Quantitative characterization of nanostructured materials using SEM techniques

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In recent years, nanotechnology has become widely used in many technology and industry sectors with applications in electronics, medical, energy and transportation. The benefit of nanotechnology depends on the fact that the structure of materials can be modified to produce stronger, lighter and more reactive materials with grain size of 100 nm or less. Consequently, the need to characterize these materials has considerably increased. Quantitative characterization of nanocrystalline materials requires high spatial resolution orientation mapping beyond the capabilities of Electron Backscatter Diffraction (EBSD) technique. As a result, Transmission Kikuchi Diffraction (TKD) in SEM was introduced few years ago as a non-destructive technique capable of delivering similar results as EBSD, but with a spatial resolution improved by up to one order of magnitude and the advantage of a much larger field of view than with TEM [1,2]. It is now a well-known method amongst EBSD users.

TKD analysis is conducted on an electron transparent sample (TEM lamella, foil, and nanoparticles) using the hardware and software of EBSD system. When the conventional EBSD geometry is used, transmitted Kikuchi patterns (TKP) are captured by a vertical scintillator whilst the sample is placed horizontally below the pole piece. Therefore, most of the transmitted signal does not reach the phosphor screen which is equivalent to a considerable loss of signal, and TKPs are distorted. These lower quality patterns can have negative effect in measurement quality. In addition, high probe currents are required which limits the spatial resolution. These limitations can be overcome using on-axis TKD detection system. The scintillator is placed underneath the sample for the transmitted signal to be captured where it is the strongest. This advantageous axial position of the detector allows to obtain diffraction patterns with minimal distortions and measurements to be done at low probe currents (1-2nA), increasing the lateral spatial resolution down to 2nm and reducing the beam-induced specimen drift [3]. The resulting improved stability and high spatial resolution allow the user to acquire quantitative data by conducting large-area TKD orientation mapping at high speed.

In this presentation, we will demonstrate that on-axis TKD is an effective technique to quantitatively characterized nanostructures, even challenging samples like non-conductive or highly deformed materials [3,4]. It is also possible to identify and discriminate phases with similar crystallographic structures when combined with simultaneous EDS measurement at a nanoscale. TKD sample preparation requirements will also be reviewed.

References:

[1] Keller R R and Geiss R H 2012 *J. Microsc.* **245** 245-251

[2] Trimby P W 2012 *Ultramicroscopy* **120** 16-24

[3] Abbasi M, *et al.* 2015 *ACS Nano* **9** 10991-1002

[4] Wollschläger et al., 2017 *Material Characterization* **131** 39-48