CCD Point Analysis Example of a Foreign Substance in a Coating Film

Outline

A foreign substance in a linear form was found in a coating material applied to coating paper. In order to learn it's composition without destroying the sample, a CCD point analysis was conducted. This CCD point analysis is designed to carry out qualitative or quantitative analysis by specifying the specific measuring positions on the sample surface by the joint use of a CCD camera, self-contained in the analytical system, and a sample stage driving mechanism. This is a new function added to the Rigaku ZSX X-ray spectrometer system. In addition, mapping analysis was made around the detected components by use of the sample stage driving mechanism. The outcome of this analysis is also introduced here.

1. Sample preparation and equipment

1.1 Equipment

Rigaku / Sequential XRF model **ZSX100e**

X-ray tube	: 4kW, End window, Rh target
Sample observation system	: CCD camera
Analytical point positioning	: By sample stage
Analysis area	: 1 mm dia.

1.2 Sample preparation

The sample was installed in a dedicated sample holder and was measured as is.

1.3 Measurement condition

Element	F~Mg	Al, Si	P, S	Cl	K, Ca	Ti~U
kV – mA	30-120	30-120	30-120	30-120	40-90	50-72
Slit	Standard	Standard	Standard	High resol.	Standard	Standard
Crystal	TAP	PET	Ge	Ge	LiF200	LiF200
Detector	F-PC	F-PC	F-PC	F-PC	F-PC	SC
PHA	Diff.	Diff.	Diff.	Diff.	Diff.	Diff.
X-ray path	Vac.	Vac.	Vac.	Vac.	Vac.	Vac.

2. Measurement result

A picture of a portion of the sample surface, photographed with the built-in CCD camera is shown at right. One can confirm that a linear foreign substance is mixed into the coating material.

Qualitative analysis was first conducted over 1mm dia. areas for analysis by designating the linear foreign substance area, the blank coating film area and the coating paper area, respectively. As a result, a large difference was observed in terms of CuKa. Further, Ti and Fe were verified as the coating film components and Al, Si and Ca as the coating paper components.
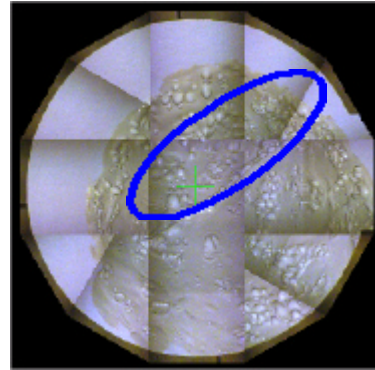
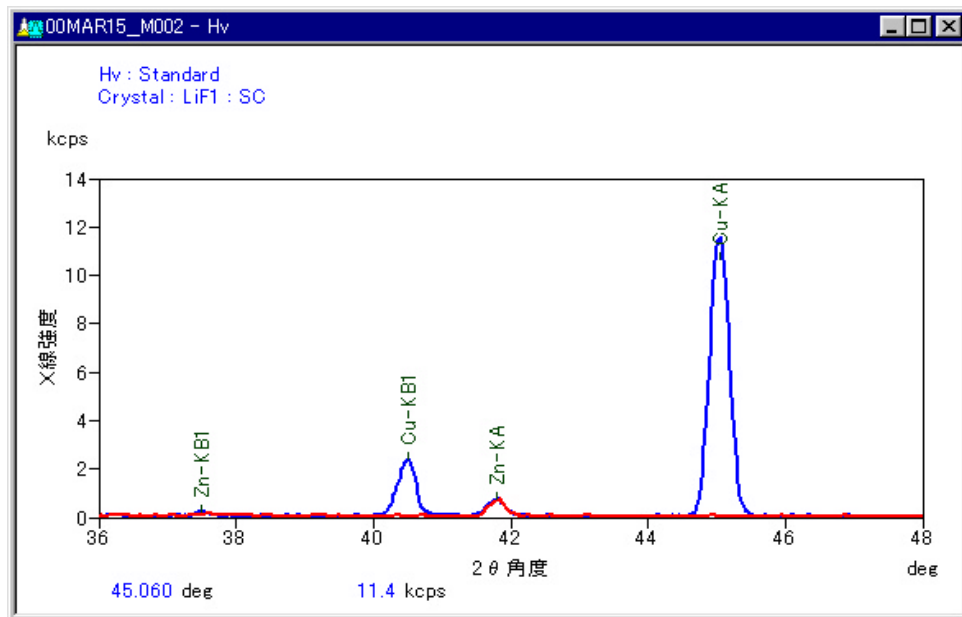

Fig.-1 Composite photo of CCD

Chart 1 Qualitative analysis chart of the foreign substance (indicated in blue) and the coating film (in red)

Mapping analysis was carried out based on the component information obtained from the qualitative analysis. In the case of this mapping analysis, too, the sample itself is moved to a point for analysis by means of the sample stage driving mechanism. This assures stable measurement because the optical section related to the angular resolution remains still. This mapping analysis shows that a piece of copper wire had been mixed into coating material.
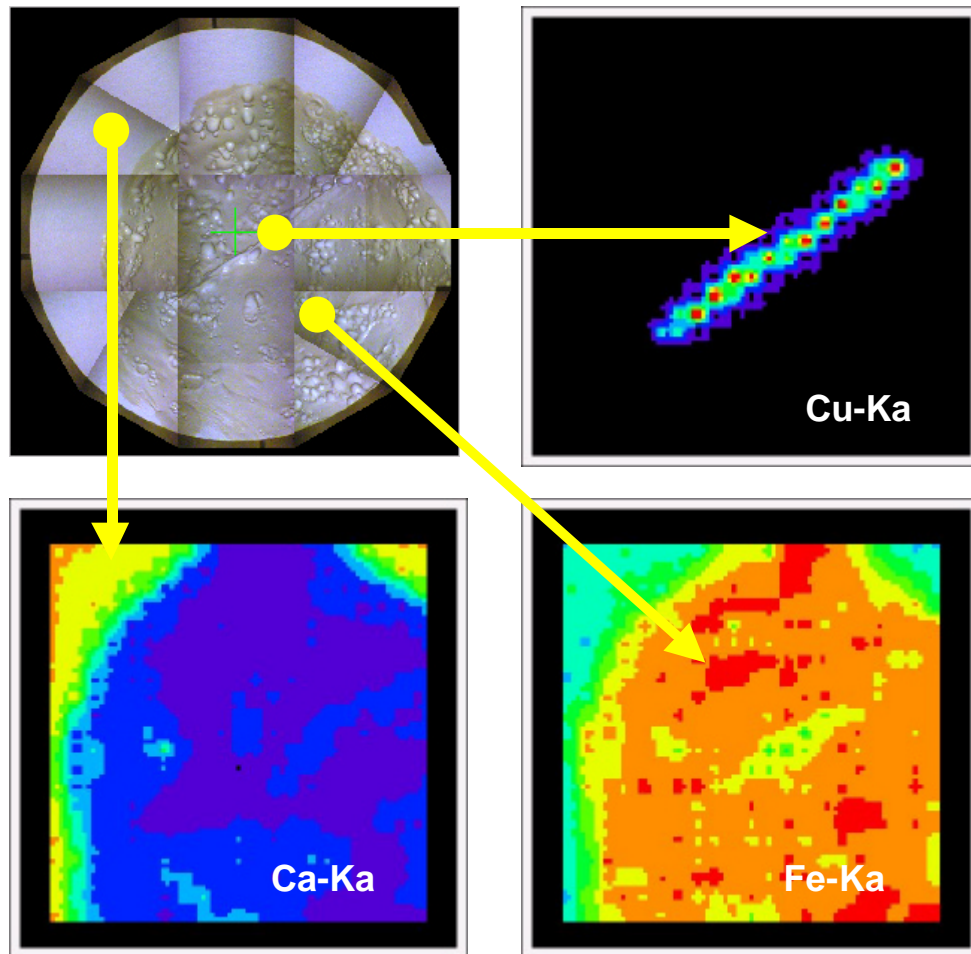

Fig.-2 Mapping contour line of typical components

3. Summary

Unlike the analysis technique using electrons (SEM/EDX), the spectra obtained by fluorescent X-ray spectrometry are free from the charge-up effect. Thus, no specific sample treatment is required. Moreover, since in-depth information is obtainable with fluorescent X-rays as compared with electrons, details of the underside state of the thick coating film were successfully obtained.