High-sensitivity micro-spot elemental analysis using a WDXRF spectrometer equipped with a polycapillary lens

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1. Introduction

There is growing demand for high precision and sensitivity micro-spot elemental analysis of microelectronic components. EPMA (electron probe micro analyzer) and EDXRF (energy-dispersive X-ray fluorescence) are commonly used for this purpose. However, two of the drawbacks for EPMA and EDXRF are their relatively low sensitivities, and spectral resolutions.

A wavelength dispersive X-ray fluorescence (WDXRF) spectrometer equipped with a high-energy-type polycapillary lens has recently been developed by Rigaku (Fig. 1)(1),(2). This WDXRF spectrometer can be used for a 100-µm spot-size measurement and has high precision as well as sensitivity even for light elements. In this article, the newly developed Rigaku WDXRF spectrometer equipped with a polycapillary lens is briefly described and examples on its applications to micro-spot analysis are given.

2. WDXRF spectrometer equipped with a polycapillary lens

As shown in Fig. 2, The main features of the newly-developed Rigaku WDXRF spectrometer include a 4-kW rhodium-target end-window tube for high intensities, a r-θ sample stage together with a high-resolution camera for observing and positioning a sample for an 100-μm spot-size XRF measurement, and a polycapirally lens for high precision and resolution measurements of X-rays with high energies up to 35keV, Similar to a common WDXRF spectrometer, the new Rigaku spectrometer also has a primary-beam-filter changer, a ten-crystal changer, two detectors (SC and F-PC) and different-size diaphragms (e. g., 30 mm or 20 mm in diameter). Therefore this spectrometer can be used as a general purpose WDXRF spectrometer. Since the analysis software used in this new spectrometer is the same as that used in the original ZSX spectrometer system, accurate and precise quantitative analysis of thin films using the standardless fundamental parameter method can also be preformed.

Fig. 1. Rigaku WDXRF spectrometer system equipped with a polycapillary lens for micro-spot elemental analysis.

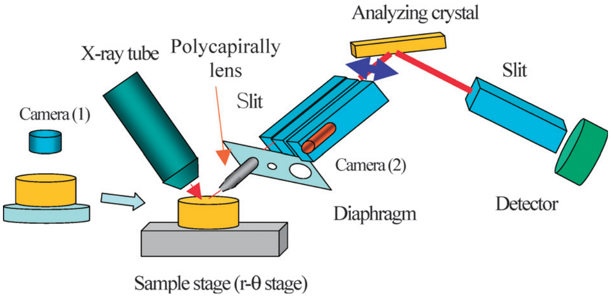

Fig. 2. Schematic diagram of the X-ray optics for the newly developed Rigaku WDXRF spectrometer.

3. High-energy-type polycapillary lens

A polycapillary lens is made of bundles of capillary tubes with inner diameters as small as a few microns. When X-rays entering the polycapillary lens and striking the interior of the tubes at grazing angles, they can be guided along the tubes by multiple total reflection, and these totally reflected X-rays exit the other end of the capillary lens either as a focused or paralleled X-ray beam.

There are two types of polycapillary lens, the low-energy-type polycapillary lens mounted on the former mode of a WDXRF system is used for X-rays with energies up to 15 keV (Sr-Kα). While, the newly developed WDXRF system equipped with the new high-energy-type polycapillary lens can be used for X-rays with high energies up to 35 keV.

For a comparison, a stainless steel sample was measured by of these two types of polycapillary lens, and their XRF spectra are shown in Fig. 3. In the region with X-ray energies higher than that of Mo-Kα (2θ angles below 15° in Fig. 3), the background intensities from the low-energy-type policapillary lens are very much higher than those from the new high-energy-type policapillary lens because no total reflection occurred for most of the high-energy X-rays inside the low-energy-type policapillary lens.

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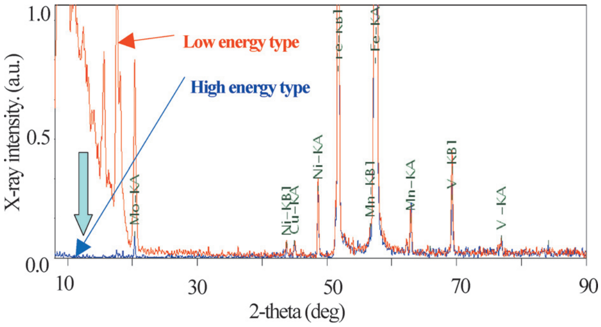


Fig. 3. Comparison between the high-energy-type and the low-energy-type polycapillary lenses.

4. Spatial resolution

A sample of copper in one half and tin in the other half was used to study the spatial resolution of the high-energy- type polycapillary lens. The XRF intensities of Cu-Kα; and Sn-Kα; emitted from the sample were measured as the sample stage was moved by 0.1 mm step across the boundary between the copper and the tin (Fig. 4: knife-edge scan). The results of this measurement show that the resolution of this lens is 50–80 μm.

5. Applications

5.1. Analysis of a gold-coated terminal

A gold-coated terminal from a cellular phone was analyzed. An electronic camera was used to direct and observed the analysis position on the gold-coated terminal (Fig. 5). A qualitative analysis was carried out with a high-energy-type polycapillary lens (spot size: 100 μm in diameter) and a LiF (200) analyzing crystal. As shown in Fig. 6, XRF peaks for Ni, Cu and Au were detected. The structure of the gold-coated terminal is Au (top layer)/Ni (middle layer)/Cu (substrate). A thickness analysis for the Au and Ni layers was performed using the fundamental parameter method for thin films. The thicknesses of the Au and Ni layers were determined to be 73 nm and 5.9 μm, respectively. The Au-Lα;, Ni-Kα; and Cu-Kα; X-rays mapping images are shown in Fig. 7. The results show that 0.1-mm lines can be clearly observed.

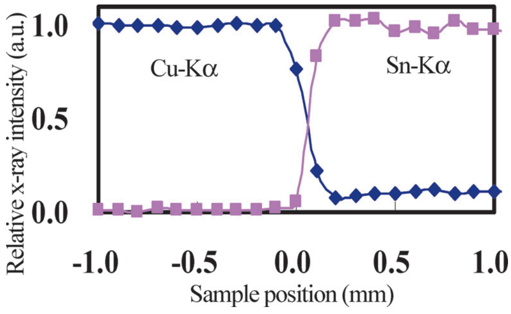


Fig. 4. Spatial-resolution test results (knife-edge scan).

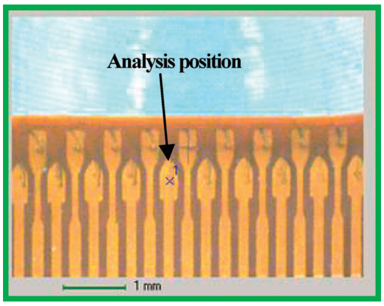


Fig. 5. Analysis position shown by an electronic camera.

5.2. Analysis for an ITO thin-film layer

A piece of an ITO thin-film layer deposited on a Si wafer was analyzed. The XRF spectra from the ITO layer are shown in Fig. 8. The Sn-Lα; peak is completely separated from the In-Lβ1 peak. For a comparison, the XRF spectrum for this sample obtained by an EDXRF system was also shown in Fig. 9. The spectrum resolution of WDXRF is clearly better than that of EDXRF, and therefore more accurate results can be obtained by WDXRF than those by EDXRF. The

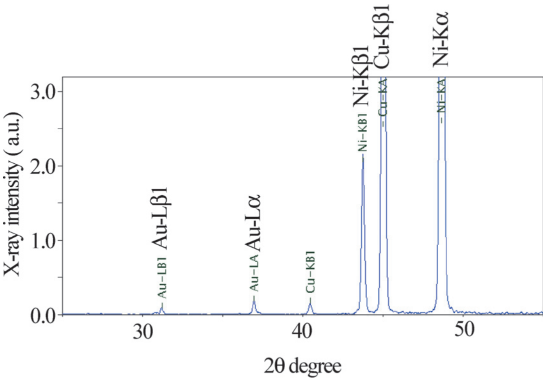


Fig. 6. XRF spectrum for the gold-coated terminal.

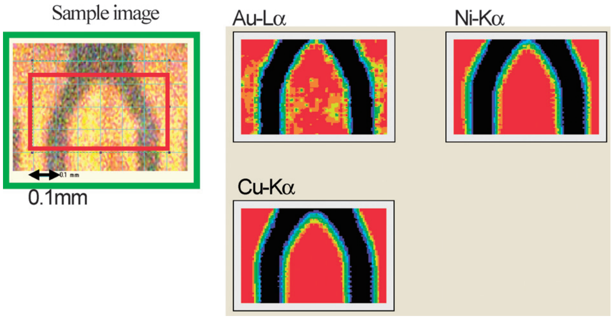


Fig. 7. Mapping analysis result of Au-Lα, Ni-Kα and Cu-Kα.

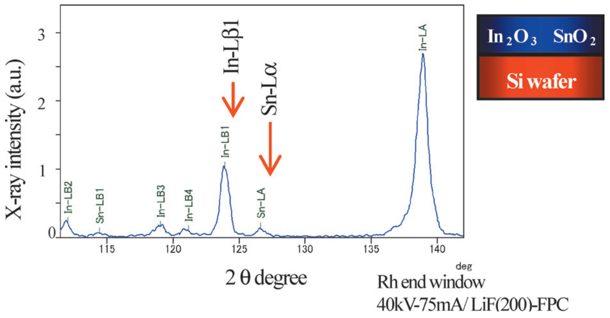


Fig. 8. WDXRF Spectrum showing In-L and Sn-L lines.

concentration and thickness of the ITO layer were determined by the fundamental parameter method for thin films. Experimental conditions are shown in Table 1 and the analysis results in Table 2. The resulted values agree with the target values, and the coefficient of variance (relative standard deviation) for the film thickness is less than 2%.

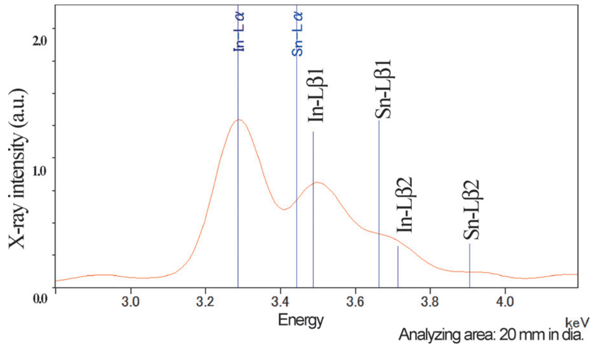


Fig. 9. EDXRF spectrum showing In-L and Sn-L lines.

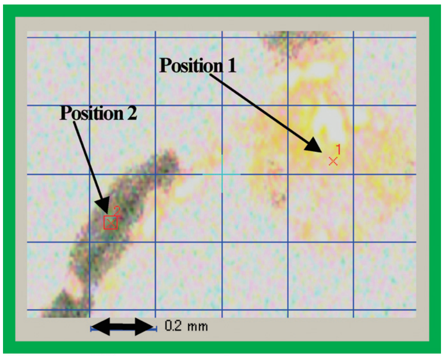
Table 1. Experimental conditions.

Element	In	Sn
Measured line	Lα	Lα
Analyzing crystal	LiF(200)	LiF(200)
Detector	F-PC	F-PC
Measuring time	100sec.	20sec.

Measuring area: 100µm in diameter
X-ray tube: 4 kW Rh-End window

Table 2. Quantitative XRF analysis of the ITO layer.

	Thickness(Å)	SnO_2 (mass%)	In_2O_3 (mass%)
Cycle=1	760	4.16	95.8
=2	739	3.19	96.8
=3	761	3.86	96.3
Average	753	3.68	96.3
Std. dev.	12.4	0.48	0.48
C.V.(%)	1.65	13	0.50
Target value	780	5	95



0.2mm

Fig. 10. Measuring positions 1 and 2.

5.3. Analysis of foreign substances in polymer

A micro-spot XRF analysis of foreign substances in a polymer sample was carried out using the semi-quantitative analysis (SQX) based on the standardless fundamental parameter method. The sample image and the positions of the micro-spot analysis are shown in Fig. 10. The XRF spectra from positions 1 and 2 are shown in Fig. 11, and the SQX results were tabulated in Table 3. It shows that Ir and Au were detected at position 2 but essentially undetectable in position 1, indicating that the

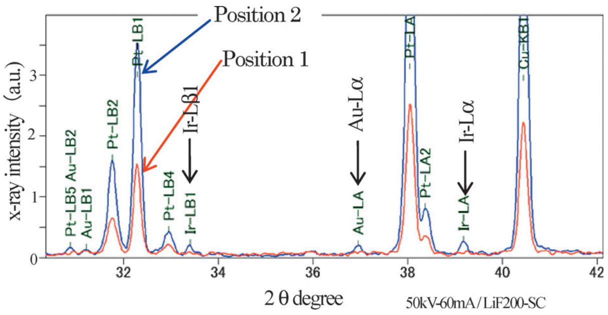


Fig. 11. XRF spectra obtained from positions 1 and 2.

Table 3. SQX results for positions 1 and 2 (unit: mass%).

Element	Fe	Cu	Pt	Ir	Au
Position 1	0.8	52	47.2	-	-
Position 2	0.8	54	44	0.4	0.8

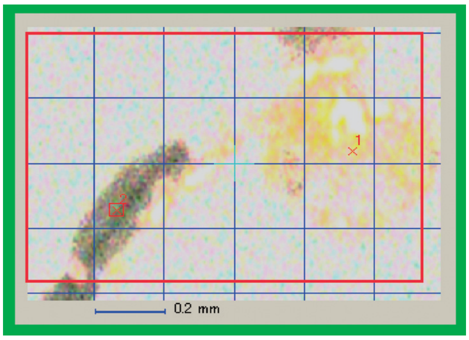


Fig. 12. Mapping area is shown by the red rectangle.

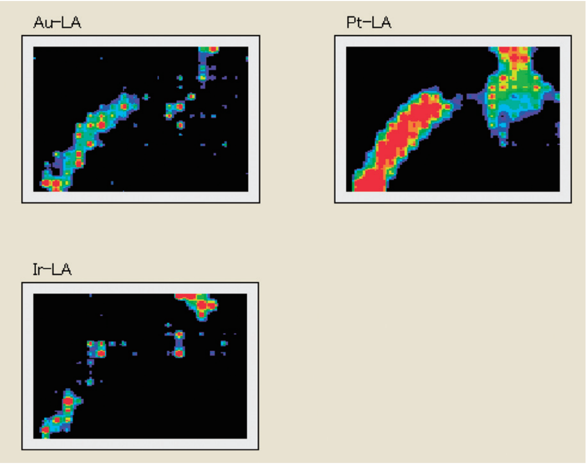


Fig. 13. Mapping analysis results for Au-Lα, Pt-Lα and Ir-Lα X-rays.

compositions at positions 1 and 2 are different.

XRF mapping analysis of the XRF intensities of Au-Lα;, Pt-Lα; and Ir-Lα; was performed inside the area defined by the red rectangle shown in Fig. 12. The results of the mapping analysis show that Au and Ir were concentrated at position 2, but practically neither Au nor Ir was detected at position 1 (see Fig. 13).

6. Summary

Our results show that the new WDXRF spectrometer equipped with a high-energy-type polycapillary lens was used successfully for high spectral resolution and sensitivity elemental analyses of 100-μm spots on thin films and foreign substances.

References

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