

XRF

Non-destructive Thickness and Composition Analysis of NiP/Au Plating on Cu Contacts using Micro-XRF





Application Note

Electronics XGT24

## Chiya Nishimura<sup>1</sup>, Lian Hock Chuan<sup>2,</sup> Tomoki Aoyama<sup>1</sup>

<sup>1</sup>HORIBA, Ltd., Japan, <sup>2</sup>HORIBA Instruments (Singapore) Pte Ltd., Singapore

**Abstract**: We carried out plating thickness and composition analysis on dual-layer NiP/Au plating on Cu contacts using a HORIBA X-ray analytical microscope. We successfully detected Au of ultra-thin layer without any sample preparation. The thickness results of Au and NiP plating were consistent with the provided values and with good repeatability.

Keywords: Electronics, flexible printed circuits, Cu/NiP/Au, plating thickness and composition analysis, micro-XRF

#### Introduction

Dual-layer NiP/Au plating is a popular plating for Cu contacts on flexible printed circuits. The plating thickness is important to control the electrical conductivity, corrosion resistance, and mechanical behavior<sup>[1]</sup>.

Scanning electron microscopy observation is a popular approach for plating thickness measurement. However, the thickness of the Au top plating layer is normally tens of nanometers. Such an ultra-thin layer requires not only high-resolution scanning electron microscopy, but also skillful techniques of cross-section preparation. On the other hand, an X-ray fluorescent spectrometer provides an easy, non-destructive way to calculate plating thickness, as well as its composition. It can get plating thickness and composition values without the need for cross-section and time-consuming sample preparation. Among XRFs, micro-XRF is suitable for plating on small areas of electronics like Cu contacts.

In this application note, we introduce non-destructive thickness and composition analysis of dual-layer NiP/Au plating on Cu contacts on a flexible printed circuit using the HORIBA XGT-9000 X-ray analytical microscope.



Figure 1. A flexible printed circuit with Cu contacts.

## XGT-9000 X-ray Analytical Microscope

The XGT-9000 X-ray analytical microscope (Figure 2(a)) is an energy-dispersive X-ray fluorescence microscope with micro-probes down to 10  $\mu$ m, high-resolution CCD cameras, and a motorized XYZ stage. These features enable users to focus a target area even on small patterns. In addition, the XGT-9000 software can offer an optional feature of plating thickness and composition calculation based on the fundamental parameter method (FPM). It allows the calculation with/without standard samples.

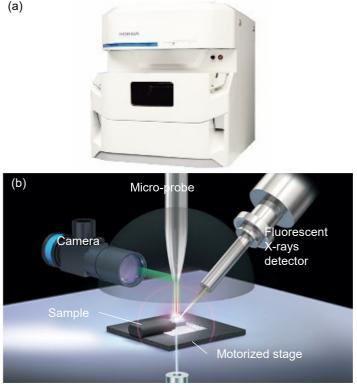


Figure 2. (a) HORIBA XGT-9000 X-ray analytical microscope (b) the optics diagram inside the instrument.

HORIBA



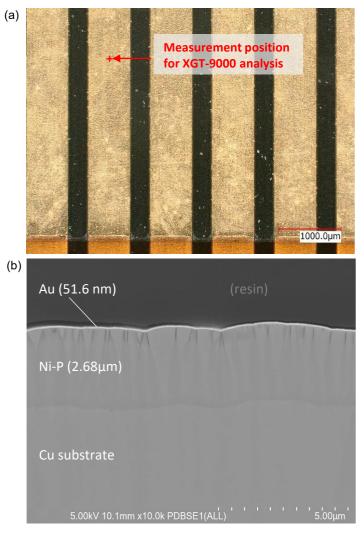


Figure 3 (a) Dual-layer NiP/Au plating on Cu contacts analyzed in this application note. (b) The plating thickness values are known as 51.6 nm of Au top layer and 2.68  $\mu$ m of NiP layer.

### Sample information

To evaluate the accuracy of standard-less calculation by the XGT-9000's Multilayer FPM module, we prepared dual-layer NiP/Au plating on Cu contacts on a flexible printed circuit whose plating thickness values are known as 51.6 nm of the Au top layer and 2.68  $\mu$ m of the NiP middle layer (Figure. 3).

#### Measurement

We first conducted spectrum analysis on the surface of a contact using the XGT-9000. The measurement position is shown in Figure 3(a). The XRF spectrum was obtained using a 100  $\mu$ m ultra-high intensity probe under whole vacuum condition. The voltage and current of the X-ray generator were set to 50 kV and 100  $\mu$ A, respectively. The measurement time was set to 180 seconds to get a better

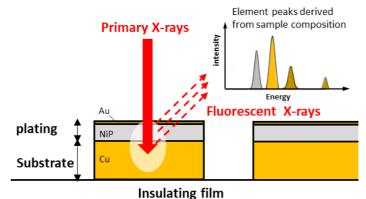


Figure 4: Schematic diagram of thickness calculation using XRF on a multilayer structure.

signal-to-noise ratio of Au peaks from the ultra-thin Au top layer. It was repeated three times on the position to check the repeatability. As shown in a schematic diagram in Figure 4, XRF spectrum contains various characteristic peaks of elements in a sample. While the detected peaks tell us the composition of the sample, the peak intensities of the elements can be correlated with its concentration. In a case where a sample has a multilayer structure, an XRF spectrum can also be used to calculate the thickness of the layers using the peak intensities of the elements of the plating compositions.

#### Result

Figure 5 shows XRF spectrum obtained from the NiP/Au plating on the Cu contact. It presents peaks of P, Ni, Cu and Au, which are all the components of the sample. It means that the primary X-rays penetrated through the plating and reached the substrate, and that fluorescent X-rays generated from the layers reached a detector successfully.

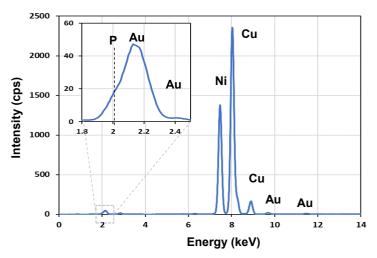


Figure 5. XRF spectrum on the NiP/Au plating on the Cu contact on a flexible printed circuit. The measurement position is shown in Figure 3(a).

HORIBA



Next, we calculated the plating thickness and composition using the Multilayer FPM module of the XGT-9000 software. The Multilayer FPM module calculated using an obtained spectrum and a user-defined layer model. We defined the layer model as shown in Figure 6. Thanks to the calculation based on FPM, we can calculate both thickness and composition, even without standard samples. Moreover, this calculation includes peak separation, therefore we can get accurate results even if spectral interference occurs like P and Au in this sample.

Table 1 shows the results (n=3) of the calculated plating thickness and composition values. It shows 49 nm of of the Au top layer and 2.54 µm of the Ni middle layer, on average. The results were consistent with the provided values. The calculated composition result showed that P content was 5.8 % in the NiP middle layer, on average. These values were consistent with the P content range reported in previous research<sup>[2]</sup>. Thus, thanks to the Multilayer FPM module, we could obtain consistent results of plating thickness and composition with good repeatability without any standard samples.

		<ul> <li>Layer Count</li> </ul>		~ Analysis	method FPM	~		xport setting
Known	Concentration [	Known Thickne	iss 🔲 Known De	ensity				inport betailig:
	Composition 1		Composition 2		Composition 3		Composition 4	
Layer Bulk	Name	Line	Name	Line	Name	Line	Name	Line
Layer 1	Au ×	Auto ~		Auto ~		Auto ~		Kα
Layer 2	Ni ×	Auto 🗸	P X	Auto 🗸		Κα ~		Kα
Layer 3 🗹	Cu ×	Auto ~		Auto ~		Auto ~		Auto
1								
				Update Preview				

Figure 6. Layer model used for this sample. Standard-less calculation using Multilayer FPM module was applied.

Table 1: Plating thickness and composition results of NiP/Au layers (n=3) calculated by the XGT-9000.

	F	Result ( <i>n</i> =3)				
Thickness (µm)	1	2	3	- Average		
Au	0.049	0.050	0.049	0.049		
NiP	2.55	2.54	2.54	2.54		
Composition (mass%)						
Au content	100	100	100	100.0		
Ni content	94.1	94.4	94.1	94.2		
P content	5.91	5.62	5.87	5.80		

#### Conclusion

XGT-9000 provides a fast and non-destructive analysis of thickness and composition of dual-layer NiP/Au plating on a Cu contact. We successfully detected Au of the ultra-thin layer without any time-consuming sample preparation. Our plating thickness results of Au and NiP were consistent with the provided values, and with good repeatability despite standard-less condition. Thus, the XGT-9000 is a powerful system for analyzing these types of plating.

#### Reference

[1] A. Bahramian et al (**2019**) Cu/Ni/Au multilayers by electrochemistry: A crucial system in electronics -A critical review, *Microelectron Eng.*, 206(1), pp.25-44.

[2] A. M. Pillai et al. (**2012**) Electrodeposited nickel– phosphorous (Ni–P) alloy coating: an in-depth study of its preparation, properties, and structural transitions. *J. Coat. Technol. Res.*, 9, pp785–797.



# info.sci@horiba.com

USA: +1 732 494 8660 UK: +44 (0)1604 542 500 China: +86 (0)21 6289 6060 Taiwan: +886 3 5600606 
 France:
 +33
 (0)1
 69
 74
 72
 00

 Italy:
 +39
 06
 51
 59
 22
 1

 India:
 +91
 (80)
 4127
 3637

 Brazil:
 +55
 (0)11
 2923
 5400

www.horiba.com/scientific Germany: +49 (0) 6251 8475 0

Germany:	+49 (0) 6251 8475 0
Japan:	+81(75)313-8121
Singapore:	+65 (6) 745-8300
Other:	+33 (0)1 69 74 72 00

