A Methodology to Obtain Traceability for Standards for the Mass per Unit Area of Thin Metal Alloy Layers for X-Ray Fluorescence Tools

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Statistic process control as well as process capability demand for calibrated determination of layer thicknesses in various industries, e.g. automotive, aerospace, microelectronics manufacturing. Calibration requires well known and well characterized samples. A calibration laboratory accredited according to DIN EN ISO 17025 has the objective to distribute standards traceable to SI units to industrial laboratories for quality control of manufacturing of various products. Especially, the thickness determination of thin metallic coatings e.g. from galvanic processes or layer deposition using X-Ray Fluorescence (XRF) can be significantly improved by customized calibration samples. This is essential as the measurement uncertainty directly correlates to the capability of reliable process control. For calibration laboratories, the validation of results using round robins and the direct comparison to national metrology institutes is a prerequisite to demonstrate the competence to do calibration services.

Fundamental parameter based XRF was used to determine the mass per unit area of very thin layers below 100 nm e.g. Au, Ti, or Ta. Proficiency testing with a national metrology institute was successfully applied to validate those results. The measurement uncertainty was improved by an accredited gravimetric method.

For alloy layers e.g. Ni alloys the determination of measurement uncertainty as well as proficiency testing is much more complex. In this paper a strategy to obtain traceability for thin alloy layers as well as results from round robins and proficiency testing are presented. The combined use of the accredited method for determination of mass per area from measurement of mass and area combined with standard free XRF [1] as well as chemical analysis of dissolved samples is deployed for alloys as Ni_xZn_{1-x} as well as Ni_xP_{1-x}. The obtained results are compared to reference free XRF at the BESSY II laboratories of Physikalisch-Technische Bundesanstalt. An excellent agreement of the obtained measured values as mass per unit area and alloy concentrations from the different applied methods within the measurement uncertainty was observed for both Ni alloys, respectively. These results demonstrate the successful performed traceability of the standards to SI units in combination with a validation of results by national metrology institutes and the round robin approach.

[1] V. Rößiger, B. Nensel, Analysis of Layers, Handbook of Practical X-Ray Fluorescence Analysis edited by B. Beckhoff, B. Kanngießer, N.Langhoff, R. Wedell, and H. Wolff, Springer-Verlag, Berlin Heidelberg, ISBN-10 3-540-28603-9, 2006, pp. 554