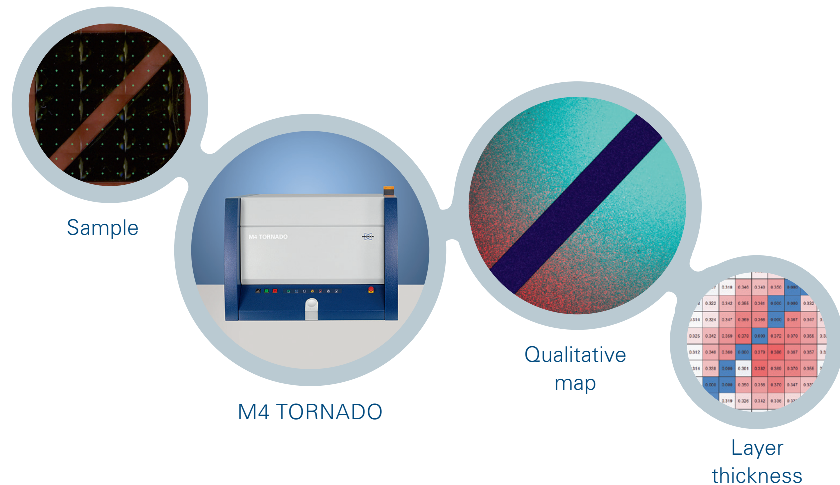


Lab Report XRF 462

Spatially resolved layer thickness analysis of thin metal coatings

Manufacturers have to regularly check their coating processes and monitor the output product quality, i.e. the thickness of the coatings. As the coatings are often composed of expensive materials, it is necessary to find a reasonable balance between cost-effective thin layers on the one hand and a sufficient layer thickness that guarantees the product's quality on the other.

The analysis of thin layers or coatings is a common task in Micro-XRF spectrometry. Both the non-destructive nature of the method and the ability of X-rays to penetrate into the sample and obtain information on the material beneath the surface make this method attractive for the purpose of analyzing single or multiple layers. With the polycapillary optic of the M4 TORNADO an exciation spot of <20 µm can be achieved, enabling a layer thickness analysis with high spatial resolution.

This lab report describes the analysis of a glass substrate coated with a copper-aluminum layer to determine the layer thickness and the Cu:Al ratio across the sample. Additionally, map results were compared with single point measurements.

Functional principle

Due to their high photon energy, X-rays have the capability to penetrate matter. On their way through the material they induce characteristic X-ray fluorescence. This fluorescence in turn can leave the sample.

In a coated structure with sufficiently thin layers, fluorescence is produced in all layers and radiated back out of the sample (Fig. 1). These secondary X-rays can be detected and the ratios of the fluorescence lines from the individual layers can be used for the calculation of the thickness and composition of each layer.

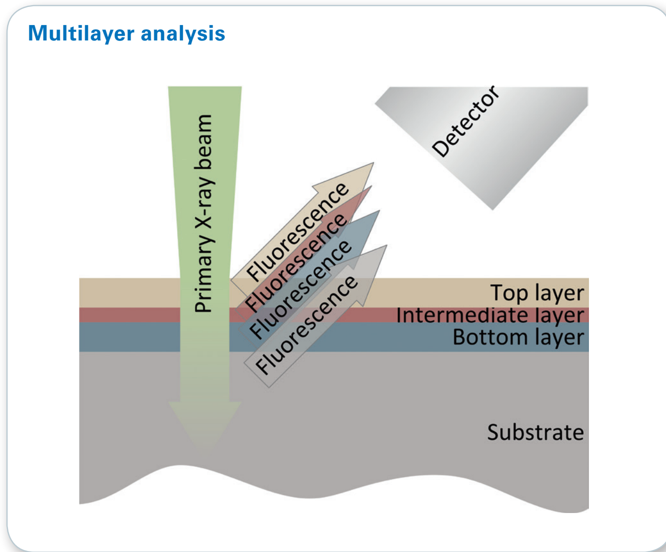


Fig. 1 Functional principle of layer fluorescence

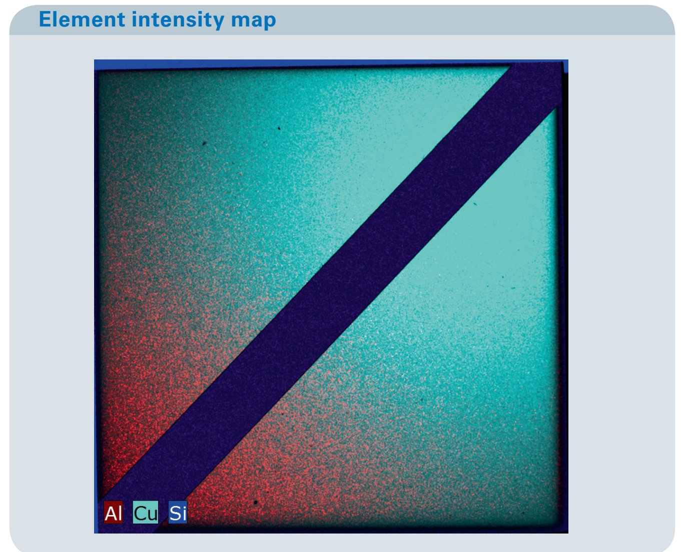


Fig. 3 Elemental distribution of the Cu and Al coated glass substrate

Sample

The sample to be analyzed is a 5 cm x 5 cm glass substrate coated with a copper-aluminum layer. It was produced by magnetron sputtering of a dual target containing Cu and Al.

Measurement conditions

The measurements were performed with a Bruker M4 TORNADO equipped with a Rh X-ray tube and a polycapillary lens. The instrument combines high spatial resolution with fast data processing and a motorized high-speed X-Y-Z stage for sample positioning. The following standard measurement conditions were used:

- tube voltage of 50 kV
- current of 200 µA
- no primary beam filter
- chamber pressure of 20 mbar.

Two relatively large coated regions are well separated by an uncoated area which runs diagonally across the sample. The analytical task calls for a spatially resolved quantitative analysis of the thin metallic layer.

In the Area workspace of the M4 TORNADO software, an overview of the sample, which can be seen in Fig. 2, was generated using the Mosaic function. A map area of 1000 x 1000 pixels (green frame) was defined with a pixel size of 50 µm and a dwell time of 50 ms/pixel. The measurement required a total time of 15 hours to complete.

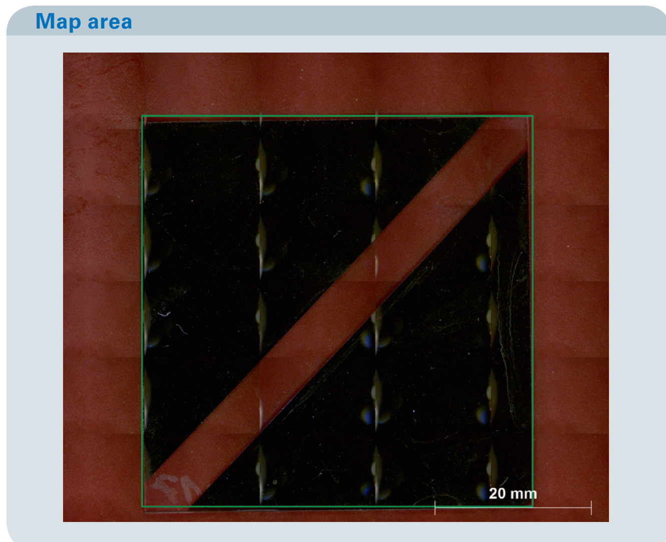


Fig. 2 Mosaic sample image with defined map area

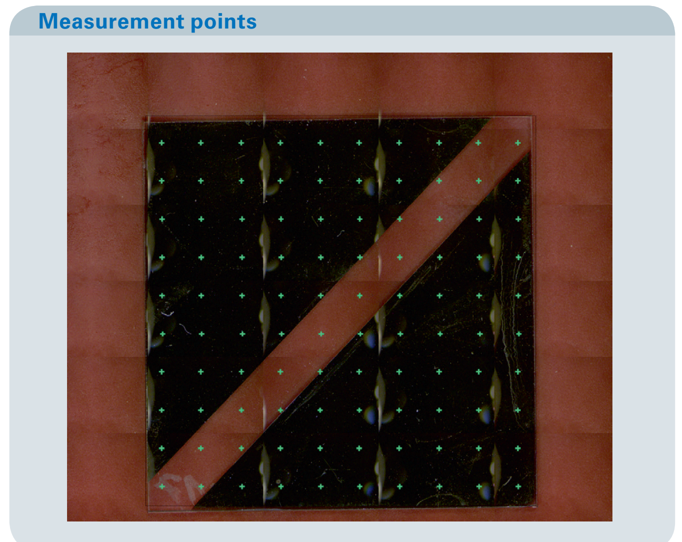
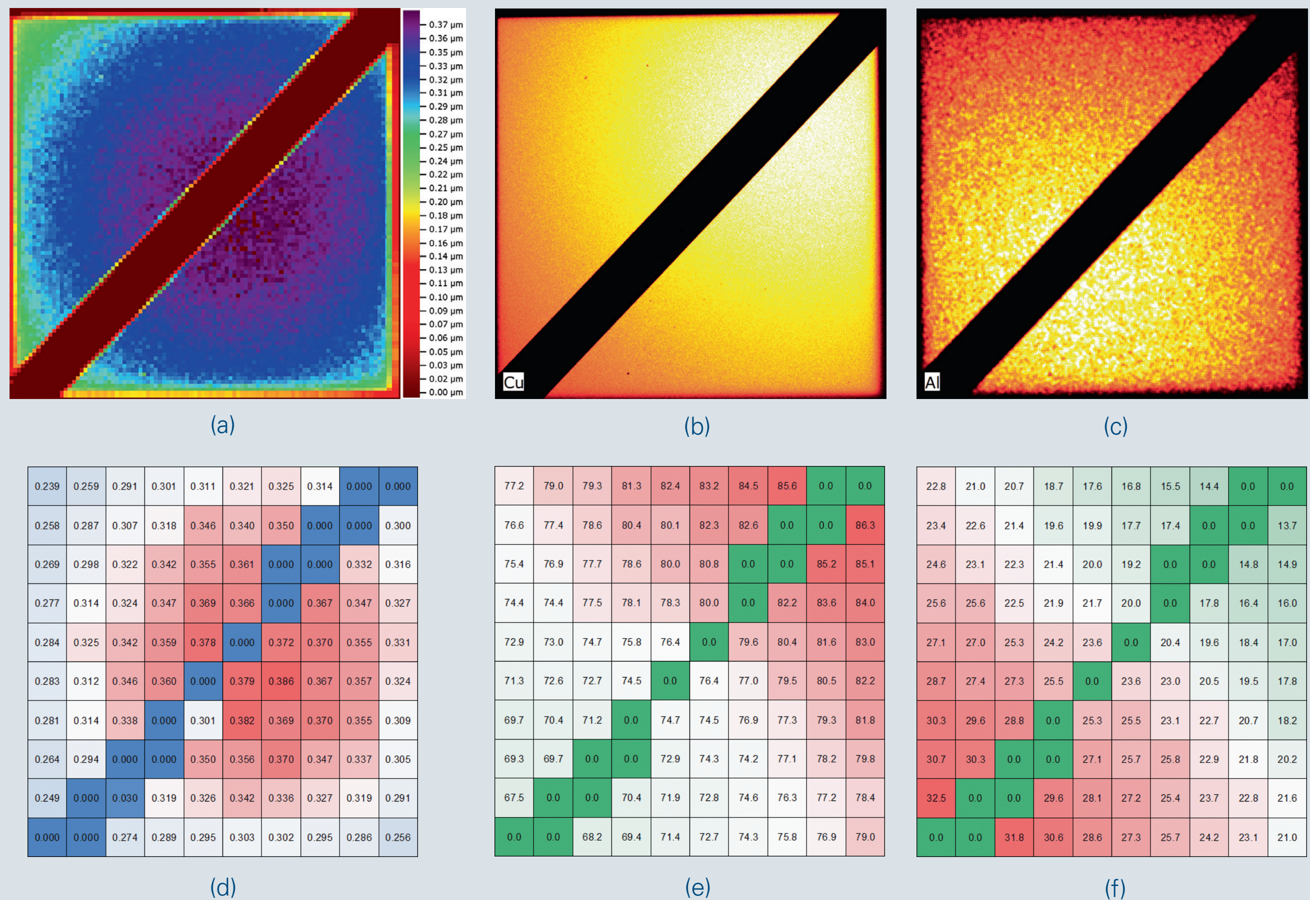


Fig. 5 Mosaic sample image with defined single measurement points



0.239	0.259	0.291	0.301	0.311	0.321	0.325	0.314	0.000	0.000
0.258	0.287	0.307	0.318	0.346	0.340	0.350	0.000	0.000	0.300
0.269	0.298	0.322	0.342	0.355	0.361	0.000	0.000	0.332	0.316
0.277	0.314	0.324	0.347	0.369	0.366	0.000	0.367	0.347	0.327
0.284	0.325	0.342	0.359	0.378	0.000	0.372	0.370	0.355	0.331
0.283	0.312	0.346	0.360	0.000	0.379	0.386	0.367	0.357	0.324
0.281	0.314	0.338	0.000	0.301	0.382	0.369	0.370	0.355	0.309
0.264	0.294	0.000	0.000	0.350	0.356	0.370	0.347	0.337	0.305
0.249	0.000	0.030	0.319	0.326	0.342	0.336	0.327	0.319	0.291
0.000	0.000	0.274	0.289	0.295	0.303	0.302	0.295	0.286	0.256

(d)

77.2	79.0	79.3	81.3	82.4	83.2	84.5	85.6	0.0	0.0
76.6	77.4	78.6	80.4	80.1	82.3	82.6	0.0	0.0	86.3
75.4	76.9	77.7	78.6	80.0	80.8	0.0	0.0	85.2	85.1
74.4	74.4	77.5	78.1	78.3	80.0	0.0	82.2	83.6	84.0
72.9	73.0	74.7	75.8	76.4	0.0	79.6	80.4	81.6	83.0
71.3	72.6	72.7	74.5	0.0	76.4	77.0	79.5	80.5	82.2
69.7	70.4	71.2	0.0	74.7	74.5	76.9	77.3	79.3	81.8
69.3	69.7	0.0	0.0	72.9	74.3	74.2	77.1	78.2	79.8
67.5	0.0	0.0	70.4	71.9	72.8	74.6	76.3	77.2	78.4
0.0	0.0	68.2	69.4	71.4	72.7	74.3	75.8	76.9	79.0

(e)

22.8	21.0	20.7	18.7	17.6	16.8	15.5	14.4	0.0	0.0
23.4	22.6	21.4	19.6	19.9	17.7	17.4	0.0	0.0	13.7
24.6	23.1	22.3	21.4	20.0	19.2	0.0	0.0	14.8	14.9
25.6	25.6	22.5	21.9	21.7	20.0	0.0	17.8	16.4	16.0
27.1	27.0	25.3	24.2	23.6	0.0	20.4	19.6	18.4	17.0
28.7	27.4	27.3	25.5	0.0	23.6	23.0	20.5	19.5	17.8
30.3	29.6	28.8	0.0	25.3	25.5	23.1	22.7	20.7	18.2
30.7	30.3	0.0	0.0	27.1	25.7	25.8	22.9	21.8	20.2
32.5	0.0	0.0	29.6	28.1	27.2	25.4	23.7	22.8	21.6
0.0	0.0	31.8	30.6	28.6	27.3	25.7	24.2	23.1	21.0

(f)

Fig. 4 Area scan results. a) Quantified layer thickness along the sample, b–c) Qualitatively measured element intensity of Cu and Al, d) Quantification results of the point measurements for the layer thickness in µm, e) Mass fraction of Cu in wt.%, f) Mass fraction of Al in wt.%

High resolution map results

Fig. 3 shows the qualitative layer composition of the Cu-Al layer and Si in the glass substrate. The gradient of the Cu:Al ratio is clearly recognizable. However, no layer thickness information can be seen in this map. Therefore, by means of the new XMethod software tool, an analysis method is set up to calculate the layer thickness.

Fig. 4a displays a false color rendering of the layer thickness map using 10 x 10 pixel binning. This results in a 500 µm spatial resolution with an overall measurement time of 5 s per binned pixel.

The center of the radially symmetric layer thickness gradient is not exactly in the middle of the sample but is slightly offset to the right. The maximum layer thickness is calculated to be 380 nm, and the minimum at the edges of the sample is only 270 nm.

Fig. 4b and 4c show the false color presentations of the measured element intensities for Cu and Al. The highest intensity for Cu is in the top right corner of the sample. The maximum Al signal is not found in the bottom left corner but slightly shifted to the right.

Quick Multi-Point measurement results

Instead of acquiring map data for many hours, a smaller number of points can be set in a grid and analyzed for thickness. Using a measurement time of 10 seconds per point over a 10 x 10 matrix (Fig. 5) allows a quick overview of the sample's layer thickness in approximately 20 minutes.

After creating the measurement method in the XMethod editor and recording and quantifying the point spectra, the individual measurement results were exported to MS Excel® and visualized as shown in Fig. 4d–4f. The different colors emphasize the variation of the thickness of the layer and its elemental composition.

The layer thickness measurement results as shown in Fig. 4d exactly match the results of the map data. The point measurement results for the Cu concentration in the layer given in Fig. 4e show maximum values for the top right corner of the sample. This is in line with the qualitative element intensities displayed in Fig. 3.

Fig. 4f displays the Al concentrations at 100 measurement points. The maximum again is found in the lower left corner of the sample. This is in agreement with the qualitative results. The decrease of the measured Al intensity is more attributed to the lower layer thickness in this area than to the change in the element ratio. Altogether the Cu:Al ratio changes from 86:14 in the sample's top right corner to 67:33 in the bottom left.

Conclusion

Regardless of the analytical approach of full area scans and single point measurements, identical layer thickness values were found. Hence, the combination of M4 TORNADO with the optional XMethod tool provides a useful tool to analyze and draw well founded conclusions on thin metallic layers.

It is noteworthy that a stable quantitative fundamental parameter analysis of the Cu-Al layer is possible with spectra which were accumulated over only 5 s.

A higher spatial resolution is, as usual, at the cost of measurement time. However, it may be needed for samples where the layer thickness varies over short distances. For quality assessment, measuring a coarser grid is usually sufficient and more time effective.

Acknowledgements

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