Geophysical Research Abstracts
Vol. 21, EGU2019-16303, 2019
EGU General Assembly 2019

Bridging scales in microstructure and texture analysis: a multi-methodological (SE-, BSE-, FSE-imaging, EBSD, EPMA, FIB, STEM) investigation of reactive corundum – spinel interfaces

Gerlinde Habler (1), Chen Li (1,2), Roman Schuster (1), Lisa Baldwin (1), and Rainer Abart (1)

(1) Department of Lithospheric Research, University of Vienna, Vienna, Austria (gerlinde.habler@univie.ac.at), (2) Stuttgart Center for Electron Microscopy, Max Planck Institute for Solid State Research, Stuttgart, Germany

Investigating the nature of microstructure and texture development during spinel layer growth in natural and experimental settings required the application of various electron beam analytical methods to sample areas ranging from centimeter to nanometer scale. Correlating data collected with different analytical instruments and methods at different magnification and in separate measurement sessions over an extended time period poses challenges to the reproducibility, comparability and correlativeness of the different datasets. A comprehensive EBSD (electron backscatter diffraction) dataset correlated with SE (secondary electron), BSE (backscattered electron) and FSE (forward scattered electron) images, FEG-EPMA (field emission gun electron probe microanalysis) element distribution maps and STEM (aberration-corrected scanning transmission electron microscopy) investigations applied to FIB (focused ion beam) prepared specimens, provides the basis to identify possibilities for analytical improvements to such a multi-methodological approach.

Microstructural data collected at various sample tilt angles are potentially affected by scan distortions for various reasons such as beam scan distortions, tilt correction errors or drift effects. As a result, the orientation of linear features in maps, e.g. grain boundary traces, may be rotated in distorted maps, which impedes their orientation correlation with crystallographic plane traces from crystal orientation data.

When multiple EBSD scans are collected in temporally separated measurement sessions, errors can be introduced by imperfect sample alignment. Selection of particularly arranged multiple reference points that survive repeated chemical polishing stages, reference images taken at sufficiently high magnification, proper working distance adjustment and EBSD pattern center calibration help to minimize effects of imperfect sample alignment in multiple measurement sessions. This is especially critical when small angular orientation variations are considered. Thirteen EBSD scans collected from different segments of a spinel corona around a centimeter sized corundum single crystal document the remaining scatter in orientation data derived from multiple sample mounting. In addition, comparison of crystal orientation data from these datasets allowed distinction of statistical from systematic crystallographic orientation variations associated with different spinel corona segments.

In order to enable direct correlation of EBSD crystal orientation data with atomic scale resolution STEM data a new strategy of plan-view TEM specimen preparation has been developed (Li et al, 2018a). This method allows bringing atomic scale resolution data into direct context with micro- and millimeter scale features that are accessible with EBSD and EPMA analysis. Furthermore, we performed EBSD based sample orientation during FIB preparation at defined inclination angles with respect to the sample surface. These defined section orientations ensure the accessibility of selected zone axes of the studied crystals in viewing direction during STEM analysis. Direct comparison of EBSD and STEM data showed that methods yielding observations at different spatial resolution can provide diverse information content (Li et al, 2016; Li et al, 2018b).

The studied spinel corona around a centimeter sized corundum single crystal allowed testing and improving the reproducibility and correlativeness of data derived from methods working at different scales of observation.

References:

Li et al (2016) Philosophical Magazine, 96, 2488-250
Li et al (2018a) Ultramicroscopy, 184, 310-317
Li et al (2018b) Acta Crystallographica, A74, 466-480