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New Products & Technologies

Application of 400-kV High-Resolution Analytical Electron Microscope to Materials Research*

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1 Introduction

Microstructure control plays a critical role in improving mechanical and electromagnetic properties of steels, ceramics, semiconductors, and magnetic materials. To date, the analytical electron microscope has been an extremely powerful tool for the investigation of microstructures and it has provided useful data on lattice defects and for microanalysis. Microstructure control at the atomic level is now required for improving various properties of materials to the highest standards. It is now necessary to investigate atomic arrangements and lattice defects, and to identify the species and concentration of elements simultaneously in specific areas of specimens. However, conventional electron microscopes have proved to be insufficient both in resolving power and in the analytic capability of light elements. On this basis, Kawasaki Steel has introduced a new 400-kV high-resolution analytical electron microscope. This report describes specifications of the equipment and some of the data obtained by using it.

2 Features of the Equipment

This equipment has such a high resolving power and various analytical functions that observation of atomic arrangements and simultaneous analysis of micro area are capable. In order to increase the resolving power of the analytical electron microscope, it is necessary to minimize the spherical aberration of an objective lens by raising accelerating voltage. Accordingly, an accelerating voltage of 400-kV which is higher than conventional levels is used.

Under the limitations of the objective lens pole piece, the 400-kV electron microscopes are roughly divided into two types: one with emphasis on the high resolving power (0.19 nm) and the other with emphasis on analytical function (resolving power: 0.25 nm). The subject equipment is designed to have both high resolving power (0.22 nm) and analytical function by employing an objective lens pole piece of special specification.

Further, the 400-kV accelerating voltage provides the electron microscope with the following characteristics:

(1) Since the penetrating power of the electron beam increases, thicker specimens can be observed with little surface effects, and lattice defect images, such as dislocations, can thereby be obtained under the same conditions as for bulk specimens.

(2) In the case of element analyses by X-ray spectrometer and electron energy loss spectrometer, the background intensity is lowered and a spectra having a high S/N ratio can be obtained.[1]

The appearance and the specifications of the equipment are shown in **Photo 1** and **Table 1**. With these characteristics, the following measurements and analyses can be performed:

Table 1 Specifications of 400-kV high-resolution analytical electron microscope

Accelerating voltage	(kV)	400
Resolution	(nm)	0.14 (TEM lattice image) 0.22 (TEM point resolution) 1.0 (STEM image) 2.0 (SEM image)
Element analysis		Na~U (Conventional EDX) C~U (UTW-type EDX) Li~U (EELS)
Area of electron diffraction	(nm)	5
Other faculties		Analysis of atomic image Convergent beam electron diffraction Electron channeling pattern Reflective electron diffraction Reflective electron image

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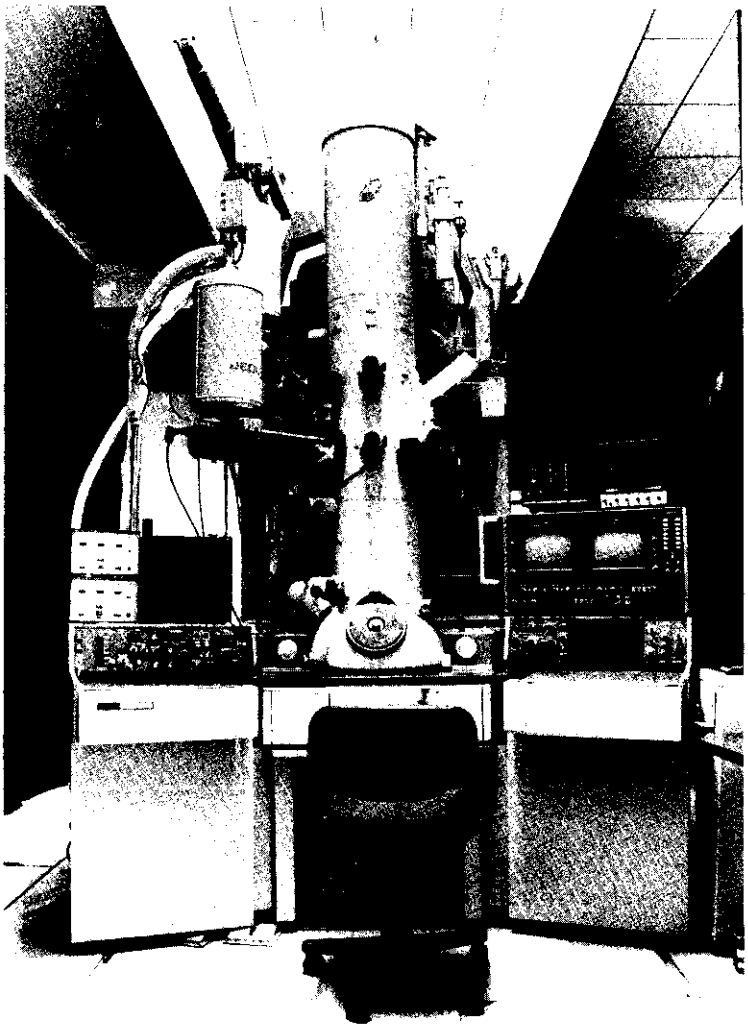


Photo 1 Appearance of 400-kV high-resolution analytical electron microscope

(1) Atomic images can be observed in metals and other materials, with its high resolving power.
(2) Atomic image contrast can be calculated theoretically by the image analyzer and the location of the atom can be determined by comparing observed atomic images with the calculated one.
(3) Elements ranging from Li to U can be analyzed with high sensitivity by combining the energy-dispersive X-ray spectrometer (EDX) and the electron energy loss spectrometer (EELS), with EDX consisting of the conventional spectrometer type and ultra-thin window (UTW) type.
(4) Atomic arrangements near the surface can be determined using reflective electron diffraction and reflective electron images.
(5) Specimen thickness, accelerating voltage and the Burgers vector of dislocations can be determined using convergent beam electron diffraction.
(6) Crystal direction and surface strain can be determined using electron channeling pattern.

3 Examples of the Measurement

3.1 High-Resolution Image of Silicon Wafer

Since SiO_x precipitates in the silicon wafer deteriorate the electrical properties and act as the nucleation site for stacking fault formations during the heat treatment process, they should be strictly controlled. Consequently, for their above-mentioned analyses, a high-resolution image is indispensable for high quality wafer production. **Photo 2** shows the lattice image of a silicon wafer with the electron beam parallel to the [001] direction. Lattice fringes corresponding to the (220) and ($\bar{2}20$) planes which cross each other at right angles at spacings of 0.192 nm were clearly observed. No disappearance or bending of lattice fringes indicates that there is no precipitate in the area. **Photo 3** shows both an image of the SiO_x precipitates formed under heat treatment at 800°C, for 8 h from the [001] direction and an example of ele-

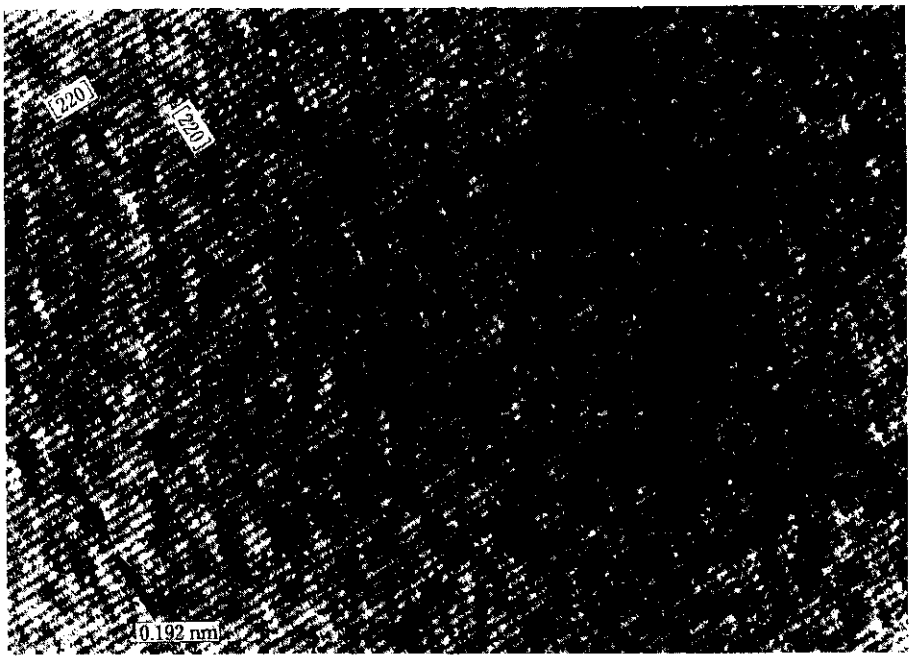
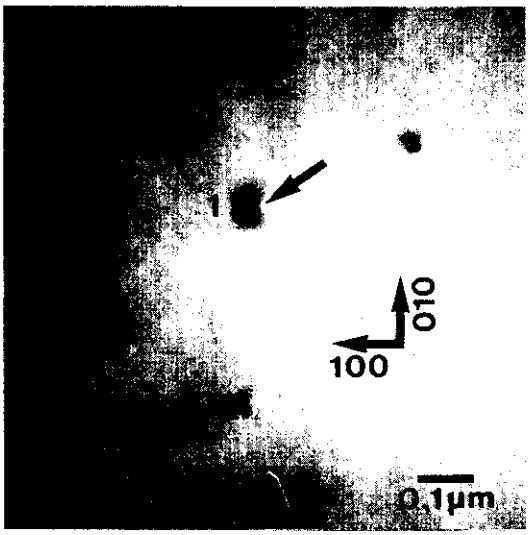


Photo 2 Lattice image of a silicon wafer



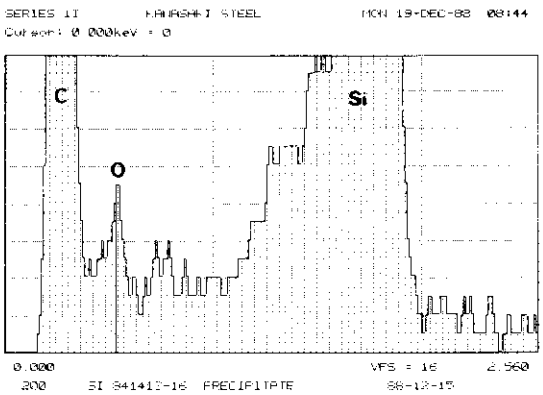


Photo 3
Fine oxygen precipitates (above) and EDX spectrum from the precipitate indicated by the arrow (below) in the silicon wafer after annealing at 800°C for 8 h

ment analysis with UTW-EDX from the position of the arrow. In the black patterns indicated by 1 and 2, a long white stripe extending in the directions of [100] and [010], respectively, is clearly observed. The white stripe represents precipitates and the black pattern around them is considered to have come from a strain in the matrix. In the X-ray spectrum of the UTW-EDX, a K-line from oxygen is clearly seen. It has been indicated that the in-silicon oxygen precipitates formed during a low temperarure heat treatment around 800°C are plate-like in shape lying on the {100} silicon lattice plane, and their peripheries lie in the ⟨110⟩ direction.[2] If the precipitates are projected in the [001] direction, they would appear a long stripe in the directions of [100] and [010]. This analysis well corresponds with the observed results indicated in Photo 2. Consequently, the bright stripe is judged to be a fine SiO_x precipitate.

The observation and analysis of such fine precipitates were made possible by the combination of high-resolution observation and element analysis unit.

3.2 High-Resolution Image of Nd-Magnets

Nd magnets are expected to replace Sm-Co magnets as permanent magnet materials in the future. In this magnet the second phase, as well as main phase, affects magnetic properties significantly, and the control of grain boundaries is important in improving resistance to corrosion. **Photo 4** shows the lattice image near the boundary between the main phase (tetragonal structure) and the second phase. The incidence direction of electron beam is $[1\bar{1}0]$ of the main phase and the lattice fringes which correspond to the (001), (110) and $(\bar{1}\bar{1}1)$ lattice planes are clearly recognizable. Lattice fringes were also recognizable in the second phase. Analysis of electron diffraction patterns indicates that it is a Nd-rich phase (face centered cubic structure, lattice constant = 0.52 nm)[3] and the lattice fringe corresponds to the (111) plane. It is shown that the lattice fringes in both phases continue through the phase boundary indicated by the arrow. This observation indicates that there is no third phase at the boundary between the main phase and the second phase. With the conventional electron microscope, it is impossible to identify the existence of the extremely thin third phase at the boundary. However, with the high resolution image with the diffraction condition for the simultaneous formation of lattice images with the main and the second phases, the accurate observation about microstructure of boundaries can be obtained.

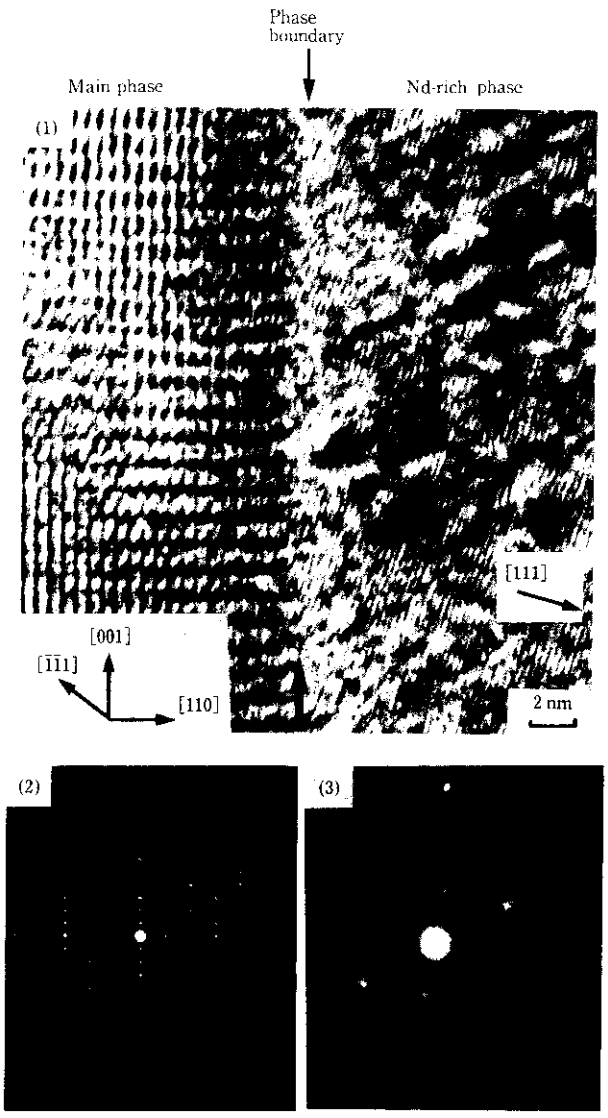


Photo 4 Lattice image of the boundary between the main phase and the Nd-rich phase in the Nd magnetic material (1) and electron diffraction patterns from the main phase (2) and the Nd-rich phase (3)

References

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