Excel@FIT 2023
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Measuring the Thickness of Material Layers Removed from a Sample in an Electron Microscope

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Abstract

This poster focuses on measuring the thickness of material layers, which are removed from a sample in an electron microscope during a 3D destructive analysis. The aim of this project is to develop a more efficient measurement method compared to the existing ones and to find a way of obtaining a ground truth for the measured results. The proposed methods are based on detecting features in a series of images of a sample and calculating changes in their positions. To be able to compare the methods and evaluate their precision, I came up with a concept for obtaining the ground truth. Hundreds of tiny circles are carved into a sample surface and gradually removed with the material layers. Knowing the arrangement of the circles, the actual thickness is determined by detecting their numbers in each image. This ground truth obtaining concept was confirmed as feasible, the measurement is under way.

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1. Introduction

Motivation The motivation for this thesis arises from the aim of the Thermo Fisher Scientific company to develop a method capable of measuring the thickness of material layers ("slices") removed from a sample in an electron microscope, which would be more practical from a user point of view compared to the existing methods. Unfortunately, there is no way of how to compare such methods and evaluate their precision, because there is no ground truth. To be able to do so, a ground truth for the measurement must be obtained.

Problem definition Iterative removing of material layers and imaging the sample surface from which the material was removed is a method for destructive 3D sample analysis in the electron microscope [1] [2]. The proposed methods measure the thickness of such layers by detecting simple features and calculating their distances in the images acquired during the process of material removal. The methods should be both accurate and easy to use, so that they can be integrated into a 3D analysis without slowing it.

Additionally, a way to obtain the ground truth is needed for comparing the methods and finding their accuracy. As the ground truth obtaining method should only serve for evaluating the other methods' precision, there is no need of developing a simple or fast solution. On the other hand, it is crucial that the ground truth values correspond to the real ones.

2. Related Work

So far, there is only one state-of-the-art method for measuring the thickness of removed material layers. In 2010, Edward Principe proposed a solution [3] lying in carving a chevron pattern into the sample surface and quantifying the slice thickness by measuring the changes in pattern position between consecutive images. This is an elegant solution as for the measurement, but the requirement of carving the marks before the start of the material layers removal adds an extra overhead to the entire process.

3. Proposed Solution

This project proposes two new methods, which both measure the slice thickness by detecting features already present on the sample surface, meaning that they do not require any preparation steps. One of these features is the sample edge, at which material layers are being removed, causing a gradual change of its position, and the other one may be any sample feature whose position does not change over time. The resulting thickness is then determined by comparing distances of both features in two consecutive images. The methods differ from each other in the modality of the acquired images.

To obtain the ground truth, a pattern of hundreds of tiny circles of a predefined shape is carved into the sample surface near the sample edge, before the iterative process of material removal and sample imaging occurs. In each iteration, a number of circles is removed from the sample surface. By detecting their numbers in each image, the slice thickness can be determined, because the actual distances between the circles in the pattern are known.

3.1 Slice Thickness Measurement Methods

Figure 2 and **Figure 3** both illustrate a principle of the Top-Down FIB method *(orange)* on two consecutive slice images. The horizontal lines represent detected positions of both image features, based on which a slice thickness value is determined.

A principle of the Y-Shift SEM method *(yellow)*, depicted in **Fig. 1**, is similar to the Top-Down FIB one, except that the Y-Shift SEM processes slice images acquired using a focused beam of electrons from an angle, instead of a beam of ions aiming perpendicularly to the sample surface.

The **Figure 1** shows the idea behind the Chevrons method *(grey)* too, lying it detecting positions of a chevron pattern on the sample cut-face, where the material layers are being removed.

3.2 Ground Truth Obtaining Method

The **Figures 4 to 11** illustrate the principle of obtaining a ground truth for the measurement. In a pattern of tiny circles, (*dark blue* rectangle in **Fig. 1**) a set of keypoints is extracted using the SIFT detector first, based on which a template image is cropped (**Fig. 5**). By performing a template matching on the entire image, some of the circles center points are find, which are then used to fit a 36×10 grid, corresponding to the circles arrangement, on the image.

A region is cropped around each of the grid lines intersections, representing an expected area of a circle occurrence. By sliding the template over each of the grid region images, a normalized cross-correlation coefficient is calculated determining the measure of similarity for each couple (the highest and lowest similarity scores are shown in **Fig. 8** and **Fig. 9**). Knowing such scores, a threshold for circles counting is calculated (**Fig. 10**) and based on the fact that the individual circles are shifted by 2 nm in the y-direction, the thickness of a removed layer can be determined.

Apart from this automated ground truth obtaining method, a manual alternative, lying in annotating the individual images by hand, was created too.

4. Experimental Results

All the methods, including the ground truth obtaining ones, were tested and compared on three different datasets, each of them consisting of 11 to 13 slice images. In all slices 40 nm thick material layers were expected to be removed.

Table 1 presents detailed results measured on the the first of the three datasets, showing calculated slice thickness values for consecutive images by all the methods. The bottom rows of the table display the statistical summary measures for each method. Below the table, a bar chart in **Fig. 12** shows the individual measured values as a graph.

Lastly, **Table 2** presents a table comparing standard deviations of slice thickness values measured by the methods in each of the three datasets.

5. Conclusions/Summary

The goal of the thesis was to develop a more efficient method for the slice thickness measurement compared to the existing ones and to obtain a ground truth for the measured results, to be able to find the methods' accuracy. I proposed two new methods, do not requiring any redundant preparation steps, and a concept for obtaining the ground truth, based on counting tiny circles carved into a sample surface in a series of images, which proved to be realisable.

References

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[3] Edward Principe. High-density fib-sem tomography via real-time imaging, 2010.