Analytical Techniques for Problem Solving



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The Identification of Particulate Surface Contaminants Using SEM/EDS

Connector contacts typically rely on clean, debris-free surfaces to maintain electrical integrity and low contact resistance. Immediately after plating and packaging, these conditions are easily achieved. However, through subsequent handling operations, such as post-forming, insertion and molding operations, an increased likelihood of contamination occurs. This contamination may take the form of thin transparent layers, as is the case with grease, oils and other thin films. It may also be in the form of particulate matter from machining, grinding, forming, and insertion operations or packaging materials.

The detection and analysis of these minute particles require specialized instruments and the appropriate personnel trained in their operation. Optical microscopes can be used to view these surfaces directly, and to record images either on film or in digital format. They are limited, however, in their magnification and focal range. Scanning electron microscopes (SEMs) have much higher limits on the available magnification and produce more detailed images as a result of both increased resolution and depth of field. Many SEMs also have the added capability of being able to analyze the X-ray signals generated within the sample, while being bombarded by the electron beam. This capability, known as energy dispersive spectroscopy (EDS), allows for identification and quantification of various elements present on the surface and within the sample. It can be performed in spot mode, where the beam is held in a single position, or in raster mode, where a given element's concentration is plotted against the beam's position to produce an element or "dot" map.

Problem

After being assembled into final devices, a component manufacturer noticed "black spots" on the interior surface of socket connector terminals. Samples were submitted to identify the composition of the spots, and to identify a possible source of the unknown material.



Fig. 1—Optical photo of suspect connector component showing small black areas of particulate contamination.

Analysis

The samples were carefully split apart to expose the contaminated surface and examined using optical microscopy, SEM and EDS.

Optical microscopy (Fig. 1) gives an overview of a suspect contact showing the contaminated surface at 30X. The "black" contaminant is clearly evident in the functional area of the gold-plated surface.

SEM examination confirms that the sample consists of a smooth gold surface, typical of electroplating. However, it is covered with suspect mate-



Fig. 2—Scanning electron microscope (SEM) images of contact area at 30, 150 and 800 X.

rial that is not an integral part of the plating process or corrosion product, but appears to have been deposited on the surface well after the plating operation.

Spot-mode EDS analysis of the material seen in Fig. 2 shows it to be chiefly composed of Fe and Mn, with smaller amounts of O and Zn (Fig. 3).

The plate-like structure of the contaminant, seen at higher magnification in Fig. 4, sug

higher magnification in Fig. 4, suggests it may be a mineral-type deposit.

Last, an X-ray "dot" map was performed at 120X. This map shows the distribution of Fe throughout the contact surface. Mn is visible to a lesser amount, but in the same spatial distribution as Fe, whereas Zn and O are in lower quantities and not easily discerned in this manner.

Solution/Conclusion

This information led the manufacturer to an assembly operation where Mncontaining ferrite cores were installed near the contacts in the completed device. Residual ferrite dust was removed from the insertion tool on a more frequent basis, thereby eliminating further contamination. *Pass*F





Fig. 4-SEM micrograph (1500X) reveals plate-

like structure of individual particle resembling

Fig. 3—Energy dispersive spectroscopy (EDS) spectra of particulate material found on contact surface.



Fig. 5—EDS "dot" maps showing the distribution of Fe (left) and Mn (right) on the sample surface.