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In-situ sample preparation of oxidizing and contaminating samples for high quality EDS and WDS quantification using FIB-SEM

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Energy dispersive and wave dispersive X-ray spectroscopy (EDS and WDS) are very important tools in materials research to obtain information about the chemical composition of a sample. Many materials like thermoelectrics show a significant loss of efficiency with elemental compositions slightly differing by a few percent. In quantitative measurements conductivity, contamination and, above all, homogeneity and the geometry of the sample have a significant influence on the quantification results with EDS or WDS systems. Especially for WDS the quality of the sample surface is very important to achieve an accuracy below 1%. Many materials are susceptible to oxidation (Fig. 1, right) or carbon contamination on their surface decreasing the accuracy and quality of WDS and EDS measurements. Furthermore, this prevents the quantification of initial oxygen or carbon contents.

The use of FIB allows the preparation of smooth cross-sections with very good smoothness and planarity (Fig.1, left). With an EDS/WDS system installed at a FIB-SEM, an in-situ, site specific preparation followed by analysis without leaving high vacuum is feasible. This enables a contamination- and oxidation-free preparation and analysis, significantly improving the quality of the measurement.

Cross-sections prepared with the FIB are usually not perpendicular to the electron beam, which is a pre-requisite for a proper quantitative EDS or WDS analysis. In this study, a new procedure is presented allowing an accurate quantification with EDS and WDS by using different tilt angles for the preparation and analysis step.

Initially, the cross-section of a thermoelectric sample is prepared at a tilt of -10° producing a flat angle between sample and Ion beam on a system including an angle of 52° between electron and ion column. By tilting the sample to +28° afterwards, the cross-section is orientated perpendicular to the electron-beam enabling a quantification with EDS and WDS (Fig: 2). The results sum up to a total weight of 100.23% indicating a reliable combined EDS/WDS measurement (Tab. 1, left). For the sample surface produced via metallographic state-of-the-art procedures, which is oxidized due to atmospheric exposure, a weight of 92.23% is measured indicating a non accurate measurement caused by the high oxygen contamination layer on the surface (Tab. 2, right). This comparison indicates that this procedure significantly improves the quality of EDS and WDS quantitative compositional results for fast oxidizing and easy contaminating samples.

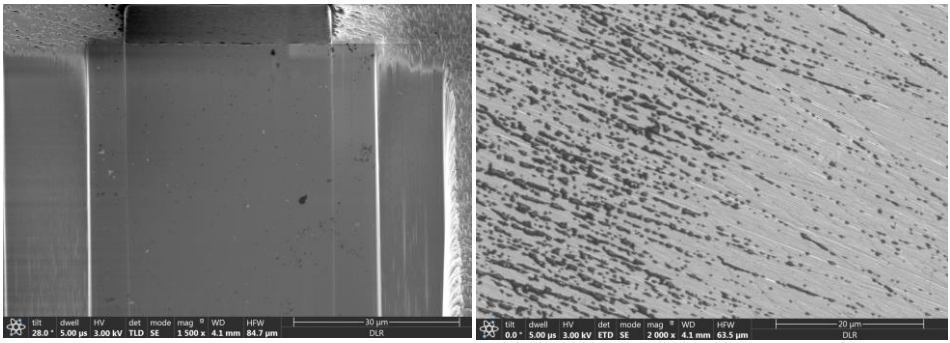

Fig. 2: Left: Surface of FIB prepared Cross-Section in a flat angle. Right: Oxidized surface of the sample

	FIB Prepared		Metallographic Preparation	
Element	Weight [%]	Atom [%]	Weight [%]	Atom [%]
Mg+	8.82	31.28	8.74	33.14
Ag	40.74	32.65	37.53	32.06
Sb	50.67	35.97	45.96	34.79
Total	100.23	100.00	92.23	100.00

Tab. 1: Left: Quantification with WDS of MgAgSb in a FIB prepared cross-section with corrected tilt. Right: Quantification with WDS of the same sample, but on its surface prepared by conventional metallography procedures. Mg was measured by WDS, AG and Sb by EDS.

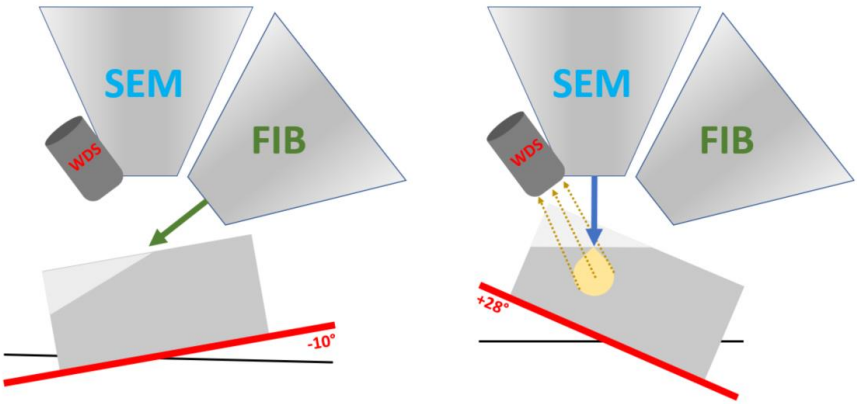

Fig. 2: Left: Cross-Section with -10° stage tilt resulting in a -28° tilt compared to the sample surface; Right: Stage tilt of +28° to achieve the geometry necessary for WDS quantification.