ProcessDiffraction: a SAED-based method to analyze phases and texture in nano-crystalline thin films in the TEM

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Investigation of thin films in the transmission electron microscope (TEM) combines examination of structure in real space from images with analysis of structure in reciprocal space from the diffraction pattern. There are several advantages of selected area electron diffraction (SAED) based structure analysis over the similar X-ray diffraction (XRD) based analysis, namely: smaller analyzed volume, the combined information from real and reciprocal spaces and the visual selection of the analyzed volume from the image. Disadvantages include more severe instrumental effects, like geometrical distortion by lenses, more difficulty in attaining strict reproducibility and broader angular width associated with Bragg-reflections. Since the strength of interaction of electrons with matter is stronger than that of X-rays by several orders of magnitude, coherent multiple scattering (dynamic diffraction) is almost always present in SAED, in contrast to the situation experienced in XRD.

The general term structure analysis includes three, completely different tasks. First, structure of an unknown phase needs to be determined, including both setting up a rough model by listing the atoms and their approximate locations and refinement of the structure by determining the atomic locations as accurately as it is possible. That task is mainly done by XRD, neutron diffraction (nD) playing a smaller role and electron diffraction was only used to determine a significantly lower number of unknown structures. The second task covers identification which known phases are present (qualitative phase analysis) and determination of how much of them is present (quantitative phase analysis). In a poly-crystalline sample texture frequently exists, so determination of quantities from diffracted intensities can not be separated from identification and quantification of texture components. The Rietveld-method in XRD and in nD is widely used for the first two tasks. Similar method did not exist for SAED. The ***ProcessDiffraction*** method was developed to fill this gap. The third usual task deals with analyzing defects in crystals. The present contribution concentrates only on the second task (qualitative and quantitative phase analysis) and does not attempt to encompass either the first or the third type of tasks.

Large grains (with lateral dimension exceeding 100 nm) can be treated as single crystals in the TEM. Simultaneous identification of phase and orientation is done by indexing a SAED pattern. Reduced accuracy makes this indexing ambiguous sometimes, since distinct planes of two (or more) different 3D reciprocal lattice may coincide within the experimental error. ProcessDiffraction eliminates this ambiguity by simultaneously evaluating several SAED patterns (from the same grain) from a tilting series, taking into account the experimental goniometer settings.

Phase identification in any polycrystalline thin films is done visually, by comparing the experimental intensity vs. the scattering vector length diagram (obtained by angular averaging from the 2D SAED pattern) with “Markers”, showing the diffraction lines of

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known phases. The XRD database can also be used from within ProcessDiffraction for this purpose.

Quantitative determination of phase fractions and texture can only be done for small grain size (about 10-20 nm in beam direction) and small film thickness (about 10-50 nm, depending on the phases in question). The limitation mainly arises from dynamic diffraction, which is approximated for this range by the Blackman-correction (2-beam type approximation). Textured component can only be taken into account for special type of texture and special sample orientation. Fiber-textured samples must be oriented in the TEM so that the beam direction should be close to the texture-axis. Almost uniform concentric rings must be seen for such samples (similar in appearance patterns from randomly oriented samples). Examples of application are shown in the conference.

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2. J.L. Lábár, Microsc. Microanal. **14** (2008) 287-295
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4. The program can be downloaded free of charge from http://www.mfa.kfki.hu/~labar/ProcDif.htm
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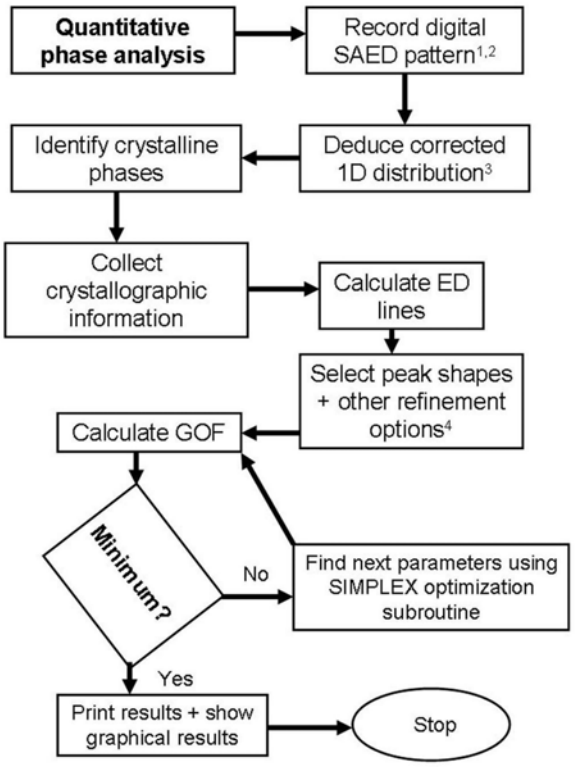

Figure 1. Block diagram of quantitative phase analysis from nanocrystalline thin films (reproduced from [2]). Randomly oriented fraction and fiber-textured fraction of the same phase are treated as separate components. Grain size (in beam direction) is limited by dynamic diffraction to about 10-20 nm. Film thickness is limited by multiple diffraction to about 10-50 nm, depending on the phases in question.