Fischer Traceability Report VR 2011 01

On the Re-calibration of Au-Layer Master Reference Standards

Helmut Fischer GmbH uses master reference standards to quantify "standard calibration set" products. This report describes the re-calibration of several sets of master reference standards for Au layers on top of various substrate materials. Massive Ni, Ni coated Cu, standard PCB material (Cu layer coated with NiP over epoxy with glass fibre and Br), and flexible plastic PCB material are included, as well as self-supporting foils.

Basic reference material

The above-mentioned master samples are calibrated with an ensemble of reference material (Table 1) including:

Type A: Self-supporting Au-foils quantified according to a gravimetrical procedure accredited by either DKD or DAkkS /1/.

Type B: A set of reference materials quantified by a combination of gravimetry /1/, FP-based XRF analysis /2/, and Rutherford backscattering /3/.

Experimental

Fischerscope© XDAL, 50 kV, 1 mm Al-primary filter. Aperture Ø0.6 mm.

The foils samples were measured with a special "radiation trap" sample holder.

Unless otherwise indicated: 9 measurements with 120 s uniformly distributed over an area of 2 mm x 2 mm.

Since the measuring distance affects the geometry factor, it must be fixed within a small tolerance. The Fischerscope© X-ray system's autofocus tool achieves an acceptable range of about 20-50 µm.

The measurements were each repeated several times to ensure consistency. Over the course of these experiments (3 days) no drift was detected.

No.	Code	Thickness (nm)	Standard measuring uncertainty (nm)
1	ABVAZ	1494	5
2	ABVBQ	2300	10
3	ABVGH	4270	25
4	ACXYI	213,8	3
5	ACXYJ	496.8	5
6	ACXYK	117.5	1.3
7	ACXYL	114.1	1.3
8	ACXYQ	48.1	1
9	ACXYR	44	1
10	ACXYS	45.8	1
11	ACXYT	11.8	0.3
12	ACXYP	28.4	0.6

Table 1 *Au thickness on the reference samples used for re-calibration. Nos. 1-3 are self-supporting foils (Type A). The rest (Nos. 4ff) are Type B. The unit of measure is nm, whereby thickness values were converted from mass per unit area data assuming a density of 19.32 g/cm^3.*

Spectrum evaluation (DefMA, *cf.* /2/)

The Au thickness or the Au mass per unit area is determined from the Au-L characteristic radiation. The evaluation setup takes into account the type of substrate material, as well as the enhancement effect due to an intermediate Pd layer which occurs in some of the Type B samples in Table 1. Enhancement from other base material elements does not occur. For control purposes, various uncoated samples were checked for Zero.

Results of re-calibration

All values are nm of Au, converted from the mass per unit area data with the density of Au = 19.32 g/cm^3.

1. "Au/Ni/Cu" Ser. No. 19372 "Flexible PCB"

Code	Old stated value	Re-calibrated	Standard measuring uncertainty (k=1)
ACXXL	0	**0**	0
ACXXW	52.3	**51.1**	1.7
ACXXT	138.9	**142.0**	3
ACXXU	140.4	**142.6**	2
ACXXS	139.3	**142.8**	2
ACXXY	54	**52.7**	1
ACXXZ	52.2	**50.4**	1
ACUQN	142.2	**143.1**	2

2. Self-supporting Mylar foils, Au sputtered

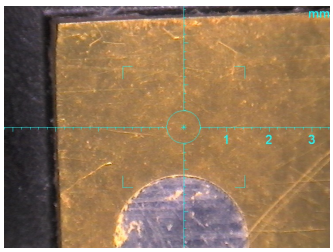
Code	Old stated value	Re-calibrated	Standard measuring uncertainty (k=1)
ACGVD	54.5	**53.4**	1.5
ACGVM	109.3	**107.1**	2
ACGVQ	258.2	**260.0**	3
ACGVT	510.5	**520.0**	6

3. "Zweischicht Master" XX27

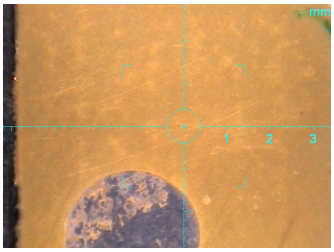
Code	Old stated value	Re-calibrated	Standard measuring uncertainty (k=1)
XX27-2	1710	**1711**	20

4. Master set "Au/Ni/Cu"

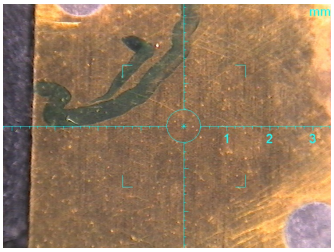
This set consists of nine double-layer samples which have been analysed by coulometry. Hence, they all have at least one dissolution spot. Therefore, the current measuring positions are defined by the following figures.



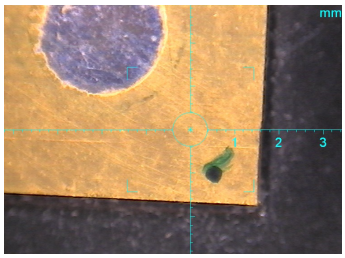
AASDK



AASDR



AASDP



AASDM

Code	Old stated value	Re-calibrated	Standard measuring uncertainty (k=1)
AASDK	580	**589**	5
AASDR	1360	**1346**	15
AASDP	1180	**1205**	14
AASDM	900	**899**	10

5. BCR-Set

Probe Code	Old stated value /4/	Re-calibrated	Standard measuring uncertainty (k=1)
BCR I	447	**447**	6
BCR II	813	**787**	10
BCR III	3014	**3013**	35

These reference samples have been worked out by the European Bureau of Certified Reference based on round robin tests /4/.

6. Au/Ni GN 243 DKD-K33101, Ser. No. 16526

Probe Code	Old stated value	Re-calibrated	Standard measuring uncertainty (k=1)
ABVAU	1461	**1453**	14
ABVBS	2310	**2296**	25
ABVGI	4250	**4250**	35
ABVUX	6476	**6525**	120

7. Au/Ni GN 653 DKD-K33101, Ser. No. 19285

Probe Code	Old stated value	Re-calibrated	Standard measuring uncertainty (k=1)
ADGNV	1310	**1322**	14
ADGNQ	2320	**2299**	25

This set is based on gravimetry according to /1/. The deviations between old and re-calibrated values stem from the inhomogeneity of the rolled foil material.

Discussion

As shown in the tables above, in most cases the re-certified values are very close to the old ones. The differences are of the same order of magnitude as the measuring uncertainties. The reasons for some remaining (small) discrepancies cannot be explained in each case, the spatial inhomogeneity is probably an important issue which has not previously been accounted for. Consequently, the re-calibrated results are mean values valid for the very restricted area of just 2 mm x 2 mm.

Further basic reference material will be produced in future to achieve improved data with smaller measuring uncertainties.

References

/1/ Reg. No. D-K-15076-01-00.
/2/ V. Rößiger and B. Nensel, in “Handbook of practical X-Ray fluorescence analysis”, Springer 2006, p. 554.
/3/ S. Dill and V. Rößiger, Circuit world, 37 (2011), 2, 20.
/4/ Certification report BCR CRM 328 (EUR 11564) 1988.