技術情報 Technical Information

XPS for Environmental Studies
Analysis of Hexavalent Chromium in Compliant to WEEE/RoHS

1. General

Chromating of electrogalvanized items is widely used to provide corrosion resistant surfaces for steel and automotive materials.

As part of the regulations for hazardous material disposal, WEEE/RoHS directives require all chromated materials be tested for hexavalent chromium and trivalent chromium.

Since there is no equipment capable of analyzing hexavalent chromium, testing is usually done by removing the chromated layer to be analyzed in emission spectrography such as ICP.

XPS is attracting attention as a tool effective in identifying the valence number of chromium, because it acquires information from areas a few nm deep from the surface, and is capable of high sensitivity analysis of the oxidation state (valence number) of thin materials.

2. XPS Analysis of chromated screws

Figure 1 is an optical micrograph of a chromated screw used for analysis. A chromium coating of a few nm thick was developed on Ni plated on the screw. Two samples, A and B, were analyzed. The sample A contained very little hexavalent chromium, while the sample B a great deal of hexavalent chromium. The instrument used was a photoelectron spectrometer (JEOL JPS-9010MC). The area analyzed was 1 mm in diameter, and each sample was analyzed for 20 minutes.

Figure 1. Optical micrograph of a chromated screw

Figure 2 shows Cr2p photoelectron spectra acquired in XPS. The chemical shift of the Cr2p3/2 in the spectra was used to determine the valence number of Cr. Table 1 shows the chemical shift of the bond energy of Cr2p3/2. The table shows that the bond energies of Cr2p3/2 of trivalent chromium and hexavalent chromium have peaks at 576.0 eV and 578.9 eV respectively. The energy difference between trivalent chromium and hexavalent chromium oxides was 2.9 eV, demonstrating XPS's effectiveness in detecting peaks and determining the valence numbers.

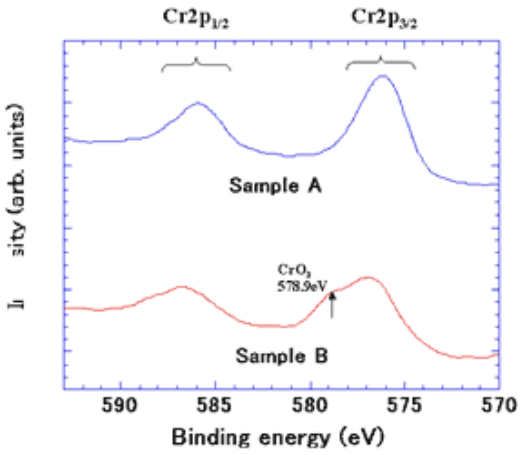

Fig.2 Cr2p photoelectron spectra of chromated screws

	$Cr2p_{3/2}$ bond energy (BE)
Cr	574.3eV
Cr_2O_3	576.0eV
CrO_3	578.9eV

Table1. Chemical shifts of chromium compounds

The Cr2p3/2 peak of the sample A was detected at 576.0 eV, corresponding to the trivalent chromium used for surface treatment. The Cr2p3/2 peak of the sample B was detected as an overlap due to the chemical shift. When this peak was separated by the waveform separation program, a peak was detected at 578.9 eV, corresponding to the hexavalent chromium used in surface treatment. XPS has thus proven effective in detecting hexavalent chromium in the chromated surface of the screw.

3. Summary

XPS was used for analysis of the chromated film on two screws, successfully determining valence numbers of 3 and 6. XPS, a high sensitivity surface analyzer, is effective in determining valence numbers of samples coated with concentrated chromium or with extremely thin chromate film on the surface.

References

1. JEOL JPS-9010MC brochure
2. JEOL web/Technical Information (Chemical Bond Analysis in XPS)
3. JEOL News Vol. 36, (2004), page 38 – Members only publication

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