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Application Brief

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Measuring trace amounts of lead in lead-free solder coating using fluorescent X-ray analysis and the thin-film FP method

1. Introduction

Controlling trace amounts of lead concentration in lead-free solder plating is crucial to comply with RoHS/ELV directives.

In this application brief, the SEA1200VX, an X-ray fluorescence analysis unit, is used to measure trace amounts of lead in lead-free solder plating. (For examples using the SEA1000A, SEA2210A and SEA5120A, see Application Brief No.29)

2. Testing

The SEA1200VX was used to measure trace amounts of lead in the following items:

- Lead-free solder plating on copper leads.
- Tin plating on nickel-plated copper leads.

The thin-film FP method was used to acquire the plating thickness and lead concentration.

When using the thin-film FP method, the sample area must be larger than the X-ray irradiation radius. The size of the individual lead samples was insufficient for measurement so they were bundled together to create a sufficiently large and flat measurement surface. Table 1 shows the measurement conditions.

Table 1 – Conditions for measuring lead in lead-free solder plating using the thin-film FP method

Measurement Instrument	SEA1200VX	
	Condition 1	Condition 2
Measurement time (seconds)	300	60
Collimator (mm)	φ 8.0	
Excitation voltage (kv)	40	50
Tube current (μA)	1000	1000
Filter	For Pb	For Cd
Lead analysis ray (ROI)	L β	
Sample environment	Air	

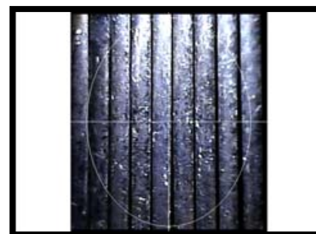

Figure 1 – Sample configuration when measuring lead terminals

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3. Results

Measuring lead in the lead-free solder plating on copper (Pb in Sn-Ag-Cu solder)

A sample with 5 μm of lead-free solder plating containing approximately 300 ppm of lead was measured 10 times at the same location. Table 2 shows the results and Figure 2 shows the spectrum. Table 2 also includes the ICP emission spectrometry (ICP-OES) results for the same sample. The average of the 10 lead concentration measurements was 349 ppm, with a standard deviation of 8.9 ppm and a CV value of 2.6 percent (see Table 2).

The lead peak in spectral image in Figure 2 clearly shows the trace amounts of lead in the solder plating.

Measuring lead in the lead-free solder plating on the nickel-plated copper

In this test, the top layer of two layers was lead-free solder. The sample was composed of semiconductor leads (See Figure 1). Table 3 shows the measurements results for lead in the 10 μm of lead-free solder. Figure 3 shows the spectral image. Table 3 also includes ICP-OES results for the same sample.

The results show that this method is sufficient for detecting lead, as even 200 ppm of lead can be detected at thickness of 10 μm (see Figure 3). It also sufficient for determining the quantity of lead, as it has a very high correlation with ICP-OES (see Table 3).

Table 2 – Thin-film FP method results for lead in lead-free solder

	Sn-Ag Plating thickness	Pb con.	ICP-OES (Pb concentration)
Avg.	5.49 μm	349 ppm	313 ppm
Standard deviation	0.015 μm	8.9 ppm	---------
CV value (%)	0.27%	2.57%	---------

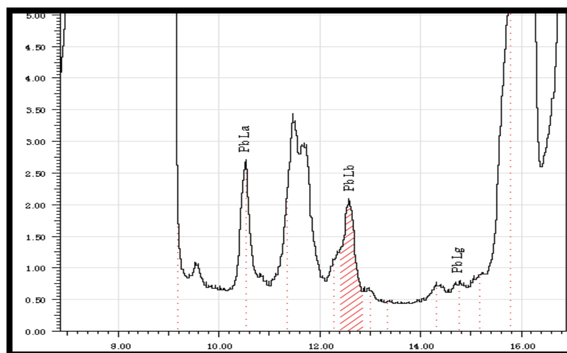

Figure 2 – Spectrum for 300 ppm of lead in 5 μm of lead-free solder on copper plating

Table 3 – Thin-film FP method results for lead in lead-free solder

	Sn-Ag Plating thickness	Pb concentration	ICP-OES (Pb concentration)
Results	10.5 μm	178 ppm	182 ppm

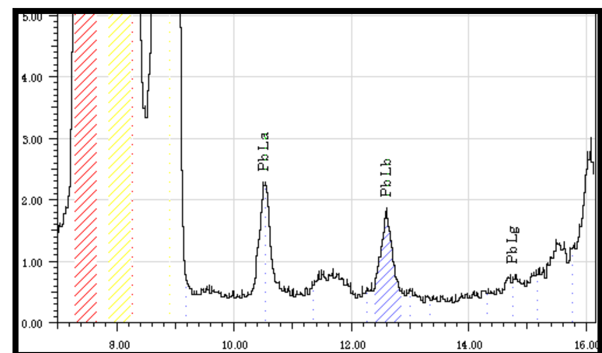

Figure 3 – Spectrum for 200 ppm of lead in 10 μm of lead-free solder

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4. Cautions when using this thin-film FP method

- The sample surface must be larger than the X-ray radiation radius
- The structure and elements of the material and plating must be known
- The element to be measured (in this case, lead) can not be present in more than one layer
- The measurement location must be flat and free of large irregularities

If the samples are small, they must be gathered together to create a flat surface. The quantitative accuracy can be improved by making a flatter surface. However, if the condition is different, the quantitative accuracy decreases.

If the sample has more than one layer containing lead, it is difficult to measure the quantity in a single layer because it is not possible to determine which layer the detected lead came from. One example of this is lead-free solder plating on electroless nickel plating.

5. Conclusion

The SEA1200VX detected the trace amounts of lead in lead-free solder plating, as shown in the spectral images in Figure 2 and Figure 3. Furthermore, quantitative results produced by the SEA1200VX and thin-film FP method had a high correlation with precision analysis ICP-OES, even with layered samples such as plated parts. These results show that the SEA1200VX is a useful tool for managing the lead concentration in lead-free solder plating.

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