Feature Article

50th Anniversary Product

Development of the XGT-5000 X-ray Analytical Microscope

Shintaro Komatani, Sumito Ohzawa

[The Development Team]

Standing from the left:
Atsushi Bando, Minoru Shimooka, Shintaro Komatani, Yukio Ohga, Sumiyo Ishikawa, Shigetoshi Arai, Yoshihiro Yokota, Masaki Tanaka, Kota Inagaki, Yohei Kawamura, Kenichi Obori

Sitting from the left:
Yoshimichi Sato, Satoko Nishikawa, Sumito Ohzawa, Aya Kametani, Shintaro Miyasaka

The XGT series X-ray Analytical Microscope is a nondestructive analyzer that does not require preprocessing and facilitates elemental analysis of microscopic parts, element distribution analysis and internal structure observation. For the XGT-5000, a 50th Anniversary product, we advanced development of the X-ray guide tube as a key device for X-ray Analytical Microscopes and achieved intensities 20 to 50 times higher than ever before. Further, we improved operability drastically as a result of improvements in the optical image observation system and software. The XGT-5000 is used in various fields including research such as living body tissue analysis and archaeological sample analysis, quality control purposes such as foreign substance analysis and defect analysis, and sensitive analysis of harmful elements in compliance with WEEE/RoHS and ELV regulations. It significantly contributes to developments in these fields.

Introduction

In recent years, the progress of science and technology has promoted higher performance and density growth in electronic and electrical devices. In addition, nanotechnology progress is increasing requirements for structural analysis, physical properties and detecting foreign substances in extremely minute parts. Conventionally, SEM-EDX[*1] has been extensively used for analyzing microscopic parts. This analysis method enables composition analysis and element distribution analysis through its extremely high spatial resolution; however, preprocessing such as coating and cutting a cross-section of the sample is required. Thus, the sample could be heavily damaged due to exposure to electron beams and a vacuum, which then makes the analysis difficult. On the other hand, XRF[*2] can conduct element analysis easily; however, the analysis area is normally large, i.e., ϕ5 to 10 mm, which makes it impossible to analyze microscopic parts. To solve these problems, HORIBA developed the XGT-2000 X-ray Analytical Microscope in 1996, and in 2002 achieved a more intense X-ray microbeam producing a sensitivity 20 times higher than before with the XGT-5000, a 50th Anniversary product (Figure 1).

*1: An analyzer that combines a scanning electron microscope (SEM) and energy dispersive X-ray analyzer (EDX).

*2: X-ray fluorescence analyzer.

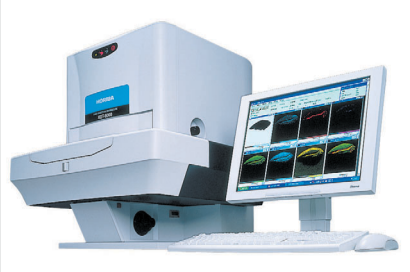

Figure 1 XGT-5000

Structure of the Analyzer

XGT-5000 X-ray Analytical Microscope consists of the following: X-ray generator, X-ray guide tube that narrows down X-rays, stage that scans a sample, fluorescent and transmitted X-ray detectors, optical image observation unit, signal processing unit, and PC.

X-rays generated from the X-ray tube are narrowed down and applied to a sample. Then, fluorescent X-rays generated from the sample and transmitted X-rays that have passed through the sample are detected. This enables elemental analysis for microscopic parts, element distribution analysis and inner structure analysis nondestructively. The structural outline of the analyzer and measurement schematic are shown in Figure 2 and Figure 3.

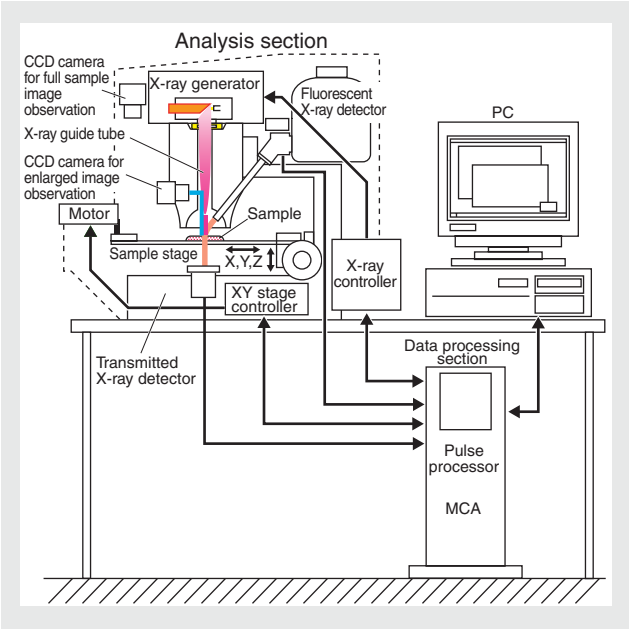

Figure 2 Structural Outline of the Analyzer

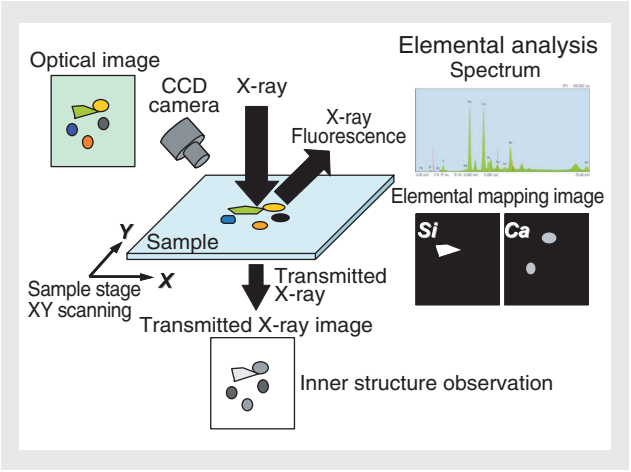

Figure 3 Measurement Schematic

Features of the XGT-5000

Features of the X-ray Analytical Microscope

This type of microscope is less damaging to samples, does not require vacuuming or preprocessing, and facilitates elemental analysis for microscopic parts and elemental distribution analysis. Further, it is possible to observe internal parts of the sample and measure diffracted X-rays, which are impossible with SEM-EDX. Thus, it has new possibilities such as analysis of living body tissue or internal defect of a sample and microscopic part analysis inside the sample. Table 1 shows a comparison of SEM-EDX and the X-ray Analytical Microscope.

Table 1 Comparison of SEM-EDX and X-ray Analytical Microscope

	SEM-EDX	X-ray Analytical Microscope
Excitation	Electron	X-ray
Spatial resolution	0.1 to 1 μm	10 μm
Elements detected	Be to U	Na to U
Depth of analysis	0.1 to several μm	Several mm and less
Transmitted X-ray	None	Possible
Vacuum sample chamber	Necessary	Unnecessary
Preprocessing (Coating, Dry process)	Necessary	Unnecessary
Sample damage	is present	Very slight

Realization of High-intensity X-ray Microbeam

Basic performance of the X-ray Analytical Microscope including spatial resolution and analytical sensitivity depends on the narrowness of the X-ray beam and its intensity. To achieve an intense X-ray microbeam, we adopted an X-ray guide tube[1]-[4] as a focusing device. The X-ray guide tube is a device that can narrow down X-rays so keeping high intensity by launching them into an extremely smooth surface at a low angle below the critical angle and utilizing total reflection without reducing intensity.

Figure 4 shows the principle of the X-ray guide tube.

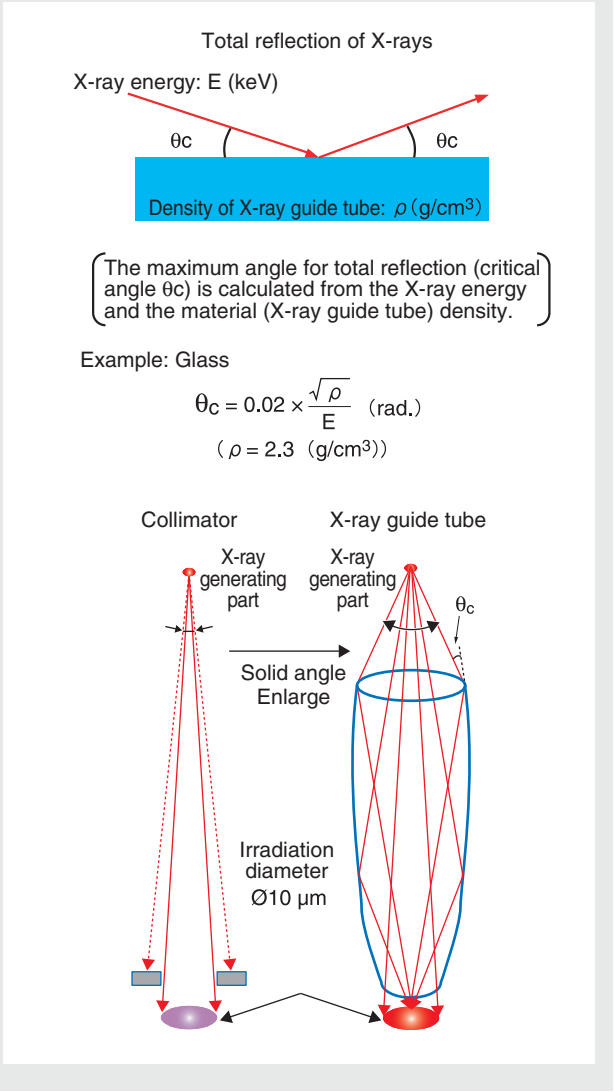

Figure 4 Principle of the X-ray Guide Tube

In the case of analyzers using an X-ray tube and collimator, the X-ray intensity decreases as X-rays are blocked and narrowed down. Thus, spatial resolution is practically limited to several hundred μm. However the XGT-2000 achieved a spatial resolution of 10 μm by means of the X-ray guide tube. Further, in the XGT-5000, the X-ray guide tube has been improved and the geometry of the detector and analyzer has been reconsidered. As a result, the intensity is 20 times higher than before, i.e. 100 times greater has been achieved as a percentage of the collimator (Figure 5).

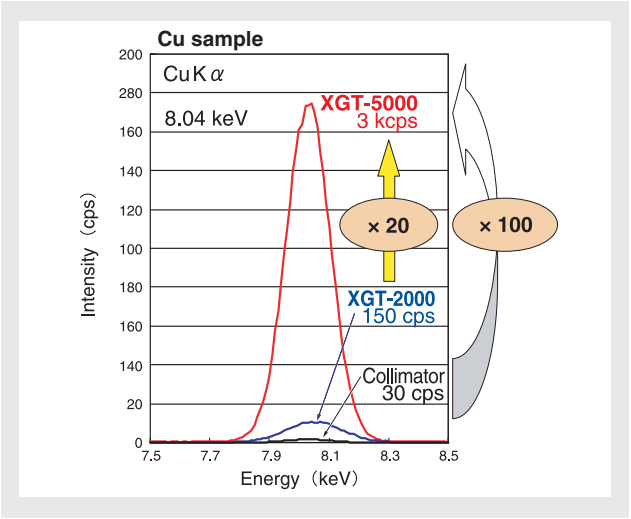

Figure 5 Improvement of X-ray Intensity

Improvement of Positioning Accuracy and Operability

Conventionally, optical images are observed from a slanting direction against X-ray beams. If the sample falls out of the focus due to projections and depressions, the optical image observation point is displaced from the measurement point. In the XGT-5000, an optical system that can reveal the analyzing position coaxially with X-rays has been developed. As a result, accurate analytical positioning from the optical image became possible even if the sample is slightly out of focus. Further, we have developed an optical image observation system by combining a low-power camera for observing the whole sample with a high-power camera for accurate positioning. Once the analyzing position has been narrowed down from the whole sample, switching to the detailed image enables accurate positioning. Time taken for analysis positioning has been greatly reduced and operability has been improved (Figure 6).

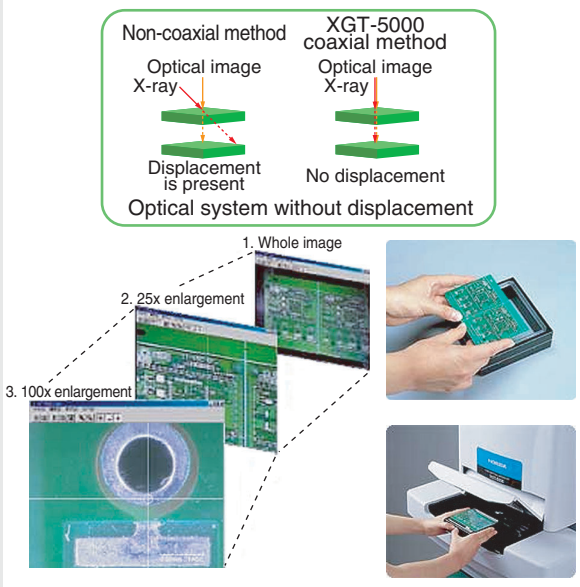

Figure 6 Improvement of Positioning Accuracy and Operability

Applications of the X-ray Analytical Microscope

The X-ray Analytical Microscope contributes to the development of various fields including living body tissue research, foreign substance analysis, defect analysis, and archaeological sample analysis. In living body tissue analysis, for example, teeth and bones are analyzed for researching recalcification. In foreign substance analysis, production processes can be improved by analyzing foreign substances in food packages. In defect analysis, ion migration of wire bonding materials inside IC's is analyzed without exposing the wires. In these ways, the X-ray Analytical Microscope provides precious information that cannot be obtained from other analyzers. Figure 7 shows an example of foreign substance analysis.

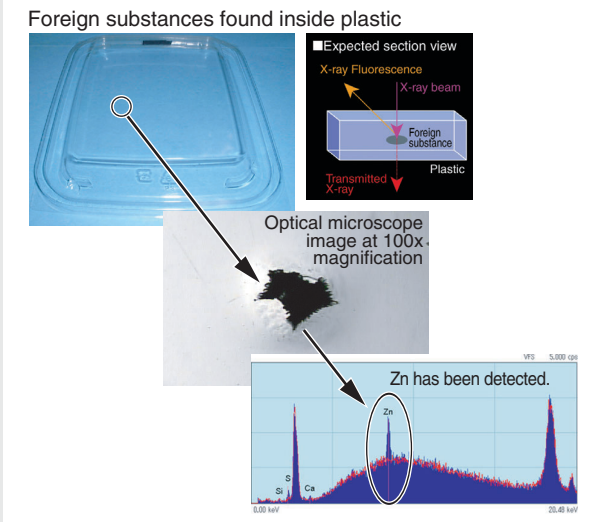

Figure 7 Foreign Substance Analysis of Plastic

WEEE/RoHS, ELV

In response to increasing awareness of environmental issues, the EU has enforced ELV regulations since 2003 that prohibit inclusion of toxic elements (Cd/Pb/Hg/Cr^{6+}) in automotive parts. In 2006, WEEE/RoHS regulations will be enforced that prohibit inclusion of toxic elements (Cd/Pb/Hg/Cr^{6+}/PBB/PBOE) in electrical products. Normally, these elements are analyzed by emission spectrometry such as ICP. However, because the number of parts is extremely large, analysis of all parts by means of ICP is not realistic. Thus, we developed the X-ray fluorescence analyzers XGT-1000WR and XGT-5000WR, which can perform screening easily in a short time. These analyzers are sufficiently sensitive as a result of the X-ray analytical microscope technology with provision of an X-ray filter suitable for toxic element analysis. Time required for analysis can be reduced drastically and large volumes of parts can be analyzed very effectively. Further, the mapping function of XGT-5000WR enables distribution analysis as well as point analysis. Figure 8 shows the lead distribution on a surface-mount printed circuit board.

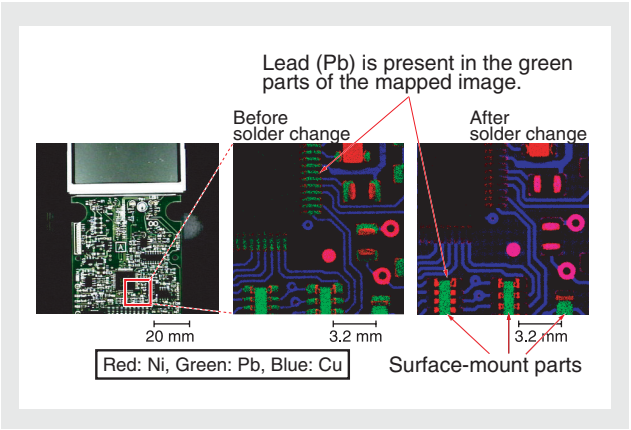

Figure 8 Lead Distribution on a Surface-mount Printed Circuit Board

Conclusion

We would like to continuously develop not only more intense and microscopic X-ray guide tubes but also minute focus X-ray tubes. This will enable miniaturization of the analyzer and make beams more intense and microscopic so that the analyzer can be used for medical diagnosis as well as R&D and quality control.

Reference

[1] No. 1828290 in Japanese patents.

[2] F.H.A. Janssens, F.C.V. Adams, *Microscopic X-ray Fluorescence Analysis* (John Wiley & Sons Ltd Chichester 2000).

[3] N.Yamamoto, A micro-fluorescent/diffracted X-ray spectrometry with a micro-X-ray beam formed by a fine glass capillary, *Rev.Sci.Instrum.* **67**, 3061- 3074 (1996).

[4] Daniel D.J.Thiel, Production of intense micrometersized X-ray beams with tapered glass mono capillaries, *Rev.Sci.Instrum.* **64**, 2872 - 2878 (1993).