Application Brief

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Investigating the limit of determination and uncertainty in ultra-thin Pd film measurements

1. Overview

This application brief reports on our investigation into quantitative limits and uncertainty when measuring ultra-thin Pd films 0.1 um or less in thickness using the SEA5000 manufactured by Hitachi High-Tech Science.

2. Quantitative Limits

(a) Definition

Limit of determination is defined by the following expression,

$$DL = 10\sigma_{BG}/m$$

where m is the slope of the calibration curve and σ_{BG} is the background standard deviation.

(b) Calibration Creation

Because the relationship of Pd film thickness and Pd X-ray intensity in an ultra-thin Pd film of 0.1 um or less can be approximated in a single equation, a linear calibration curve is created with a base and Pd 0.11 um. Figure 1 shows the calibration curve.

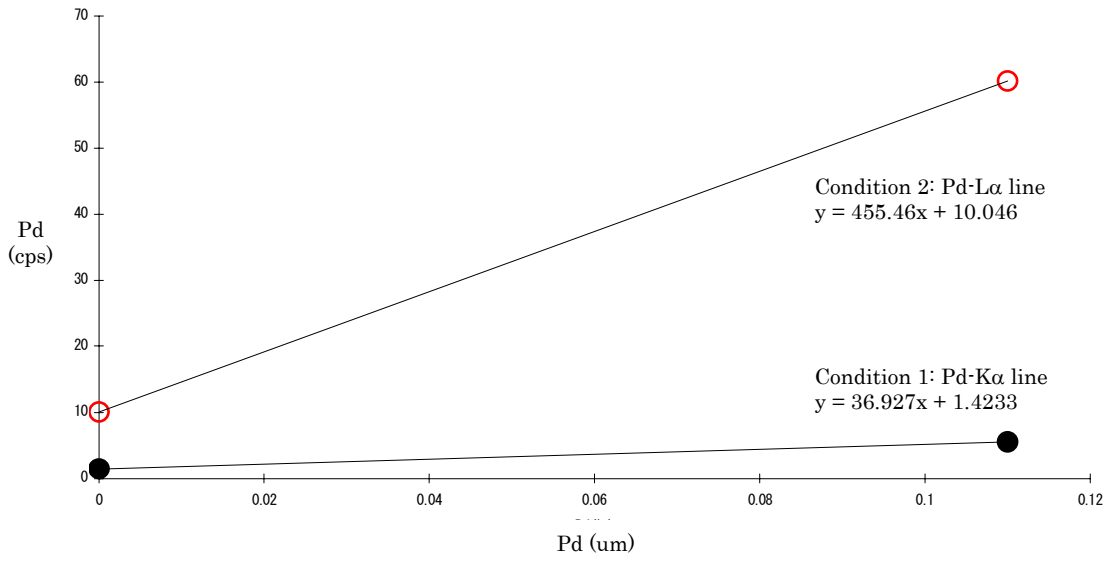

Figure 1 Calibration Curve

(c) Measurement conditions are shown in table 1.

Table 1 Measurement Conditions

	Condition 1	Condition 2
Collimator Size (mm)	0.1	1.8
Voltage (kV)	50	15
Current (uA)	1500	76
Analytical Line	Pd-Kα line	Pd-Lα line
Sample Chamber	Air	Vacuum
Measurement Time (sec)	300	300

(d) Limit of determination

Limit of determination is shown in table 2. Here, background standard deviation is the actual measured value obtained from 10 measurements.

Table 2 Limit of Determination

	Condition 1	Condition 2
m	36.927	455.46
σ_{BG} (cps)	0.054	0.254
DL (um)	0.015	0.0056

3. Uncertainty

In this section we will consider uncertainty when measuring ultra-thin Pd film thickness by the fluorescent X-ray method.

(a) Method

Data for a Pd film thickness of 0.1 um or less can be output with a two-point calibration curve using the base and Pd 0.11 um. Pd film thickness t_0 is given in equation (1) below.

$$t_0 = \frac{I_0 - I_1}{I_2 - I_1} \times t_1 \quad (1)$$

I_0 = X-ray intensity of unknown sample
I_1 = Background intensity of base measurement
I_2 = X-ray intensity of Pd 0.11 um measurement
t_1 = Pd 0.11 um

(b) Measurement by fluorescent X-ray method

Table 3 shows the measured intensity of 10 measurements of 0.11 um and base, as well as a 0.05 um sample.

Table 3 Measurement Conditions

	Condition 1			Condition 2		
	0.11μm	0μm	unknown	0.11μm	0μm	unknown
Average	5.485	1.423	3.158	60.1465	10.0462	33.2315
Standard Deviation	0.180	0.054	0.135	0.598576	0.253706	0.241256
Standard Deviation/ $n^{1/2}$	0.0570	0.0169	0.0427	0.189286	0.080229	0.076292
Unknown thickness		0.0470			0.0509	

From here the standard uncertainty in each item I_0, I_1, I_2 is given. **The standard uncertainty of t_1, from the guaranteed range of the standard sample being 10%, considering rectangular distribution, the value dividing 10% by $3^{1/2}$ of the displayed value is the standard uncertainty.** Because all items in Equation (1) are not independent, the following formula must be applied in order to find the uncertainty of t_0 found in equation (1).

$$u(y(p,q....)=[(\frac{\partial y}{\partial p})^2 \times \{U(p)\}^2 + (\frac{\partial y}{\partial q})^2 \times \{U(q)\}^2 + ... \ ]^{1/2}$$

The contribution of each variable in this equation is shown by the value squared of partial differentiation by the variable to the square of each standard deviation. Standard uncertainty can be calculated using a spreadsheet as in Tables 4 and 5 if all items are given. From the standard uncertainty obtained, if the expansion standard uncertainty is calculated as comprising of two coefficients, then, as shown below, there will be no significant difference in either party within the range of uncertainty.

Condition 1: ***0.047μm ± 0.0060μm***
Condition 2: ***0.051μm ± 0.0059μm***

Table 4 Uncertainty calculations under condition 1

		u(I0)	u(I1)	u(I2)	u(t1)
		0.042735374	0.016926	0.056966	0.006351
I0	3.1577	3.200435374	3.1577	3.1577	3.1577
I1	1.4233	1.4233	1.440226	1.4233	1.4233
I2	5.4853	5.4853	5.4853	5.542266	5.4853
t1	0.11	0.11	0.11	0.11	0.116351
t0	0.046968	0.048125281	0.046704	0.046318	0.04968
		0.001157285	-0.00026	-0.00065	0.002712
Standard Uncertainty	0.003031	1.33931E-06	6.96E-08	4.22E-07	7.35E-06

Table 5 Uncertainty calculations under condition 2

		u(I0)	u(I1)	u(I2)	u(t1)
		0.076291954	0.080229	0.189286	0.006351
I0	33.2315	33.30779195	33.23	33.23	33.23
I1	10.0462	10.0462	10.12643	10.0462	10.0462
I2	60.1465	60.1465	60.1465	60.33579	60.1465
t1	0.11	0.11	0.11	0.11	0.116351
t0	0.050906	0.05107305	0.050807	0.050711	0.053841
		0.000167506	-9.8E-05	-0.00019	0.002936
Standard Uncertainty	0.002948	2.80584E-08	9.62E-09	3.8E-08	8.62E-06