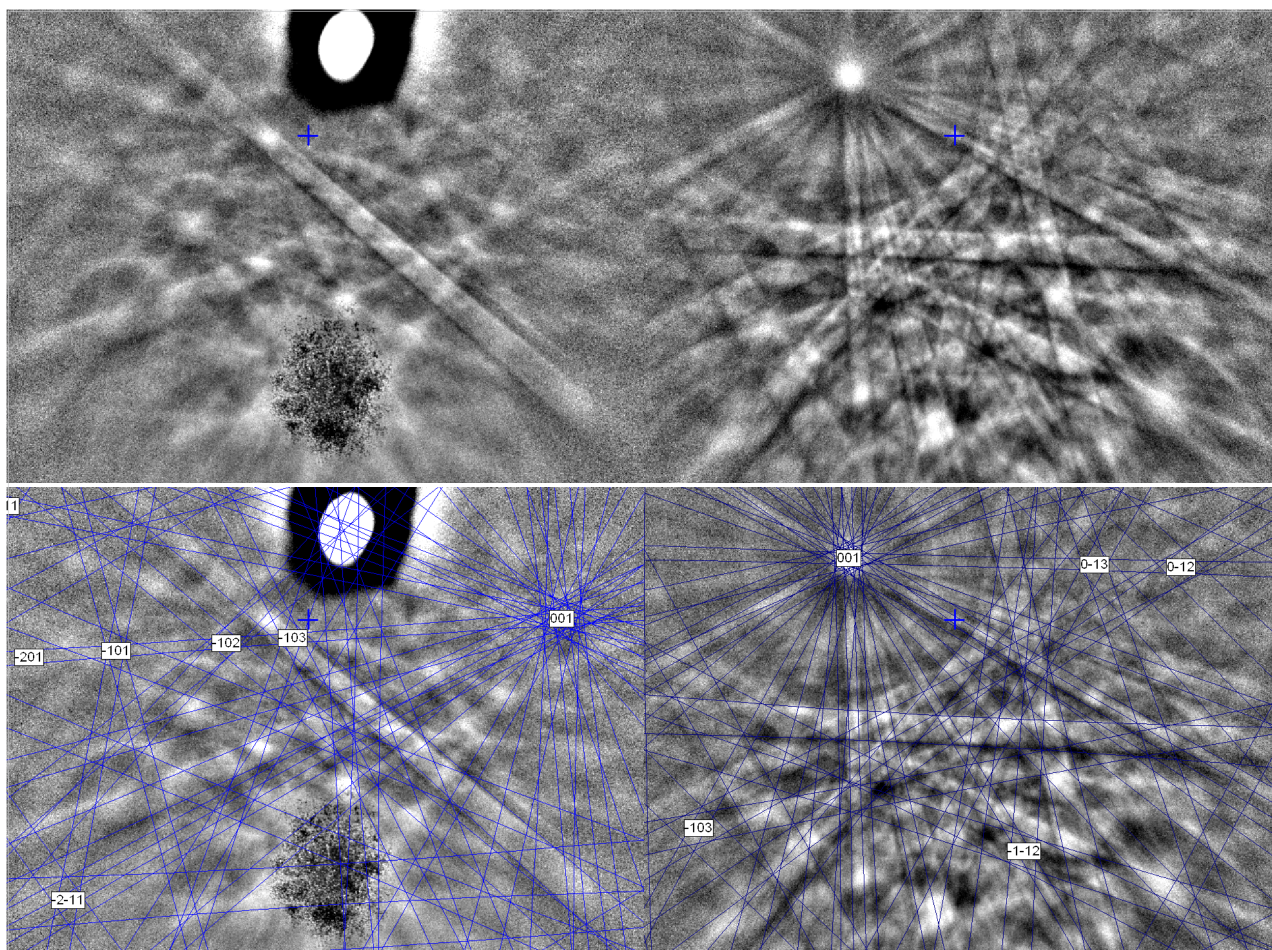

Fig 2 - *Top row: Diffraction patterns recorded at 15 kV acceleration voltage from the N_4Si_3-hex sample without (left) and with Charge Compensation (right). Bottom row: same patterns with indexing solution found by the EBSD software superimposed*

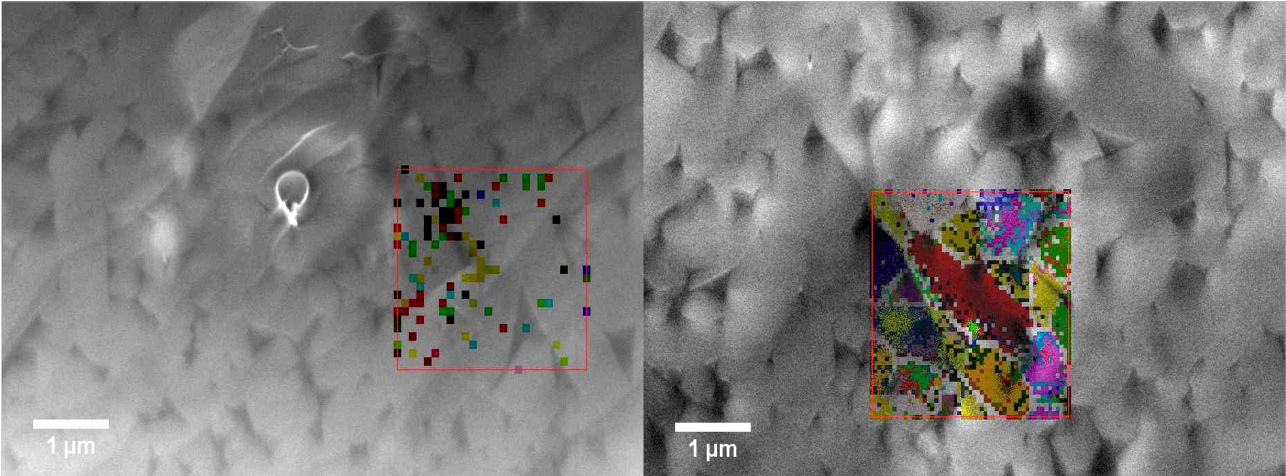

Fig 3 - *EBSD orientation maps recorded at 15 kV acceleration voltage in the marked areas without (left) and with Charge Compensation (right)*

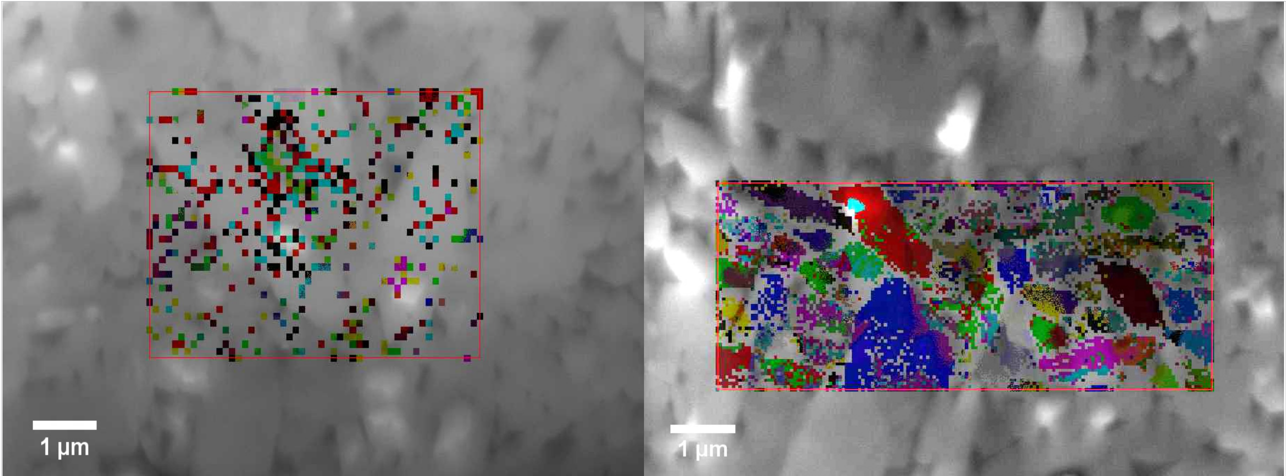

Fig 4 - *EBSD orientation maps recorded at 20 kV acceleration voltage in the marked areas without (left) and with Charge Compensation (right)*

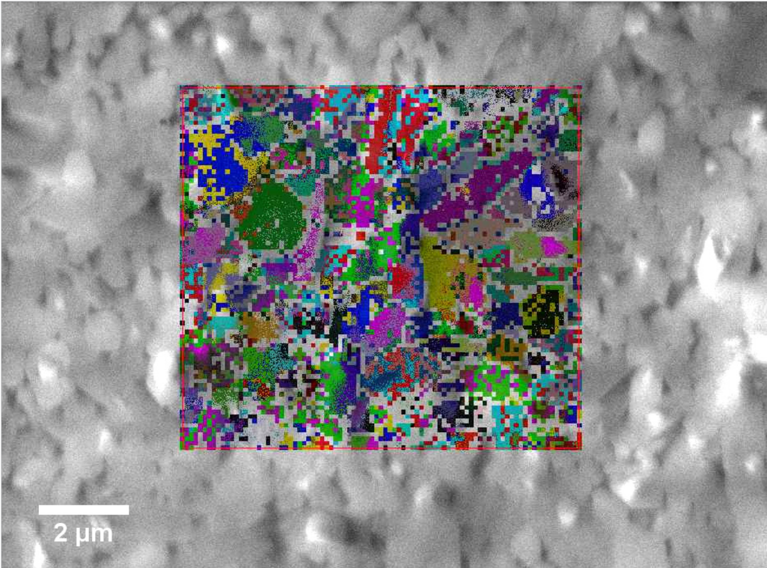

Fig 5 - *EBSD orientation map recorded in the marked area with Charge Compensation at 5 kV acceleration voltage.*

Conclusion

EBSD analysis of nonconductive samples presents an experimental challenge, as the low quality of diffraction patterns from such samples usually requires time-consuming charge mitigation techniques that decrease measurement sensitivity. In the above application example, we showed that the CC system on a ZEISS SEM allows EBSD orientation mapping of an insulating, nanocrystalline sample without special sample preparation or further countermeasures to eliminate specimen charging. Charge compensated conditions were achieved in seconds by simply inserting the CC device and switching on the gas flow.

The mentioned restrictions of sample coating or using VP-SEM apply as well to energy dispersive X-ray spectroscopic (EDS) analysis. The ZEISS CC system greatly improves EDS analysis of nonconductive samples [4]. Using it for simultaneous EDS and EBSD analysis to couple crystallographic and chemical information enables phase identification of insulating materials.

Clean sample surfaces are an essential requirement to successful EBSD analysis. The CC system provides the additional benefit of being able to remove carbon contamination from the specimen surface in-situ [5]. To this end, the region of interest is flooded with oxygen instead of nitrogen, while being exposed to the electron beam. Thus, cleaning of the sample before loading is avoided, and its surface can be repeatedly cleaned whenever necessary between EBSD measurements.

Configuration and compatibility

The ZEISS CC system is available for the following products: AURIGA, AURIGA Compact, MERLIN, MERLIN VP Compact. In order to be used with EBSD analysis, it has to be mounted to the 66mm MP-Port 1. This application requires replacement of the standard CC gas injection nozzle by the EBSD nozzle. The order number of the nozzle conversion kit is 346061-8008-000. Charge compensation using the Multi GIS with integrated CC is not compatible with EBSD analysis.

References

[1] Reimer, L.: Scanning Electron Microscopy, Springer, 1998.
[2] Automated 3D EBSD Analysis with CrossBeam, Carl Zeiss Microscopy White Paper, 2008.
[3] B. S. El-Dasher, S. G. Torres, in: Electron Backscatter Diffraction in Materials Science 2, LLNL-Book-405556, 2008.
[4] J. Stodolka, M. Albiez: Local Charge Compensation, Carl Zeiss Microscopy White Paper, 2010.
[5] J. Stodolka, W. Bühler; In-situ Sample Cleaning, Carl Zeiss Microscopy White Paper, 2008

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