Sample thickness determination by measurement of signal intensity in on-axis transmission Kikuchi diffraction

Transmission Kikuchi diffraction (TKD) is a novel scanning electron microscopy (SEM) technique for the quantitative characterization of crystal microstructure, grain size and crystallographic texture in materials. The technique is based on the acquisition and automated real-time indexing of Kikuchi diffraction patterns (Fig. 1a) from a set of positions on the sample, which are then used to reconstruct its crystalline structure in the form of a crystal orientation map (Fig. 1b).

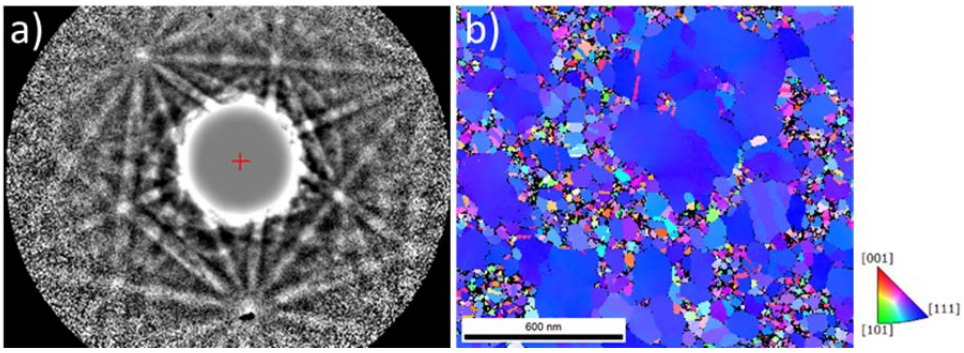

Fig. 1: a) Kikuchi diffraction pattern with background removed, obtained from an Au film; b) Orientation map of a gold film, showing the different crystal orientation of the grains in the film.

The combined determination of microstructure orientation and local thickness would be beneficial to various research areas, including for example:

- Assessment of the minimum thickness necessary to obtain an indexable Kikuchi pattern, a parameter which is material-dependent;
- Localization of sites with reduced film thickness in ultra-thin metal films, which are thought to be related to the starting point of solid-state dewetting in metallic thin films.

Preliminary data showed a quasi-linear relationship between the measured area of the oversaturated signal at the center of the diffraction pattern (Fig. 2a) and the sample thickness measured by cross-section SEM imaging (Fig. 2b).

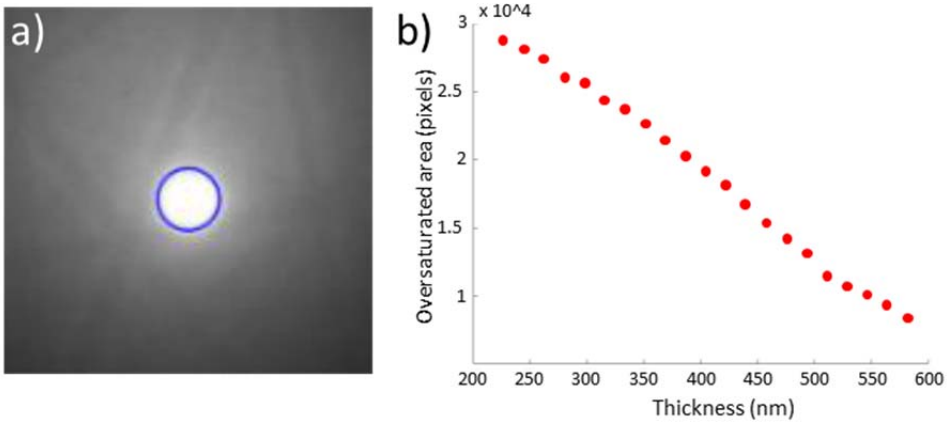

Fig. 2: a) Measurement of the oversaturated area in a Kikuchi diffraction pattern without background removal; b) Relationship between measured direct beam area and thickness of the sample.

The aim of this project is to establish a method to directly extract the sample thickness from the size of the direct beam. The broader perspective is to develop a data analysis code which can automatically extract the diameter of the primary beam in all the Kikuchi patterns of a scanned map, transform it in a relative thickness value and plot a 2D thickness variation map as a result (Fig. 3).

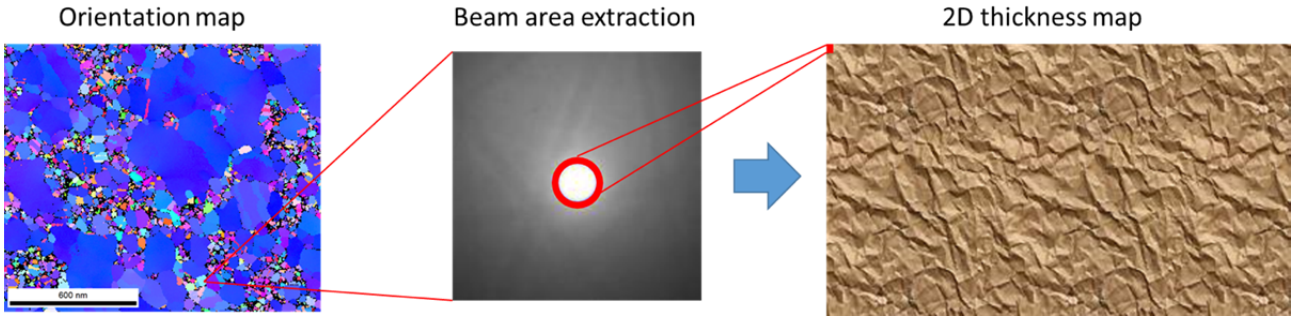

Fig. 3: The overall process of the project: 1) extraction of Kikuchi pattern forming an orientation map; 2) automatic measurement of the diameter of the oversaturated area; 3) Conversion to a relative thickness value and plot of a 2D thickness variation map of the scanned area.

The envisioned work includes:

- Learning the use of focused ion beam (FIB) and ion milling for the fabrication of samples with a well-known thickness profile, to be used as calibration samples for the thickness measurement scheme;
- Determination of the primary beam profile in TKD patterns collected in vacuum;
- Calculation of the expected oversaturated area vs. thickness trend, its range of validity, and linearity.
- Analysis of different materials, in view of compiling a library of the measurable thickness range as a function of atomic number;
- Comparison of the extracted thickness with measurements obtained independently by Electron Energy Loss Spectroscopy (EELS) and/or Convergent Beam Electron Diffraction (CBED).

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