Electron Probe Microanalyzer with Automatic Analyzing and Image Processing System*

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1 Introduction

Microprobe analyses employing electrons, ions or X-rays are widely utilized as analysis and evaluation methods for supporting the development of new technology and new products in various fields including electronic materials and new materials, as well as steel. In particular, the X-ray microanalyzer (EPMA) is used most extensively because it is superior for quantitative analysis and does not require a high vacuum.[1)]

Recently, computer technology have brought about the installation of various functions for analysis, such as the elemental mapping in wide areas and the resultant image processing.[2)] Furthermore, a multilayered X-ray dispersion element[3)] has been developed for analysis of carbon, oxgen and nitrogen, and accordingly the effectiveness of EPMA is becoming higher.

Considering this situation, the authors developed and installed a new EPMA in our company in July 1987 which has various functions such as automatic analysis without an operator, image processing, and so on. This system has to date been working well and has provided notable results for investigating various products and the processes. This paper gives an outline of the new EPMA and its application.

2 Outline of the System

Figure 1 shows the construction of the system and **Table 1** shows the main specifications. This system can roughly be divided into the measuring function and the control and data processing functions. The measuring function consists of a spectrometer, an electron beam control unit, a high-speed mapping system, and a recorder. The control and data processing functions consist of a computer, an image display system, terminals for analysis and image processing, a color hard copy device, and a printer. This new EPMA has the following additional characteristics which were brought about by advanced computer and hardware construction:

(1) Fully Automatic Analysis System

Each analytical point and each analytical mode, such as qualitative analysis, quantitative analysis, line analysis and photography, can be combined in any combination in this automatic analysis system.

Furthermore, by automatic judgment based on the results of qualitative analysis, the analytical condition of the other mode can be set up automatically. For such a full automatic analysis system to work, it is important not only to design capable software but also to stabilize the instrument. For this purpose, the electron gun was improved to stabilize the beam current, so as to control the variation in the beam current within 0.9% for 15-h unmanned operation. Moreover, a mechanism, in which the beam current

Table 1 Main specifications

Term	Specification
Accelerating voltage	0~30 kV, 0.5 kV step
Diameter of electron beam	min 100 Å
Channel	6 (full scanner 4, semi scanner 2)
Signal for analysis	X-ray
	Backscattered electron
	Secondry electron
	Sample current
Sample size	max 100 mm (W) × 100 mm (D) × 40 mm (H)
Area of measuring	max 90 mm × 90 mm
Speed of stage scanning	max 10 mm/s
Integrating time	min 1 ms
Display	CRT:19 inch (16 colors)
Data processing	VAX station GPX/II (32 bit type)
	Hard disc (71 MB)
	MT (95 MB)
	Automatic analysis and Image analysis

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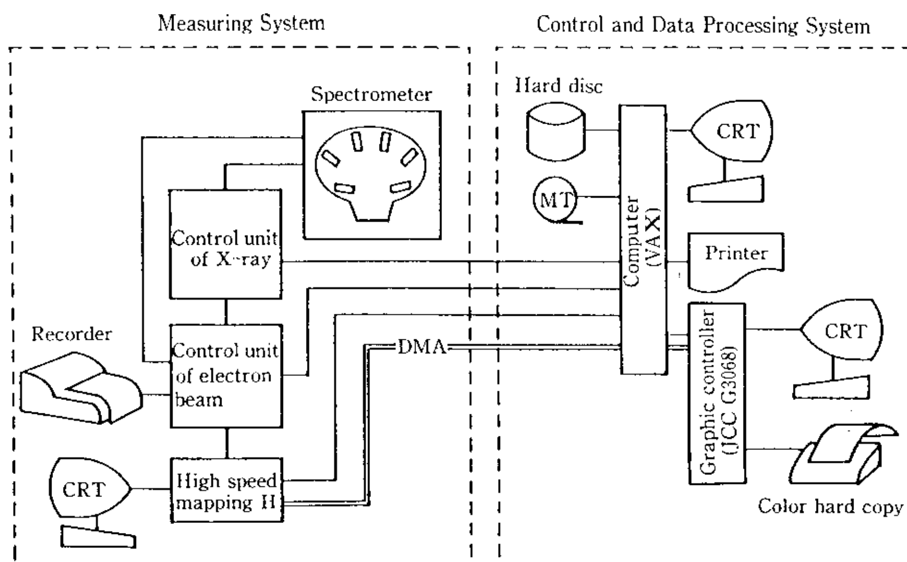

Fig. 1 Blockdiagram of system

is controlled by the monitor current, was developed to compensate for the variation of the beam current within 3% for longer periods. In addition, both the optical microscope and the electron optical system were equipped with automatic focusing mechanisms in order to bring the sample position and the electron beam into exact focus without an operator.

(2) Improvement of the Electron Optical System for Increasing the Beam Current on the Fine Beam

If the beam current becomes larger when the incident electron beam is focused to a fine probe, the intensity of characteristic X-rays become higher. Therefore, the electron optical system, including the condenser lens, was improved. As a result, for example, the beam current reaches $10^{-8.5}$ A when the accelerating voltage is 10 kV and the beam diameter is 100 nm, which is more than 10 times the value that reaches before the improvement. This has given the new EPMA an excellent measuring precision of small area and minor elements.

(3) High-Speed Mapping

High-speed mapping of a large sample, as large as 100 mm × 100 mm, is possible. The sample stage, which can hold the large sample of 100 mm square, moves with a minimum stepping size of 1 μm and a maximum scanning speed of 10 mm/s. With the high-speed mapping system, the time required for data gathering is considerably shortened. The measuring time was, for instance, about 50 min when the sample size was 50 mm × 50 mm (stepping size, 100 μm; integrating times, 10 ms). It is quite effective to increase the beam current for high-speed mapping by beam scanning in a small area. The result of high-speed mapping is displayed on the color CRT in real-time.

(4) Adoption of Synthetic Multilayered Dispersion Element

Sensitivity to ultra-light elements is high since the new EPMA has a synthetic multilayered dispersion element. The W/Si multilayered dispersion element ($2d = 72.2$ Å, $r = 4$ in.) was equipped for the detection of nitrogen whose intensity is weak if ordinary lead stearate (PbSD) is used. As a result, the intensity of N $K\alpha$ is about 6 times as large as that with 5 inches' PbSD, and the intensity of O $K\alpha$ is about 13 times as large as that with 4 inches' PbSD. The wavelength resolution (half width) of N $K\alpha$ was 1.44 Å when using the multilayered dispersion element, while it was 1.00 Å when using PbSD. Since the difference between these values is not significant for practical use, the synthetic multilayered dispersion element is quite effective for analyzing ultra-light elements.

(5) Simultaneous Analysis of 6 Elements

The main spectrometer was improved by installing four full scanners and two semi-scanners, so that the number of elements measurable simultaneously was increased from 5 to 6. Accordingly the combination of measurable elements became more flexible. For example, the $K\alpha$ of three heavy elements such as Fe, Zn, and Ni can be measured at the same time. Consequently, workability is improved in mapping, line analysis, etc.

(6) Expansion of Image Processing and Analyzing Function

The image processing and analyzing functions are expanded by adopting a 32-bit superminicomputer (Micro VAX II) of multi-processing type. An image analysis function was installed, in addition to the

image display function with which the mapping image is displayed on the CRT for controlling the color, and the image processing function with which the mapping image is converted by mathematical treatment such as filtering and contrast enhancement. The image analysis function includes, for example, a function for extracting the geometrical parameters, such as heywod diameter or flatness, and statistical treatment of them. Distribution of minor elements, and composition ratio, chemical state and the shape of impurities or dispersed particles can also be analyzed easily by using the functions described above.

3 Application

Among the many characteristics of the new EPMA described above, mapping is used most frequently because the data appeals visually. This is because the distribution of elements or components, rather than the average composition, in a material is becoming more and more important in materials investigation. The analytical results of steel and ferrite are shown below as examples of this function.

3.1 Distribution of Mn in Cast Steel

Photo 1 shows the distribution of Mn at the center of the cross section perpendicular to the rolling direction in a cast sample prepared by a casting simulator. This mapping result was obtained by stage scanning. The center segregation of high Mn content and the fine dendritic structure of the second or third order were observed clearly.

In this measurement, the sample area was 50 mm × 50 mm (10 μm step) and the measuring time was about 50 min. The obtained image was treated with a median filter.

3.2 Distribution of Ca at the Grain Boundary in Ferrite

Photo 2 shows the distribution of Ca at the grain boundary in commercial ferrite, as an example of the measurement of a minor element in a small area. The concentration of Ca at the grain boundary was observed clearly. Ca was added to the starting material or during calcining. This photograph shows the high sensitivity and resolution power of this instrument. Photo 2 is the data treated with a mean filter.

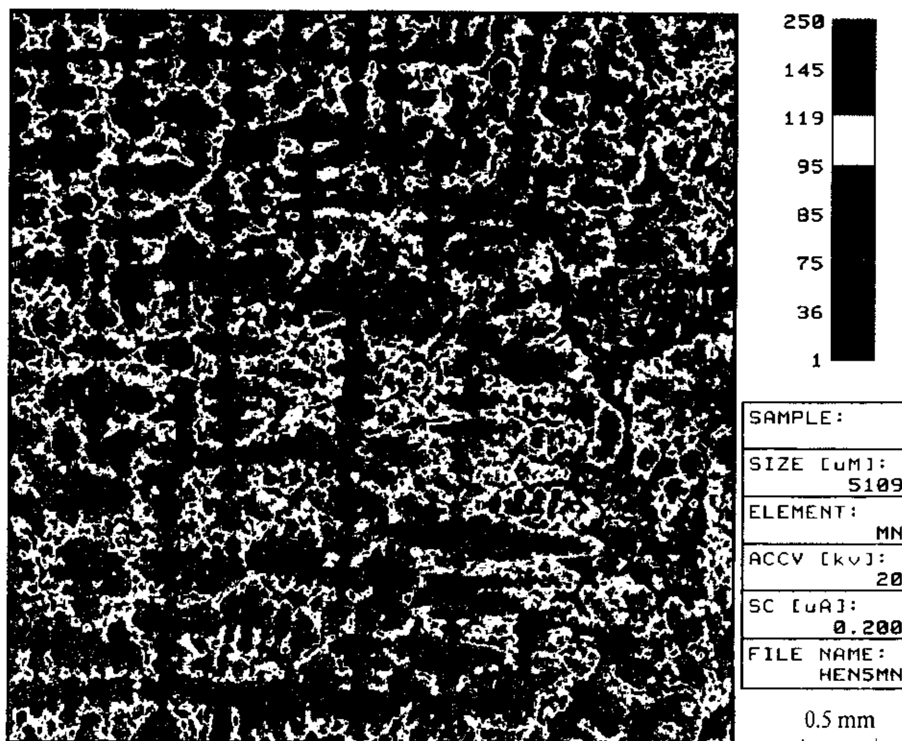

Photo 1 An elemental distribution of Mn at centerline in a steel ingot cast by a segregation simulator

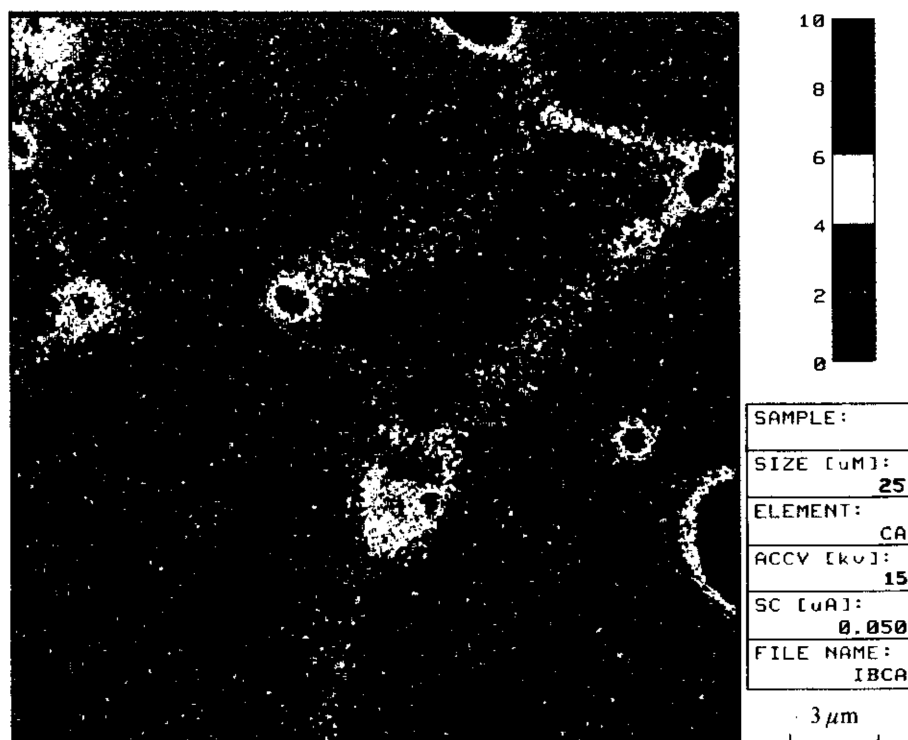

Photo 2 An elemental distribution of Ca in ferrite

4 Concluding Remarks

The outline, characteristics, the application of the new EPMA have been described. This automatic analysis system has not been fully developed yet because its software has not been completed. However, in the future, this system will be used in various fields as an analyzer with high efficiency and various functions. With such a prospect in mind, development of practical techniques, including an analysis of a thin layers in the order of semimicron thickness and nondestructive analysis in the depth direction, are in prospect.

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