Transmission electron microscopy (TEM) is a powerful tool for investigating the atomic structure and morphology of nano- and micro-objects. To reveal the structure of a material the specimen prepared for TEM should be reasonably thin in order to be transparent for the electron beam. The thickness of the sample is an important parameter one should account for when analyzing images acquired in TEM. The probability for electrons to scatter multiple times increases with the specimen thickness. This effect has, for example, a strong influence on the contrast of high-resolution TEM images. There are several techniques available to estimate the thickness by TEM [1]. These methods can be divided into several categories, e.g. imaging methods, Convergent Beam Electron Diffraction (CBED) method, electron energy-loss spectroscopy (EELS) - based methods, and methods based on X-ray spectroscopy (EDXS). The most popular technique among them is based on EELS [2], which can be used for a variety of samples and is easy to implement computationally. However if one has thin crystalline samples one often has to deal with bending due to lattice relaxation. Bending can strongly influence the thickness values obtained by EELS. Here we report about a new technique which simultaneously delivers thickness and specimen surface orientation maps. Our approach is based on analysis of rocking curves extracted from experimental dark-field (DF) images acquired at different specimen tilts. We fit the parameters that affect dynamical electron diffraction rocking curves to experimental DF images. In its simplest version, our approach uses 2-beam theory, for which the intensity of the diffracted beam is given according to C.Humhreys 1979 review [3]. To determine the thickness we fit the power spectra to a sum of Gaussians. Since the rocking curves usually exhibit oscillatory behavior reflecting the thickness of the specimen one should observe peaks in power spectra of rocking curve. Fitting was done in MATLAB by using the unconstrained nonlinear optimization routine fminsearch [4] and the Matlab Curve Fitting Toolbox. As an example the mapping was done for a commercial semiconductor device. Figure 1 shows a single slice from DF image stack of this device acquired for the {220} reflection. Figure 2 illustrates how a dark-field tilt series samples reciprocal space to demonstrate the oscillatory behavior of the extracted rocking curve (inset in Fig. 1).


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Fig. 1: Single slice from a DF image stack acquired for the (220) reflection of Si in a MOSFET structure. The red square shows the ROI used for thickness and orientation mapping. The sample was prepared by automatic tripod polishing and consequent low-voltage Ar ion milling at T of liquid N2.

Fig. 2: The Dark-field tilt series intersects the diffraction signal with the Ewald sphere indicated as green arcs at different positions along k_2 axis. The diffraction signal was calculated by FFT for a 20 nm thick slab.

Fig. 3: Thickness map in Å. The data was binned before fitting. The thickness fitted outside the crystalline area is meaningless, of course.

Fig. 4: Map of the misorientation a0.