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# Exploring thin films by using STEM techniques in a dual beam workstation

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Thin films as coatings of materials for technological applications are becoming increasingly important because they provide resistance against physical and chemical attack which otherwise would lead to serious degradation. In addition, by structuring of thin films, surfaces may be functionalized by changing e.g. the mechanical properties [1]. Because the thickness of these thin films is often below 1 µm, precise methods of target preparation are needed for further characterization. The application of the focused ion beam technique in such cases often provides the best or even unique solution.

Conventional EDX analysis on traditional FIB cross sections is not suitable for characterization of structures well below 1 µm because of the size of the interaction volume. In this study, the potential of EDX-analysis and STEM analysis using FIB prepared thin foils is explored with respect to resolution limits and possibilities of quantification. EDX analysis in STEM mode in a TEM operating at accelaration voltages of 200 kV has a resolution on the order of a few nanometers [2], hence enabling analysis of chemical interaction, e.g. short diffusion profiles [3]. At accelaration voltages of 30 kV and less the spread of the electron beam in the sample is expected to be larger than at higher energies but preliminary experiments of [4] showed the potential of decreasing the resolution down to ~100 nm.

Laser interference metallurgy is a newly developed method of structuring and functionalizing of metallic thin films [1,5]. In this case, interaction of the metallic layers occurs at a local scale well below 1 µm. In [5] the interaction was investigated in a TEM using FIB-prepared TEM thin-foils. In addition, STEM analysis in a FEI Dual Beam Strata 235 operating at 5-30 kV was carried out. The STEM detector already provides information of the microstructure not obtainable by normal SE imaging. Fig 1a shows a remaining Al layer in an interference minimum. Arround this layer a film of a second phase with a thickness of 10-20 nm was formed. In [5] these films were indentified as $Al_2O_3$ by using a TEM equipped with an EDX detector. In this study, EDX-line scans were performed (Fig. 1b) at 17 kV using the STEM detector in the dual beam workstation. The oxygen profile clearly indicates the presence of the oxide. In Fig. 1b all intensities of the different elements had to be normalized to Mg. This element is not present and therefore the intensity at this position is an approximation of the background intensity. The problem of using the STEM detector is that it is made of Al (detector structure) and Si (active area). Hence, Al and Si peaks generated by backscatter electrons are always present. Also the background intensity is greatly increased by this phenomenon, significantly reducing peak to background ratios. Hence, quantification of such spectra seems to be impossible.

For further investigation, a test sample was produced by PVD deposition of Al and Cu layers of varying thicknesses. FIB TEM preparation was carried out but instead of using the STEM-holder, a holder made of graphite was employed. The structure of the test sample is shown in Fig. 2a and a profile across the layers in Fig. 2b. Layers larger than 80 nm can clearly be resolved whereas the thin

layers of 20 nm and less appear as a single layer in the profile. The only possibility for further decreasing the resolution limit would be further thinning of the sample. For testing the possibilities of quantification of such analyses, a TEM sample of natural olivine $(Mg,Fe)SiO_4$ was prepared. The sample is the same used for diffusion studies in [6]. First results show that using theoretically calculated k-factors, quantification can be successfully performed with accuracies better than 5%.

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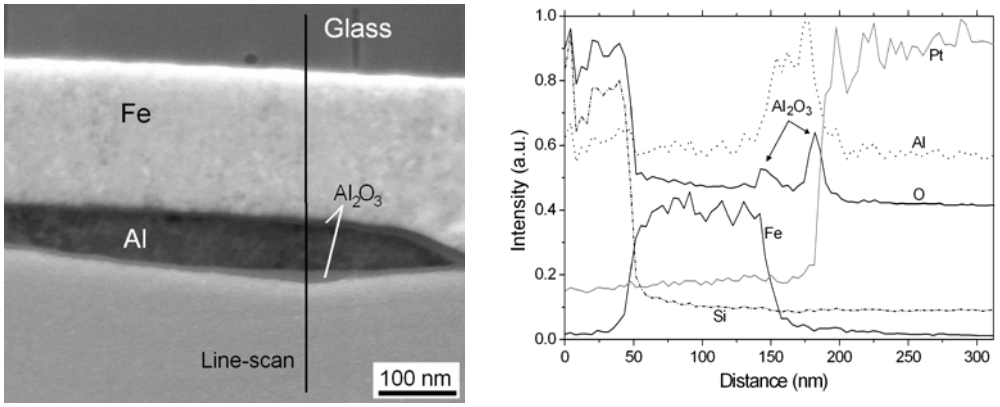


FIG. 1. (A) Cross-section of the line type-periodical structure of a Al(30nm)/Fe(120nm) sample at one interference minima. (b) EDS Line-scan of the TEM foil as indicated in (a) revealing two nano-layers of Al2O3.

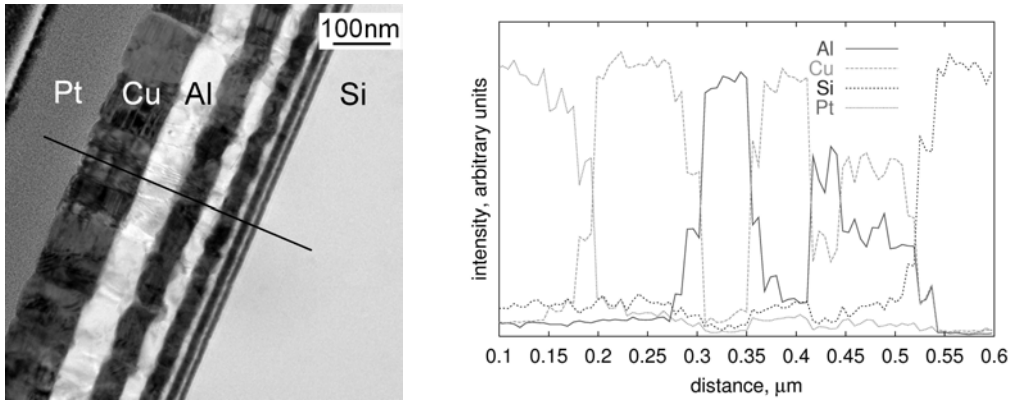


FIG. 2. In (A) a TEM micrograph of the test structure described in the text is shown. The thicknesses of the different layers range between 5 and 100 nm. In (B) an EDX linescan is shown. The presence of Si indicates the glass structure whereas at the top of the sample a Pt layer was deposited.

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