

Ultra-thick Au Coating Measurement II

1. Overview

SFT Application Brief No. 4 showed that employing a high resolution semiconductor detector in SEA5000 series instruments, allows you to measure a 5 μm Au coating over a Ni base by improving the calculation process, in addition to improving the signal to noise ratio, through application of the Fundamental Parameter (FP) method.

The results afterward of overlapping experiments showed considerable limitation in the measurable range of the base Ni coating by the calibration method even when temporarily using the SFT5000, and that the use of both the calibration method and FP method is essential.

2. Detector resolution error

Figures 1-1 and 1-2 show, respectively, the conventional SFT series detector (proportional counter) and SEA5120 detector (SSD detector). The measurement sample is permanent at base Ni, 2.55 μm , and the Au thickness surface layer is altered as shown. Measurement conditions are listed in Table 1.

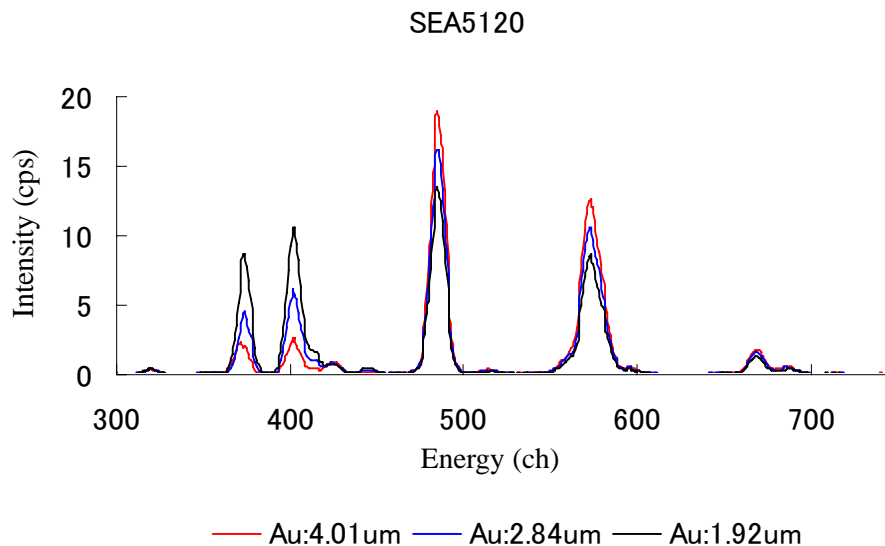


Figure 1-1 Spectrum with SEA5120

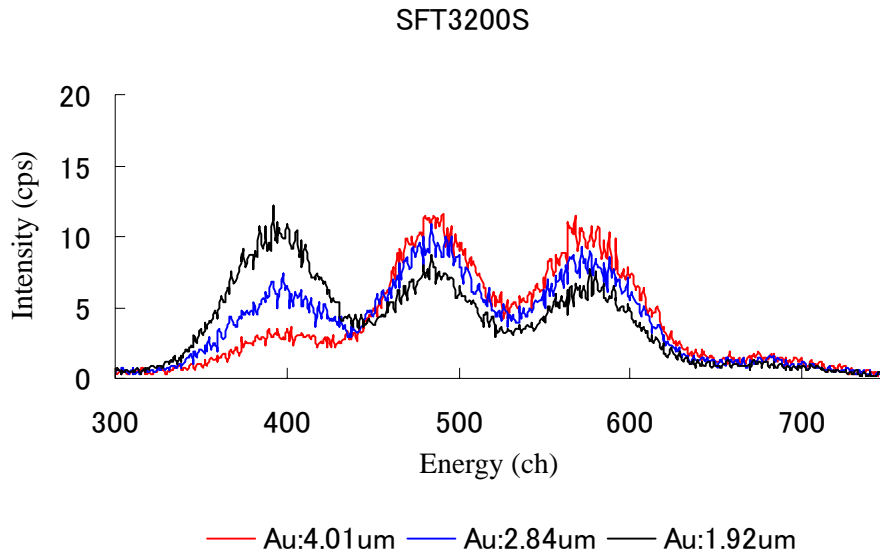


Figure 1-2 Spectrum with SFT3200S

Table 1 Measurement Conditions

	SEA5120	SFT3000
Collimator Size	0.1mm	0.1mm
Voltage	50kV	45kV
Current	1mA	1mA
Target	Mo	W
Measurement Time	300 sec	300 sec

The SEA5120 has the best resolution and as a result the signal to noise ratio is high. Therefore, even if the Au thickness changes, intensity change of middle layer Ni and the Cu base is read accurately. On the other hand, SFT3200S is unable to accurately read middle Ni and base Cu when the thickness of Au is 2 um or greater.

3. Calibration conditions

Shown below are calibration curve measurement conditions.

Table 2 Measurement Conditions

	Measurement Conditions
Measurement Time(sec)	60
Collimator	0.1mm
Excitation Voltage(kV)	50
Current(uA)	1000
Filter	none
Atmosphere	Air

Table 3 Calibration Data

No	Au	Ni	Cu	Au Intensity	Ni Intensity
1	infinite	----	----	215.049	0.906
2	----	infinite	----	0.333	777.429
3	----	----	infinite	0.234	3.077
4	1.92	----	infinite	136.664	1.678
5	4.16	----	infinite	189.041	0.941
6	1.92	infinite	----	132.358	127.713
7	4.16	infinite	----	195.547	25.791
8	----	2.01	infinite	0.341	213.924
9	----	10.20	infinite	0.272	563.232
10	2.84	5.22	infinite	165.583	38.206

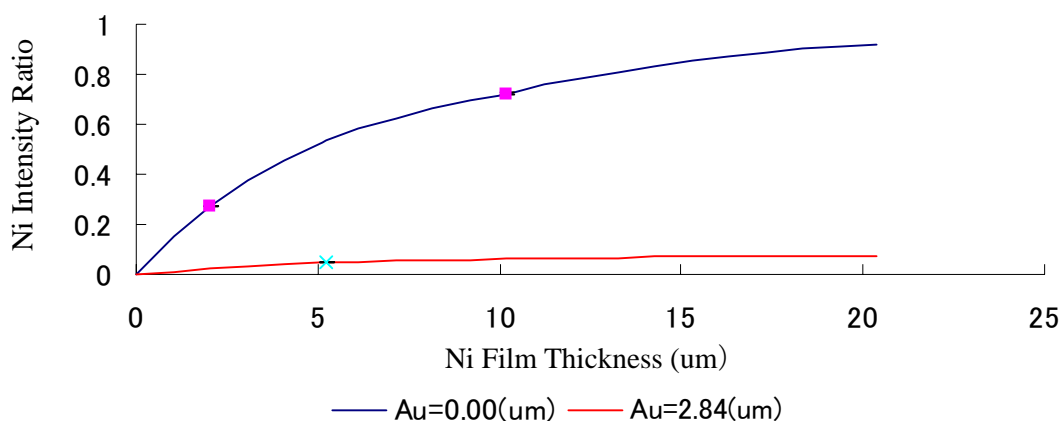


Figure 2 Au/Ni/Cu Calibration Graph

4. FP Method**Table 4 Measurement Results**

	Measurement Conditions
Measurement Time(sec)	60
Collimator	0.1mm
Excitation Voltage(kV)	50
Current(uA)	1000
Filter	none
Atmosphere	Air

Table 5 Quantitative Conditions

No	Name	Component	Thickness	Intensity
1	Au		2.84	
		Au	100.00(wt%)	161.526(cps)
	Ni		5.22	
		Ni	100.00(wt%)	37.344(cps)
	Cu		5000.00	
		Cu	100.00(wt%)	40.599(cps)

5. Comparison of Calibration Method and FP Method

Table 6

		Calibration Method						Thin Film FP Method					
	Standard	Ave	Range	SD	CV (%)	Accuracy (%)	Ave	Range	SD	CV (%)	Accuracy (%)		
1	Au	1.92	1.94	0.15	0.04	2.26	0.89	1.98	0.03	0.01	0.37	3.07	
	Ni	1.95	2.04	0.32	0.10	4.94	4.41	1.92	0.16	0.04	2.27	(1.49)	
2	Au	1.92	1.96	0.09	0.03	1.32	1.93	2.00	0.04	0.01	0.63	4.37	
	Ni	5.01	5.44	0.83	0.26	4.82	8.58	4.86	0.31	0.11	2.17	(2.89)	
3	Au	1.92	1.98	0.08	0.03	1.47	3.28	2.01	0.03	0.01	0.59	4.48	
	Ni	10.20	13.04	2.89	1.03	7.91	27.83	9.61	0.59	0.20	2.06	(5.75)	
4	Au	2.84	2.89	0.25	0.07	2.34	1.94	2.85	0.08	0.02	0.73	0.32	
	Ni	1.95	1.94	0.48	0.12	6.37	(0.41)	1.82	0.18	0.06	3.28	(6.62)	
5	Au	2.84	2.93	0.19	0.06	1.89	3.24	2.82	0.05	0.02	0.62	(0.63)	
	Ni	5.01	5.98	1.60	0.55	9.26	19.36	4.97	0.35	0.10	2.05	(0.78)	
6	Au	2.84	2.28	2.94	1.20	52.74	(19.79)	2.79	0.04	0.01	0.42	(1.94)	
	Ni	10.20	10.99	16.90	6.02	54.81	7.75	9.74	0.51	0.17	1.73	(4.54)	
7	Au	4.01	4.85	0.66	0.20	4.15	20.85	3.97	0.10	0.03	0.73	(0.87)	
	Ni	1.95	2.89	2.07	0.63	21.64	48.15	1.79	0.28	0.08	4.61	(7.95)	
8	Au	4.01	4.65	0.29	0.08	1.77	15.91	4.01	0.13	0.04	0.92	(0.12)	
	Ni	5.01	8.49	7.57	2.27	26.70	69.40	5.01	0.41	0.13	2.65	0.06	

6. Conclusion

Measurement results of the FP Method, as compared to the calibration method, characteristically display better accuracy and less dispersion. Large error is produced in the results of measuring middle layer Ni when Au is 3 μm or greater, and as a result, the effects are reflected in the Au readings. With the FP method, dispersion of both the Au thickness and middle Ni thickness is less than 5%, and measurement within accuracy of 8% is possible.

The intensity information of the Cu base being reflected in the film thickness calculation is the main factor in the FP method. The calibration method uses only Au and Ni information in thin film calculation. The FP method offsets and reduces error by using Cu intensity information. In other words, stable readings can be obtained by taking a balance with the thickness calculated from the film information and the thickness calculated from the base information.

This effect is valid even if the entire detection intensity changes because of, for example, sample slant or shifting in the focal position.

In conclusion, the following two items are the factors that allow measurement of ultra-thin Au plating on middle Ni.

- (1) Intensities of middle Ni as well as Cu can be accurately detected by using a high resolution semiconductor detector.
- (2) Improved accuracy and reduced dispersion can be realized using the FP method by reflecting base Cu information in film thickness calculations.