

SEAno.37 Measuring trace amounts of lead in electroless nickel coating using fluorescent X-ray analysis and the thin-film FP method

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1. Introduction

The RoHS / ELV Directive mandates the management of the lead concentration of electroless nickel plating. In this application brief, a fluorescent X-ray analysis unit (SEA1200VX) is used to measure trace amounts of lead in electroless nickel plating.

In application brief No.28, electroless nickel plating was measured. In this brief, a sample with a known concentration was used so that the accuracy could be verified as well.

2. Experiment

The SEA1200VX was used to measure the lead in electroless nickel plating on copper. This test uses thin-film FP method to calculate the plating thickness and lead concentration. As a prerequisite for the thin-film FP method, the sample area must be larger than the X-ray irradiation radius. Table 1 shows the measurement conditions. To calculate the concentration of P, the excitation voltage of condition 2 (15 kV) was used.

Table 1 – Conditions for measuring lead in electroless nickel coating using the thin-film FP method

Measurement Instrument	SEA1200VX	
	Condition 1	Condition 2
Measurement time (seconds)	100	60
Collimator (mm)	φ8.0	
Tube voltage (kV)	50	15
Tube current (μA)	1000	1000
Filter	For Pb	For OFF
Lead analysis curve (ROI)	L α	
Atmosphere	Vacuum	

3. Results

A sample with 3 μm of electroless nickel coating containing approximately 360 ppm of lead was measured 10 times in the same place. Table 2 shows the results and Figure 1 shows the spectrum. Table 2 also includes the ICP emission spectrometry (ICP-OES) results for a sample with the same plating.

The ten measurements of lead concentration in 3 μm of electroless nickel plating had an average of 347 ppm with a standard deviation of 26.8 ppm and a CV value of 7.7 percent (see Table 2).

Furthermore, the spectrum in Figure 1 clearly shows a lead peak and confirms the trace amount of lead in the film.

Table 2 – Results for lead in electroless nickel plating using thin-film FP method

	Ni-P Plating thickness	Pb concentration	ICP-OES (Pb concentration)
Avg.	3.04 μm	347 ppm	364 ppm
Standard deviation	0.059 μm	26.7 ppm	-----
CV value (%)	1.94	7.72	-----

n=10

4. Conclusion

These results show that lead can be detected in electroless nickel plating with a measurement time as short as 100 seconds. The repeatability had a CV value of approximately 8%. This application should be performed in a vacuum because it is necessary to measure phosphorus in the electroless nickel plating. If measurements cannot be performed in vacuum (vacuum equipment is not available), the phosphorus

concentration can be fixed before calculations to derive the film thickness while managing the lead concentration.

Furthermore, to improve the measurement accuracy, it is recommended to acquire an electroless nickel plating sample with a known lead concentration, register it under the measurement conditions in this brief, and then measure the sample.

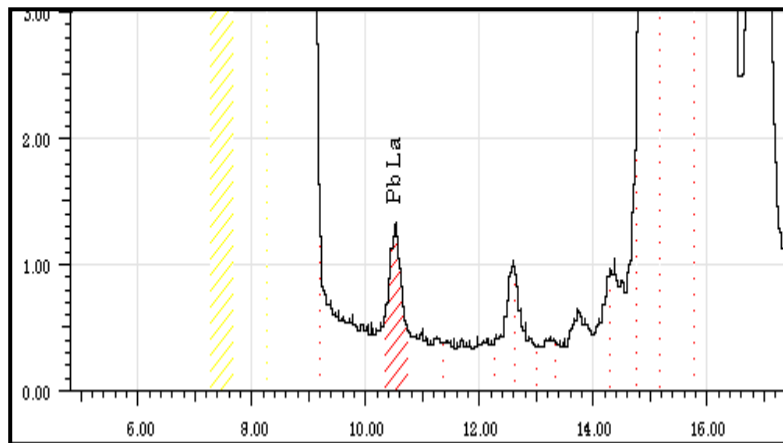


Figure 1 – Spectrum for 364 ppm of lead in 3 μ m of electroless nickel plating on copper