Possibilities and Limits of Phase Identification by Electron Backscatter Diffraction (EBSD) in the SEM

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The use of electron backscatter diffraction (EBSD) for crystallographic analysis in the SEM has been proven to be an extremely powerful tool for the identification of known and unknown phases [1-6]. The particular advantages of these techniques as compared to the competing TEM techniques are the significantly lower efforts for sample preparation, particularly for multi-phase materials, the large area of observation in conjunction with still acceptably high spatial resolution and the high degree of software support for the analysis of crystallographic and compositional data. Already 15 years ago Dingley and Baba-Kishi [1] showed that the crystallographic information included in EBSD patterns are good enough to distinguish a large number of crystal structures based on symmetry observations. Of course, symmetry alone is not enough to determine crystallographic phases but information on the lattice constants are also necessary. In principle, EBSD patterns are less suited than, for example, convergent beam electron diffraction patterns in the TEM. However, the precision of EBSD lattice constant determination can be increased significantly as it has been shown by Michael and Eades [3]. They worked out a procedure for the precise determination of lattice constants from HOLZ rings in EBSD patterns.

From symmetry and lattice spacing information the investigated phases can be characterised rather completely. The combination of EBSD with energy dispersive x-ray spectroscopy (EDX) for quantitative and qualitative composition analysis allows, furthermore, a very quick and reliable phase identification by comparison with existing data bases [4][5]. To this end EDX data are used to select possible phases from a crystallographic data base. From the resulting list of possible phases a further (and often unique) selection is done by indexing a diffraction pattern obtained from the same position as the EDX spectrum. A problem with this approach is that the crystallographic data base with the largest amount of entries, the powder diffraction file data base PDF, is based on x-ray diffraction data and it is well known that x-ray diffraction structure factors (or intensities) do not coincide with electron diffraction values. To this end, Dingley [4] worked out a procedure which allows to change the structure factors given in the x-ray data base and check in this way, whether symmetry and lattice constants of a given data base entry may coincide with the experimentally observed patterns. Once coincidence has been found it is furthermore necessary to calculate the structure factors and diffracted intensities using atom coordinates (as far as available) to proof the correctness of phase determination. It is a very important observation of the author and a co-worker (Zaefferer & Nolze, unpublished results) that the intensities of EBSD patterns can be quite well simulated using the kinematical theory of electron diffraction. Therefore, patterns, calculated based on kinematical theory, are supposed to be a good proof of the correctness of a phase determination.

Once the phases occurring in a sample (or a certain number of them) have been established in a manual procedure as described above, automatic phase mapping can be applied as it has been developed, for example, by Novell and Wright [6]. A combination of all described procedures is assumed to allow the determination of virtually all phases and their spatial arrangement in a sample. If exact compositional information on very small precipitates are required, these precipitates have to be separated from their surrounding matrix. This can be done using a focused ion beam instrument (FIB) which allows to cut out thin foils from predefined areas. Subsequently these thin foils can be analysed in electron transmission mode in a combined FIB-SEM allowing to reach a significantly higher spatial resolution of EDX than on the bulk material.


