To investigate non-conductive samples in conventional scanning electron microscopy (SEM) the sample must be sputtered or evaporated with an electrically conductive layer to prevent charging.

Evaporation with carbon is typically used for investigations with backscattered electrons or for energy dispersive x-ray spectrometry. Thin carbon layers are relatively transparent for high energetic backscatter electrons and with the exception of the carbon K\(\alpha\) line this layer does not interfere with other detected x-rays.

For acquiring high resolution secondary electron images, the sample must be metal sputtered to increase the secondary electron yield. These sputtered layers have a big drawback, the grain size within the sputtered film. The topographic information of these artefacts superimpose with information from the sample which degrades image quality especially using modern high resolution SEMs with special resolution of less than 1 nm.

All investigations were performed using a commercially available magnetron sputter coater (Leica EM ACE600) or a high frequency gas discharge apparatus (FELMI-ZFE-GEA005). The basic aim was to ensure the thinnest possible continuous film over the sample surface with the smallest grain size. Different metal targets and parameters (e.g. chamber pressure, current, layer thickness and so on...) were used to optimize the sputtered layers.

The layers were analyzed using high resolution scanning electron microscopy (HR-SEM) for topographic information, transmission electron microscopy (TEM) for crystallographic information and atomic force microscopy (AFM) for 3D information. Power spectral density analysis (PSD) was used to determine the grain size distribution quantitatively.

In figure 1 TEM bright field images of gold, gold/palladium (80/20), and chrome layers can be seen (20 nm thick, sputter coater: GEA005). The different grain sizes as well as crystalline structures can be identified.

Figure 2 shows exemplarily a 4 nm thick sputtered gold/palladium layer on glass substrate. These images were used for PSD analysis. In figure 3 the influence of layer thickness on grain size distribution using the gold/palladium target and the Leica ACE600 can be seen. The peak shifts from about 0.063 [1/nm] to 0.003 [1/nm] indicates a decreases in grain sizes from 33 nm at 6 nm layer thickness to 16 nm grain size at 1 nm layer thickness.

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Fig. 1: TEM bright field images (left to right: 20 nm gold, gold/palladium and chrome layer)

Fig. 2: Scanning electron micrograph (sample: gold/palladium (80/20) on glass substrate)

Fig. 3: Power spectral density for different layer thicknesses (sample: gold palladium layer (80/20) on glass substrate)