Re-calibration of self-supporting Cr foils with Cr/Fe reference standards

Abstract:
Self-supporting Cr foils used as Fischer secondary reference standards have been re-measured and calibrated using traceable Cr/Fe reference standards.

Experimental details

The availability of new Cr/Fe reference standards which are traceable to ICP-OES measurements (see Fischer Traceability Report JL-2012-11-Cr/Cu-Cr/Fe) permits a re- analysis and accurate check of existing self-supporting Cr-foils used as secondary reference standards. For this purpose XRF measurements have been carried out using a Fischerscope XDV-SDD and data of the foils were compared with the new Cr/Fe standards. The experimental parameters are summarized in Table I. For the measurement of all standards a 4x4 matrix of 16 equidistant measurement spots covering an area of 2 mm x 2 mm in the central region of the primary standards was defined. The dimension of all reference materials is 8mm x 8mm. The data were obtained as mean value of the 16 individual measurements per standard.

Parameter	Value	Comments
Device	Fischerscope© XDV-SDD	
Voltage, Filter	50keV, Ni 10 µm primary filter	
Aperture collimator	0.3 mm	
Software version	6.29	
Spots per sample	16	
Duration per spot	45 s	
Measured area	2 mm x 2 mm	4x4 Matrix
Anode current	1000 mA	

Table I : experimental parameters for the XRF measurement of the Cr/Fe standards and Cr foils.

The Cr foils have been placed on a massive block of iron to meet the conditions for the calibration with the Cr/Fe standards.

Data analysis and results

Table II summarizes the results of the thickness of the Cr layer from XRF measurements of the Cr/Fe reference standards. The values were obtained from the universal standard free XRF fundamental parameter method. The XRF uncertainty given (σ-XRF) is the absolute standard error of 16 individual measurements.

Material	Sample	nominal thickness [µm]	σ [µm]	thickness XRF FP [µm]	σ -XRF [µm]
Fe	-	0	0	0.000	0.0002
Cr/Fe	ADYBX	2.02	0.01	2.044	0.001
Cr/Fe	ADYBW	2.03	0.01	2.021	0.002
Cr/Fe	ADYBV	2.06	0.01	2.081	0.002
Cr/Fe	ADYBU	2.06	0.01	2.072	0.003
Cr/Fe	ADYCE	3.99	0.02	4.051	0.004

Table II : Summary of the nominal values and XRF measurements of the Cr layer using the universal standard free fundamental parameter method for the primary Cr/Fe standards serving as reference for the calibration of the Cr foils.

A graphical representation of the data is shown in Fig.1 where the absolute differences of XRF data to the nominal values are plotted against the XRF data. The overall agreement of the XRF FP data with the nominal values is excellent with a largest deviation of only 1.5 %.

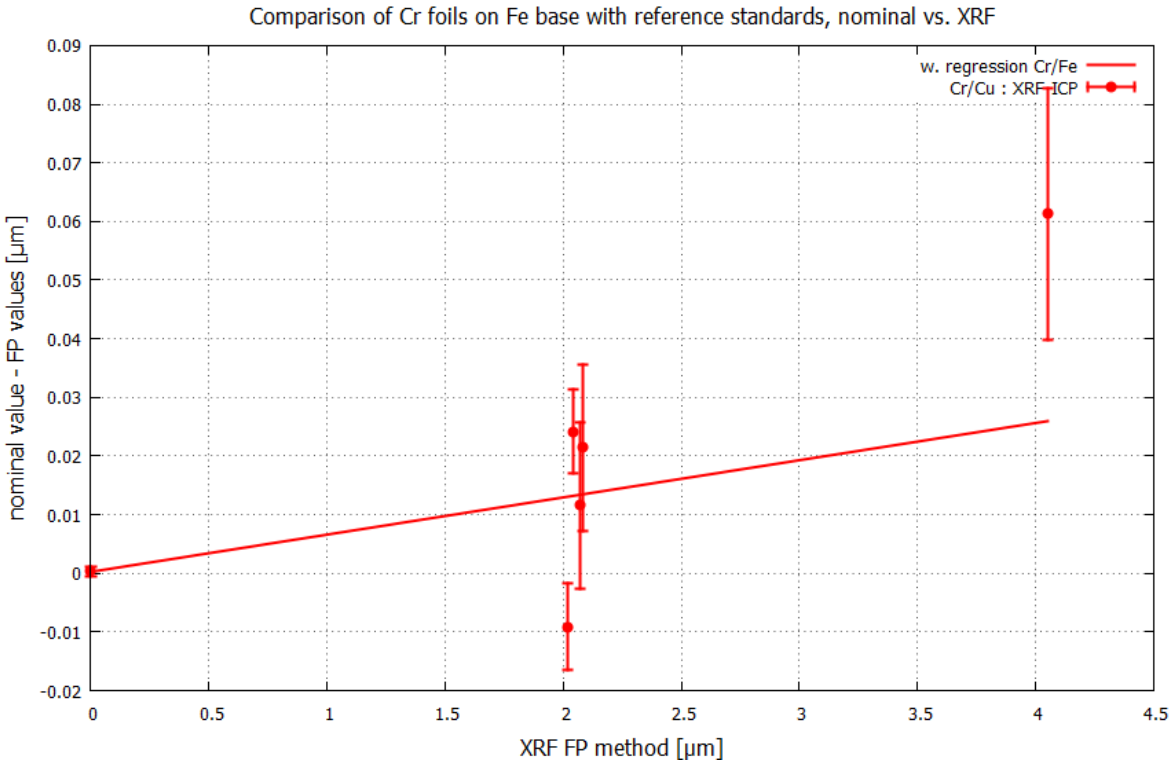

Fig 1 : Comparison of the absolute difference between nominal values of the Cr layer on the Cr/Fe reference standards from tab. II and XRF data obtained from the universal standard free XRF fundamental parameter method plotted (XRF FP) vs. XRF FP data. A weighted linear regression line is drawn to guide the eye.

From a weighted linear regression a direct linear correlation between nominal and standard free XRF data was obtained and used for the re-calibration of the Cr foils. Results of the measurements and the calibration are summarized in Table III.

Summary of standard free XRF results and calibrated values of the Cr foils

Code	nominal thickness [µm]	XRF FP [µm]	calibrated thickness [µm]	U (95% CF) [µm]	U [%]
AATHT	0,14	0,169	0,167	0,005	3,2
AAUGX	0,23	0,245	0,243	0,007	2,7
AAUHD	0,57	0,601	0,596	0,008	1,4
AAUGU	0,23	0,247	0,245	0,006	2,5
AAUGZ	0,25	0,259	0,257	0,006	2,4
AAUHF	0,53	0,555	0,55	0,01	1,5
ACGSP	0,75	0,735	0,73	0,01	1,4
AAGWG	0,94	0,958	0,95	0,01	1,4

Table III : Nominal thickness, XRF FP values and re-calibrated results of the Cr foils

Except for sample AATHT and AAUHD the calibrated results agree within errors very well with the stated values.

The same approach as been used for self-supporting Cr foils of set 19789 (Cr/Ni/Fe). A summary is given in Table IV where the upper part contains the results for the reference standards compared to the values of the standard free XRF values and the lower part the calibrated results of the Cr foils.

Code	XRF sf µm	SD µm	U(k=2) µm	Nominal µm	calibrated	U(k=2) µm
Fe	-0,003	0,001	0,0003	0		
ADYBX	2,06	0,006	0,003	2,02		
ADYBW	2,02	0,006	0,003	2,03		
ADYCI	4,04	0,013	0,007	4,01		
ADYCK	4,10	0,017	0,009	4,09		
AAUGU	0,24	0,003	0,002	0,23	0,24	0,02
AAUGZ	0,26	0,002	0,001	0,25	0,26	0,02
AAUHF	0,55	0,003	0,002	0,53	0,55	0,03
ACGSP	0,74	0,006	0,003	0,75	0,73	0,06
AAGWG	0,96	0,007	0,004	0,94	0,95	0,07

Table III : Nominal thickness, XRF FP values and re-calibrated results of the Cr foils of set 19789.

Here, the agreement is excellent.

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