Re-calibration and characterization of Ti secondary reference standards

Abstract:

Previously used Ti secondary reference standards and Ti foils with nominal thicknesses in the range of 0.4 – 7 µm have been (re-)calibrated using new Ti primary standards (**BAY Ti 150812, BBB Ti 191012**) and the Ti reference standard **ACHVY**, both characterized by gravimetric measurements.

Experimental details

XRF measurements have been carried out using a Fischerscope XUV. Experimental parameters are summarized in Tables I and II, for the measurement of the primary reference standards BAY Ti 150812, BBB Ti 191012 and the secondary reference standards or Ti foils, respectively. The WinFTM scanning mode in combination with a 20x20 x-y run was used to cover almost the whole surface of the primary reference standard. All samples were placed on a purpose-built sample holder ("radiation trap") for absorbing X-rays from the excitation of other materials, e.g. the surrounding vacuum chamber.

Parameter	Value	Comments
Device	Fischerscope© XUV-S	
Voltage, Filter	50keV, Ni 10 µm primary filter	
Aperture collimator	1 mm	
Software version	6.29	
Spots per sample	400	x-y-scan mode
Duration per spot	22 s	
Measured area	50 x50 mm	20x20 Matrix
Anode current	1000 mA	

Table I : experimental parameters for the XRF measurement of the Ti primary reference standards BAY Ti 150812, BBB Ti 191012.

For each of the secondary reference standards and Ti foils to be characterized 9 individual runs each with 60s measuring time have been performed with a spatial distribution covering a 3x3 matrix with 2mm x 2mm outer dimension in the central area of the samples.

Parameter	Value	Comments
Device	Fischerscope© XUV-S	
Voltage, Filter	50keV, Ni 10 µm primary filter	
Aperture collimator	1 mm	
Software version	6.29	
Spots per sample	9	
Duration per spot	60 s	
Measured area	2 x 2 mm	3x3 Matrix
Anode current	1000 mA	

Table II : experimental parameters for the XRF measurement of the Ti secondary reference standards and Ti foils.

Data analysis and results

The results of the Ti primary samples (Bay, BBB), the reference standard ACHVY and measurements with an empty sample holder (NULL) for zero compensation are summarized in Table III. The relation between gravimetric data and data from XRF measurements is calculated on the basis of a weighted linear regression with consideration of the individual uncertainties.

	XRF		Gravimetry	
Sample	**d [µm]**	**σ_d [µm]**	**d [µm]**	**σ_d [µm]**
Zero	0,002	0,001	0,000	--
ACHVY	1,189	0,007	1,112	0,006
BAY-Ti	5,263	0,011	4,941	0,009
BBB-Ti	6,733	0,010	6,368	0,002

Table III : Comparison of experimental results from XRF and gravimetric measurements. The thickness from the gravimetric data was calculated using the mass per unit area data and a density of ρ(Ti)= 4.51 g·cm^{-3}. For XRF data the uncertainties are given as standard error (k=1) for 400 measurements in case of BAY-Ti and BBB-Ti and 9 measurements for Zero and ACHVY, respectively.

In Figure 1 the absolute deviation of the XRF measurements from the gravimetric values is plotted against the (gravimetric) reference exhibiting a linear trend.

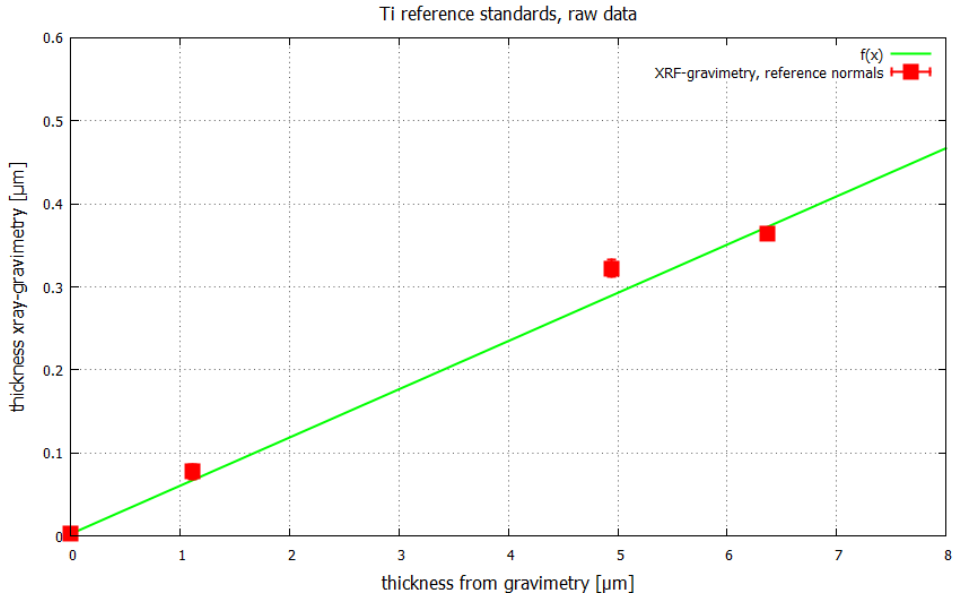

Fig.1 : Comparison of the absolute deviation of XRF results from gravimetric values. The regression line is drawn to guide the eye.

The thicknesses from XRF measurements of the individual samples were calibrated with respect to the gravimetric measurements of the primary reference standards (BAY Ti 150812, BBB Ti 191012 and ACVHY) using the calculated regression coefficients.

Table IV summarizes the re-calibrated or newly calculated thicknesses of the samples which will now regarded as secondary standards due to their direct relation with the BAY Ti 150812 and BBB Ti 191012 primary standards.

Code	measured [µm]	statistical uncertainty	calibrated [µm]	u. (k=2) [µm]
ADXRF	6,946	0,056	**6,563**	**0,053**
ADXRG	7,032	0,037	**6,644**	**0,035**
ADXRH	6,813	0,045	**6,436**	**0,043**
ADXRI	6,734	0,056	**6,362**	**0,053**
ADXRJ	6,873	0,051	**6,494**	**0,048**
ACVIK	0,442	0,002	**0,415**	**0,010**
AALTH	0,939	0,006	**0,886**	**0,012**
ACHWB	1,193	0,008	**1,126**	**0,032**
ACHWA	1,181	0,018	**1,114**	**0,034**
ACHVZ	1,172	0,015	**1,105**	**0,028**
AALBJ	2,086	0,015	**1,969**	**0,028**
AAYEJ	2,328	0,013	**2,198**	**0,024**
AAYEK	2,256	0,014	**2,130**	**0,028**
AAYEP	2,234	0,013	**2,109**	**0,026**

Table IV : Summary of measured data and results from (re-)calibration of the new secondary reference standards (in µm).

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