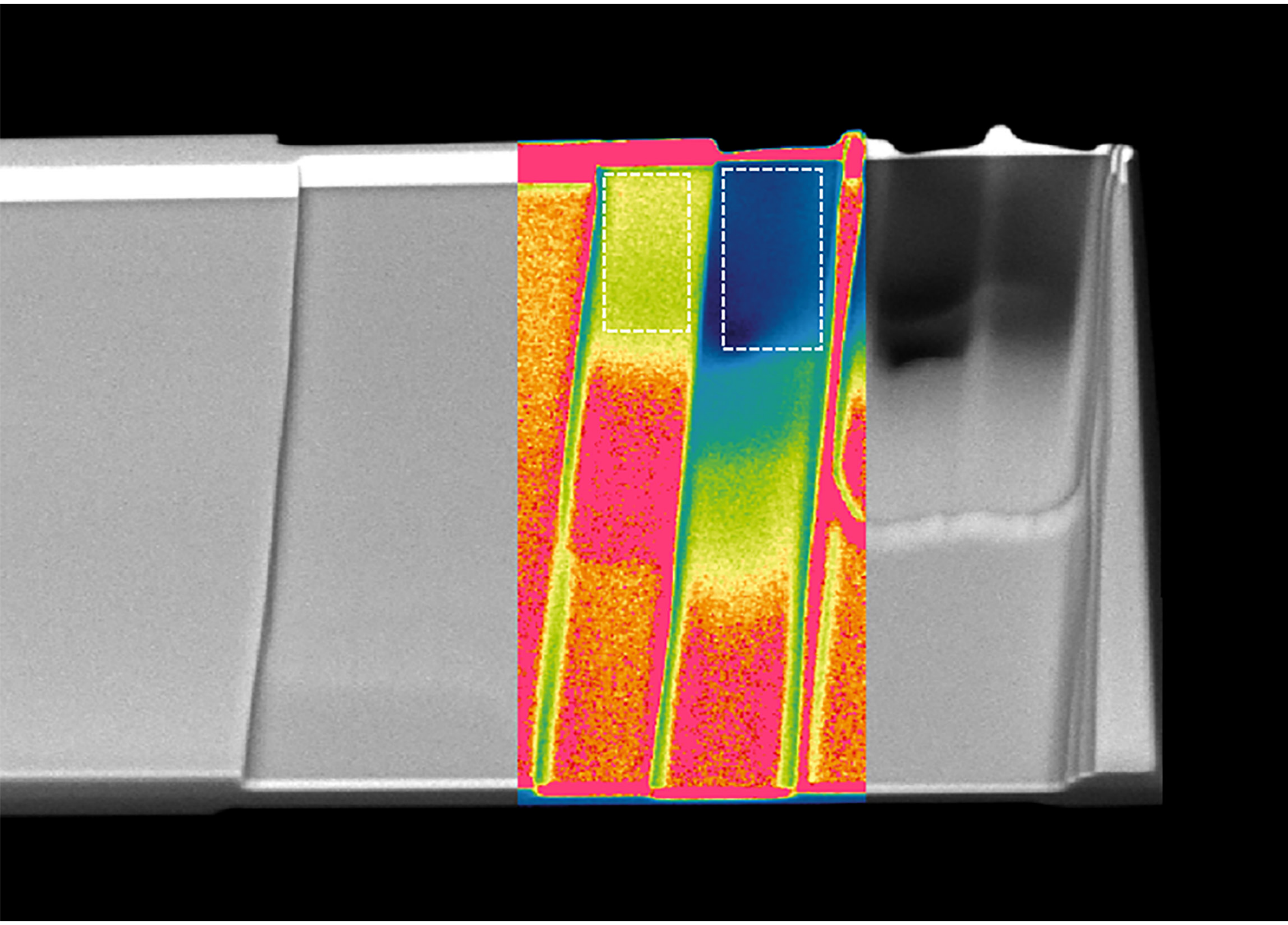

ZEISS Crossbeam

Reproducible TEM Lamella Thinning by FIB with Real-time Thickness Control and End-point Detection

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Reproducible TEM Lamella Thinning by FIB with Real-time Thickness Control and End-point Detection

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The thickness of a TEM lamella is measured by a solution, SmartEPD, that is based on quantification of the backscattered electron (BSE) signal. The thickness measurement is possible on static images but also live during the FIB thinning process. This allows automated polishing of the TEM lamella towards a predefined target thickness (end-point detection). SmartEPD is fully integrated in the microscope control software of the ZEISS Crossbeam family.

Introduction

An important application of FIB-SEM instruments in materials science and electronics is the preparation of thin lamellae for their investigation by transmission electron microscopy (TEM) or scanning TEM (STEM). As compared to classical preparation techniques such as electro polishing, mechanical grinding and polishing, or broad ion beam milling, FIB-SEM offers the advantage of being sitespecific, i.e. location and targeted preparation of a region of interest (ROI) is straightforward [1]. Further, FIB-SEM can be employed on a very wide range of materials, as almost any material can be machined by FIB. High-resolution studies of lamellae with resolutions down to the atomic level are performed on dedicated (S)TEM instruments. Another, increasingly important analysis method is STEM in the FIB-SEM or in the SEM. In this case, imaging and energy dispersive spectroscopy (EDS) is possible down to the sub-nm or few-nm level, respectively [2].

All of these experiments require a lamella that is imaged in transmission, so the specimen needs to be thin enough to be electron transparent. The maximum allowable sample thickness depends on the material and energy of the electron beam available in the chosen microscope. Today's experiments often demand even thinner lamellae. For example if a single isolated 14-nm transistor of a dense integrated circuit is to be analyzed, the lamella thickness needs to be in the order of, or smaller than the characteristic dimension of the ROI. Or for example if multiple scattering events and energy loss in the sample affect the quality of the study, the lamella thickness needs to be thinner than the inelastic mean free path length of the probing electrons. Typical lamella thicknesses for these and other experiments are in the range of 20 nm to 100 nm.

Solution

Accurate lamella thickness control during FIB polishing is a challenge. Up to now, real-time monitoring of lamella thickness during the thinning process was not possible, making it difficult to stop at a desired target value.
One possibility is the observation of the transmitted electron signal with a STEM detector during polishing. This helps when targeting a very small ROI as in the 14-nm transistor example mentioned above. The user can stop the process when the target feature appears to be reached. The method provides a rough estimation of thickness changes, too, but only qualitatively. Additionally, the contrast may be obscured by electron diffraction effects in the case of crystalline materials. The need for a dedicated sample holder that allows inserting the STEM detector during FIB work is a further drawback of this method.

Another possibility relies on the secondary electron (SE) signal detected by an Everhart-Thornley detector.

With decreasing lamella thickness the SE yield from the lamella is enhanced, because of the contribution of those SEs emitted from its backside. This contrast mechanism depends on the electron landing energy and again gives only a rough qualitative estimation of the lamella thickness.

In both cases, the experience of the operator has an influence on the final quality of the lamella, often leading to less reproducible results.

In this section, a quantitative thickness measurement solution, called SmartEPD, based on the backscattered electron signal is described. Among other parameters, the BSE intensity depends on the maximum depth inside the sample from which backscattered electrons can still escape. A thick (or bulk) sample, with a thickness larger than this maximum backscatter depth (MBD), will yield a higher BSE intensity than a thinner sample. This is because in the latter case a larger fraction of the primary beam electrons will traverse the sample and not be detected as BSE. Figure 1 illustrates this effect schematically.

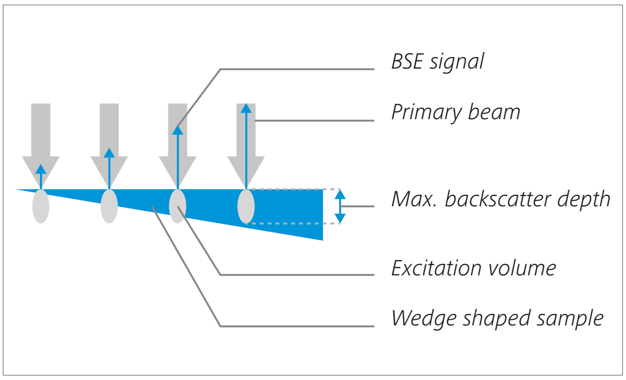

Figure 1: *Schematic drawing of the BSE signal in dependence of the sample thickness. The length of the blue arrows illustrates the intensity of the BSE signal.*

By means of Monte Carlo simulations [3], it was possible to derive a global normalized BSE transparency function (GNTF) based on a modified incomplete regularized Gamma-function [4]. The GNTF describes the normalized BSE signal as a function of thickness, primary beam energy and material parameters (density and mean atomic number) for all elements of the periodic table. SmartEPD relies on the GNTF to translate the grey values of a BSE image into thickness values.

After choosing the reference sample material and once minimum (background without sample material) and maximum (sample region thicker than MBD) signal levels have been determined for scaling, the according thicknesses are assigned to the grey levels pixel by pixel in the BSE image. This thickness information can be displayed as color-coded thickness map for the whole image or a chosen area (Fig. 2). SmartEPD leverages the BSE signal detected by the Inlens Energy selective Backscatter (EsB) detector. This signal is not affected by SEs generated during the FIB milling process or any other SEs. Thus, the thickness measurement even works live during the lamella polishing process. The decreasing lamella thickness can be observed in real-time during the FIB exposure and automatic end-point detection becomes possible. The average thickness inside a reference area is constantly checked and FIB milling automatically stopped once the target thickness is reached.

With the latest generation of ZEISS Crossbeams, the previously available stand-alone software package SmartEPD is integrated into the microscope control software. This enables live lamella thickness measurement, automatic end-point detection, and thus better and more reproducible results. It is available as a mode (i.e. EPD mode) in SmartSEM’s FIB user interface [5].

Application Example

The above-described solution has been applied to a silicon lamella. Figure 2 shows a BSE image of the lamella in side-view acquired at 5 kV with a color-coded thickness map overlay in the center.

In this area, two windows are visible where the lamella was thinned towards a target thickness with automatic end-point detection. The target thickness for window 1 (left, yellow/green) was 100 nm, while for window 2 (right, blue) 50 nm final thickness was targeted. The average thicknesses inside the two areas marked by white dashed boxes could be measured by SmartEPD as 96.2 nm and 40.0 nm respectively.

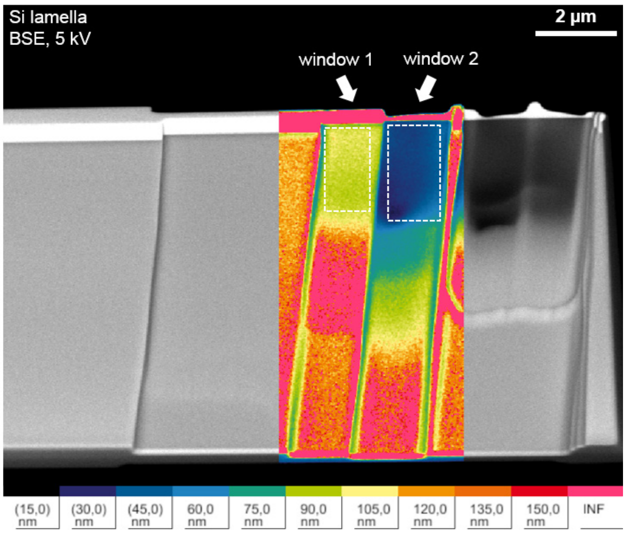

Figure 2: *BSE side-view image of a silicon lamella at 5 kV with thickness map overlay. Two windows have been thinned in the center area using automatic end-point detection, with target thicknesses of 100 nm (left, yellow/green) and 50 nm (right, blue) respectively. SmartEPD measured 96.2 and 40.0 nm respectively as average thickness inside the areas marked by white dashed boxes.*

The very same lamella is shown in Figure 3 in SE top-view. Looking straight on the top edge of the lamella it is possible to measure its thickness directly. The thickness of the windows was determined to 99 nm and 54 nm respectively. These values are in good agreement with the ones measured by SmartEPD.

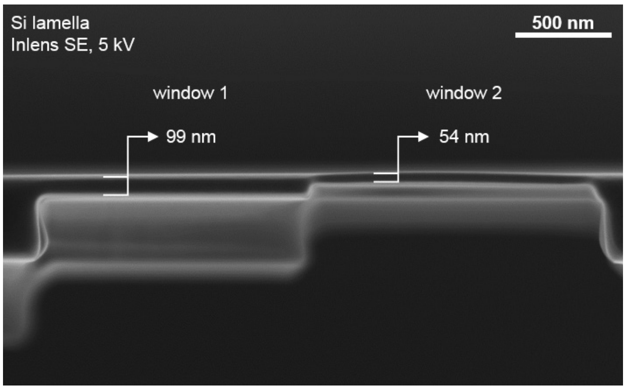

Figure 3: *SE top-view image of the same silicon lamella at 5 kV. Direct measurement of the lamella thickness yielded 99 nm for window 1 and 54 nm for window 2.*

Conclusion

The described solution for TEM lamella thickness measurement is based on BSE contrast. It is integrated in the microscope software and enables automatic lamella thinning with end-point detection.

The overall accuracy of the presented solution is estimated to be better than 20 %, which is comparable to other methods for lamella thickness measurement in the TEM, like e.g. using electron energy loss spectroscopy (EELS) or convergent beam electron diffraction (CBED). However, it has the advantage that it can be used *in situ* during the FIB polishing process, providing direct feedback about the current lamella thickness and thus facilitating reproducible results.

References:

[1] D. V. Sridhara Rao et al., “TEM specimen preparation techniques”. Microscopy: Science, Technology, Applications and Education. Eds. A. Méndez-Vilar and J. Diaz. Formatex Research Center, Badajoz (2010), p. 1232-1244.

[2] D. Willer and F. Pérez-Willard, High Resolution STEM and EDS Study of Chromium Depletion in Stainless Steel, ZEISS Application Note (2015).

[3] D. Drouin et al., Scanning 29 (2007), p. 92-101.

[4] R. Salzer et al., Microsc Microanal 15 (Suppl 2) (2009), p. 340-341.

[5] T. Volkenandt and F. Pérez-Willard, Enabling Smart FIB Work with SmartSEM, ZEISS Technology Note (2016).

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