Fischer Traceability Report SD 2012 01

Recertification of master standards with Pd layers

The Helmut Fischer GmbH applies master reference standards to quantify “standard calibration set” products. This report describes the recertification of master reference standards including Pd layers up to a thickness of 5 µm (Au on Pd on Ni on CuFe, Au on Pd on standard PCB material, which consists of a Cu layer coated with NiP over epoxy with glass fibre and Br, self-supporting Au/Pd/Ni foils and and self-supporting Pd foils.

Basic reference material

The above-mentioned master samples were recalibrated with an ensemble of traceable reference material (table 1) with low uncertainties including:

Type A: Self-supporting Pd foils quantified according to a gravimetric 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/.

***Table 1**: Pd thickness of reference master samples used for the recertification. No. 1-3 are self-supporting foils (Type A). All other samples are Type B samples. Thickness values (given in nm) were converted from mass per unit area data assuming a density of 12 g/cm^3.*

Code	Pd in nm			
	specified	u (k=1)	corrected value	u (k=1)
Type A:				
New primary reference foil PN_Pd_01_2012	4975	3	4983	27
ACIMJ (17544 GN)	2200	12,5	2199	17
ACILS (17544 GN)	4806	18,3	4800	35
Type B:				
ACXYR	92,1	0,9	91	2
ACXYO	333,2	2,6	330	4
ACXYQ	21,1	0,8	22	2
ACXYS	331,7	2,7	326	3
ACXYT	18,7	0,4	20	2

ACXYN	87,3	0,9	88	2
ACXYM	21,6	0,6	22	2

Experimental
Fischerscope© XDAL, 50 kV, 1 mm Al primary filter. aperture Ø 0.6 mm, DefMA Au/Pd/ (with background correction)
The self-supporting foils were measured with a special “radiation trap” sample holder.

All samples except of the new primary reference foil PN_Pd_01_2012 were measured with 9 measurements with 120 s uniformly distributed over an area of 2 mm x 2 mm.
The new primary reference foil was measured with 400 measurements with 120 s uniformly distributed over an area of 5 cm * 5 cm.
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 constant measuring distnce of about ±20 µm.

The measurements were repeated several times to ensure consistency. For control purposes, various uncoated samples were checked also.
Over the time period of the experiment (3 days) no drift was detected.

Spectrum evaluation (DefMA, *cf.* /2/)
The Pd thickness or the Pd mass per unit area is determined from the Pd-K characteristic radiation. The evaluation setup takes into account the type of substrate material, as well as the absorption effect due to a gold top layer which occurs in some of the samples. Enhancement from other base material elements does not occur.

Results of recalibration
All values for Pd are converted from mass per unit area into thickness data assuming a density of Pd = 12 g/cm^3. The given uncertainties are standard measuring uncertainties for a confidence level 68 % (k =1).

1. Self-supporting Au/Pd/Ni foils

Code	Pd in nm			
	old value	u (k=1)	new value	u (k=1)
ADDZM	99	3	97	3,5
ADBFA	21	1,5	21	1,5
ADDZV	250	5	244	5
ADBES	62	3	62	3
ADDZU	100	3	98	3,5

ADBFH	21	1,5	21	1,5
ADDZL	247	5	244	5
ADBFM	61	3	62	3

2. Au/Pd/Ni/CuFe

Code	Pd in nm			
	old value	u (k=1)	new value	u(k=1)
Master4	7,6	0,4	9	2
Master9	106	4	109	2
Master8	31,2	1,5	34	2
Master5	31,2	1,5	33	2
Master6	106	4	112	2
Master7	7,6	0,4	8	2

3. Self-supporting Pd foils (set 17543 GN)

Code	Pd in nm			
	old value	u *(k=1)	new value	u (k=1)
ACPME	51	-	54	2
ACPLX	102	-	106	2
ACPLT	256	-	261	3
ACPKY	504	-	507	5
ACPLP	1474	-	1480	9
ACPLK	2479	-	2470	14
ACPKX	503	-	506	4
ACNOM	505	-	510	4
ACLVW	1580	-	1559	9
ACIMK	2290	-	2282	14

*No standard measurement uncertainties were given for the old values

4. Self-supporting Pd foils (set 17544 GN)

Code	Pd in nm			
	old value	u* (k=1)	new value	u (k=1)
ACPLS	249	-	260	3
ACOSQ	98	-	104	2
ACPLA	504	-	511	4
ACLVX	1569	-	1546	11

*No standard measurement uncertainties were given for the old values

Discussion

As shown in the tables above, in most cases the recertified values are very close to the old specified ones. The differences are usually in the same order of magnitude as the measuring uncertainties. Although the reasons for these discrepancies cannot be explained in each case, the spatial inhomogeneity is probably an important issue which has not previously been accounted for. Consequently, the recalibrated 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.