HITACHI SCIENTIFIC INSTRUMENT
TECHNICAL DATA
TEM

SHEET NO. 110

SUBJECT: STRUCTURE ANALYSIS OF FINE PRECIPITATES IN METALS USING AN FIB/TEM SYSTEM

INSTRUMENT: HF-2200 COLD FIELD EMISSION TEM WITH SCANNING ATTACHMENT
FB-2000A FOCUSED ION BEAM SYSTEM

CONTENTS

1. INTRODUCTION

A specimen preparation technique using FIB/TEM systems permits an excellent specimen preparation for transmission electron microscopy allowing 0.1 μm or thinner specimens. This technique makes it possible to check the specimen conditions during FIB milling by SEM/STEM imaging at a high voltage of 200 kV. We report here on evaluation of a fine precipitate in steel using this technique.

2. METHODS

2.1 Instrumentation

We have used an FB-2000A FIB system for specimen preparation. For precise positioning of the area of interest, we have used a cold field emission analytical electron microscope, the HF-2200 with SEM/STEM attachment which allows observation of the specimen during a milling process. We have used a compatible specimen holder which allows specimen milling and TEM observation repeatedly. This compatible specimen holder can be rotated by 360° in vacuum so that it allows observation of SE images of both sides of a cross-sectional specimen without repositioning.

2.2 Thinning process of a specific area of interest

Fig. 1 shows a sequence of procedures for thinning a bulky specimen without changing or losing the area of interest. The specimen is trimmed at a size of about several tens of microns and mounted on a compatible holder. It is, then, milled to a thickness of about a few microns using an FIB system (Fig. 1a). The specimen is moved to a TEM for observation in STEM mode at an operating voltage of 200 kV (Fig. 1b). The STEM image allows examination of inner structures of the specimen which is about a few microns thick. Following the microscopy in STEM mode, it is also examined in SEM mode at the same 200 kV (Fig. 1c). By checking the distance from the surface to the object of interest in a cross-sectional specimen, determine the milling volume. By repeating the above steps (a through c), it is possible to make a site-specific thin section.

3. THINNING OF A PRECIPITATE IN STEEL

Figs 2 through 5 show an example of thinning of a precipitate in steel. Fig. 2 shows a STEM image (a) and SEM images (b) and (c) of a 2 μm thick specimen prepared by FIB and recorded at 200 kV. STEM image (a) shows a precipitate in a form of a white particle. Arrows show a precipitate selected for the object of interest. STEM image (a) shows the precipitate at about 0.2 μm in diameter. SEM image (2b) shows a part of the precipitate in the specimen. But SEM image (2c) does not show the precipitate as indicated by the arrow. From these images, we can guess that the precipitate of interest is located close to the milling edge in Fig. 2b. Fig. 3 shows a STEM image (a) and SEM images (b) and (c) of a specimen of about 1 μm thick milled by FIB at both surfaces. Fig. 3b shows a precipitate which is about the same size as shown in Fig. 3a. We can guess that the central portion of the precipitate is shown in these images. While Fig. 3c does not show the precipitate of interest. We can assume that the precipitate is located deep inside from the milling edge of the specimen. Fig. 4 shows a result of observation of the same specimen at a thickness of about 0.5 μm after further milling from the Fig. 3c condition. In Fig. 4 the images of the precipitate in STEM mode (a) and SEM mode (b) are almost the same size. Fig. 4c shows SEM image of the precipitate which is a cross-section milled halfway to the center. Fig. 5 shows STEM image (a) and SEM images (b) and (c) of the same specimen at about 0.1 μm after further milling by FIB. The three images (a), (b) and (c) show the precipitate at the same size. We can, therefore, assume that this is the cross-section of the precipitate of interest, sectioned right in the middle.

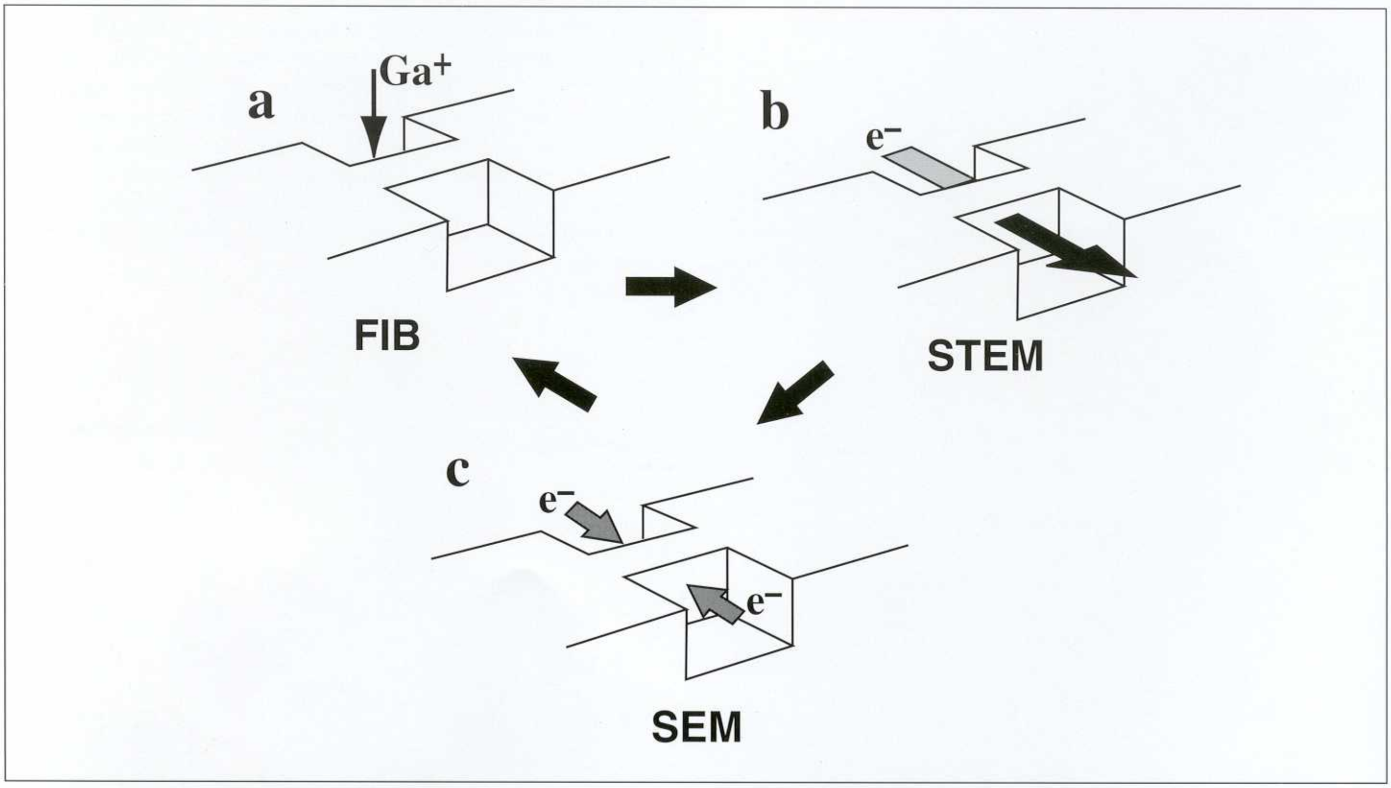

Fig. 1 A sequence of procedures for thinning a bulky specimen by combining FIB milling (a), STEM image (b) and SEM image (c)

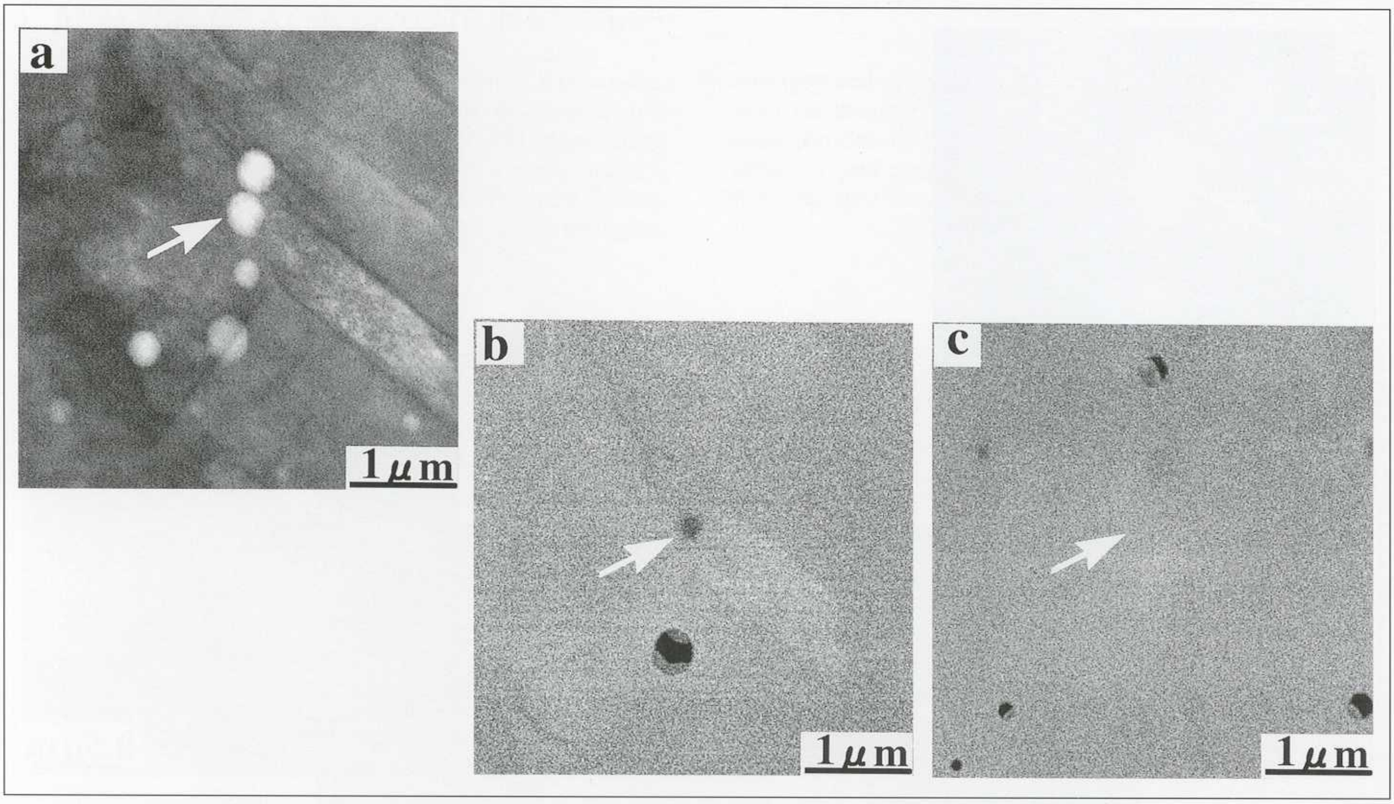

Fig. 2 A STEM image (a) and SEM images (b) and (c) of a precipitate in steel specimen

Accelerating voltage: 200 kV
Specimen thickness: 2 μm

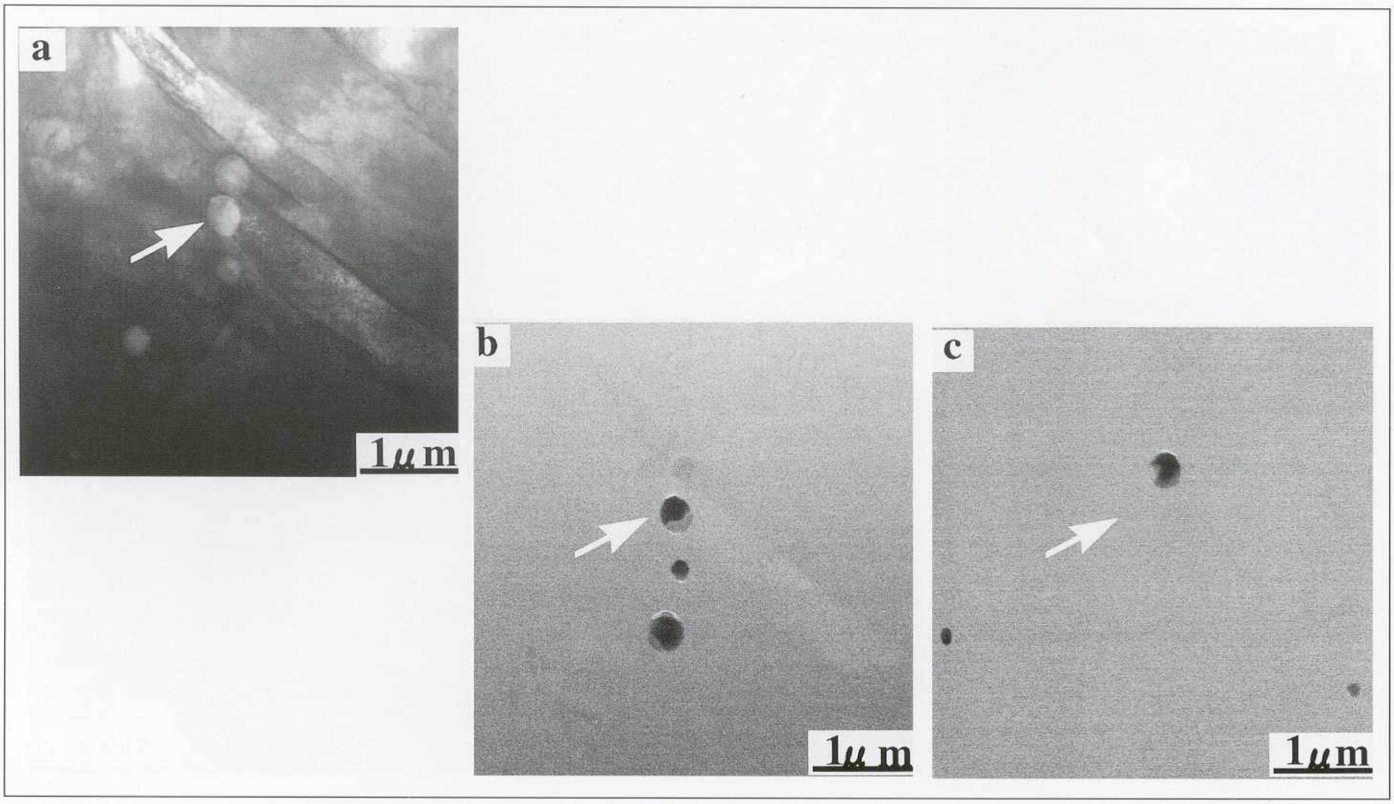

Fig. 3 A STEM image (a) and SEM images (b) and (c) of a precipitate in steel specimen

Accelerating voltage: 200 kV
Specimen thickness: 1 μm

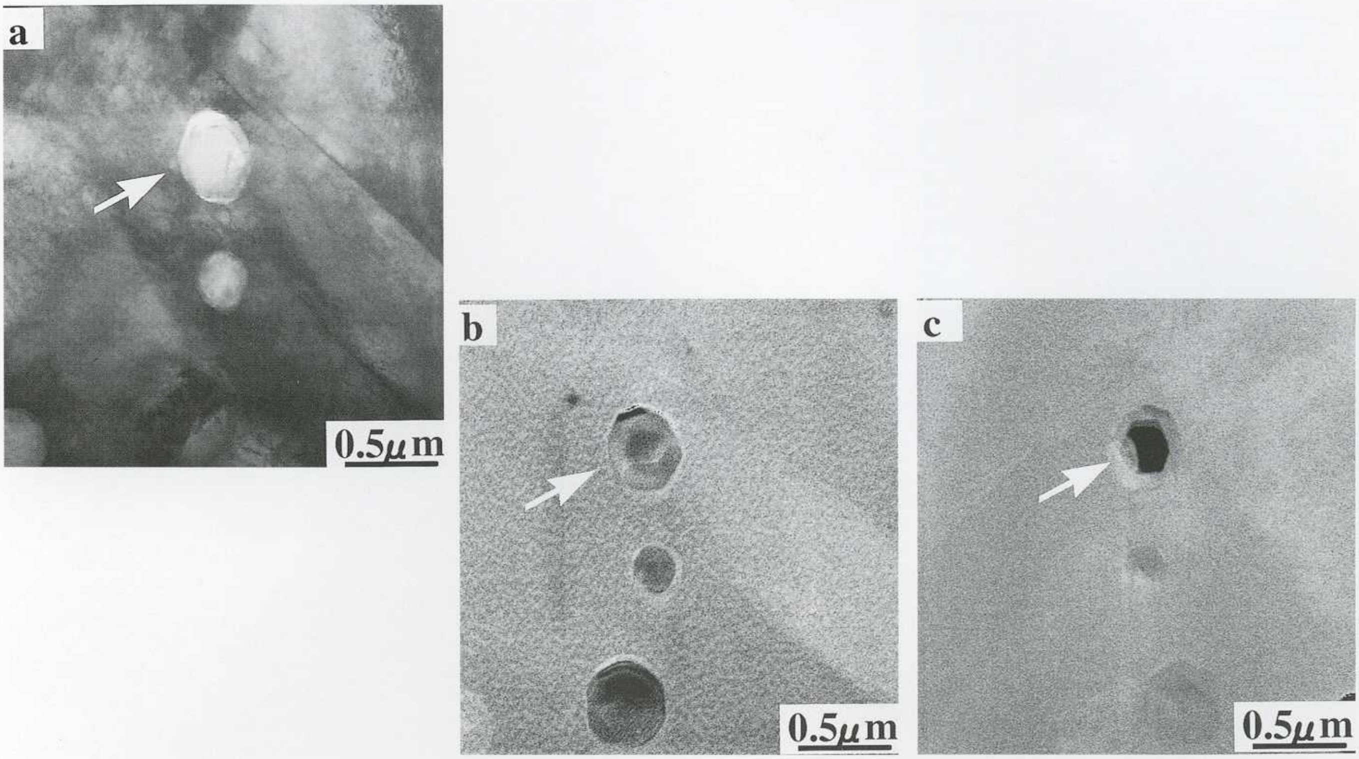

Fig. 4 A STEM image (a) and SEM images (b) and (c) of a precipitate in steel specimen

Accelerating voltage: 200 kV
Specimen thickness: 0.5 μm

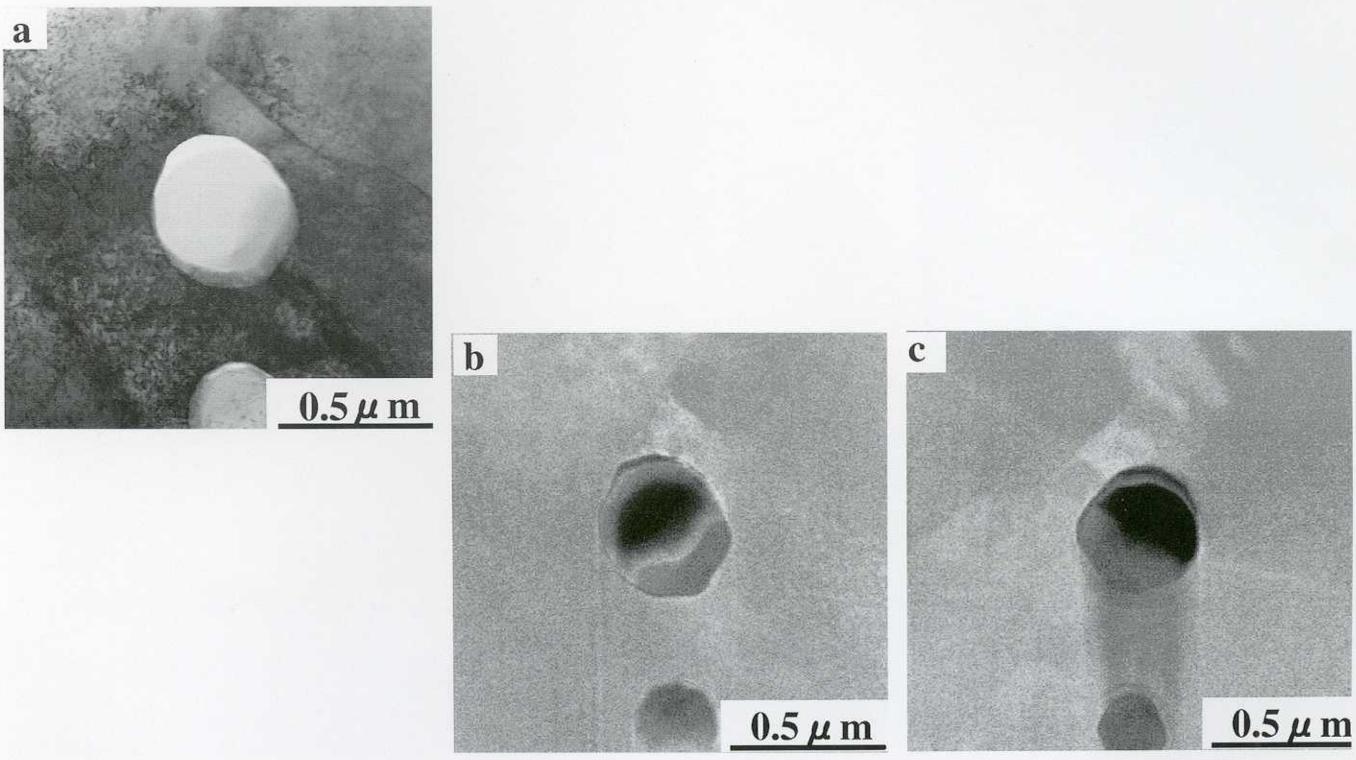

Fig. 5 A STEM image (a) and SEM images (b) and (c) of a precipitate in steel specimen

Accelerating voltage: 200 kV
Specimen thickness: 0.1 μm

4. ANALYSIS OF A PRECIPITATE IN STEEL

Fig. 6 shows a TEM image of the cross-section of a precipitate. From the contrast of this image, we can assume that there are two precipitates in this specimen. Fig. 7 shows a STEM image (a) and elemental mapping images (b) through (g) of a precipitate of a cross-sectional TEM specimen. There was no Fe-signal detected from inside of the precipitate. Large particles of the precipitate are composed of Al, Si and O. Small particles are of Mn and S. From the structural and compositional analyses, we found that the small particles are crystals of MnS. Fig. 8 shows a crystal lattice image (a) and corresponding electron diffraction pattern (b) of MnS having a spacing of 0.18 nm.

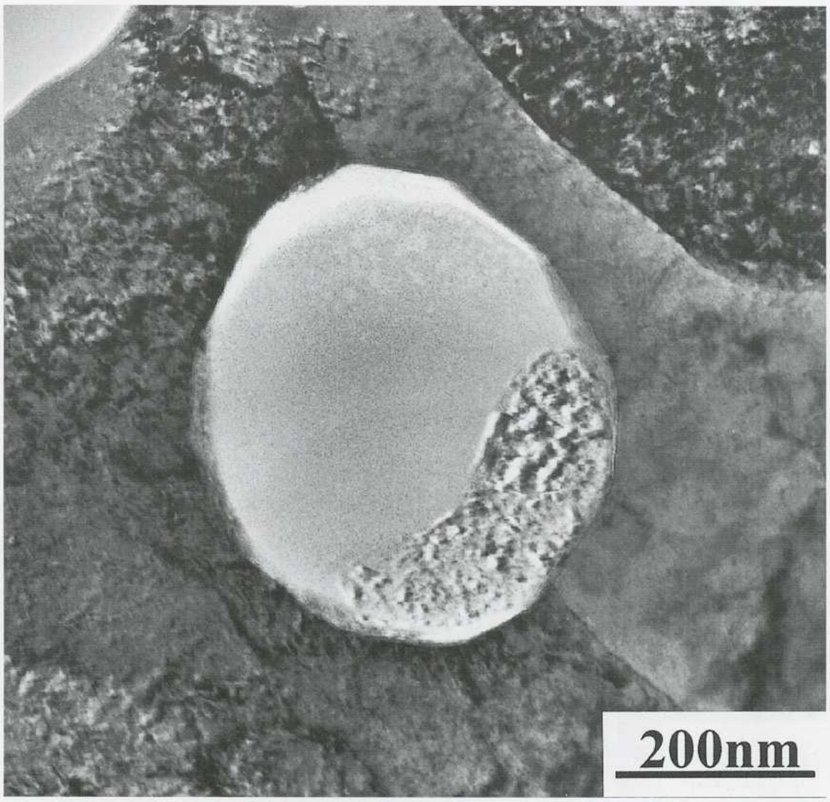

Fig. 6 A cross-sectional TEM image of a precipitate in steel specimen prepared by FIB milling

Accelerating voltage: 200 kV
Specimen thickness: 0.1 μm

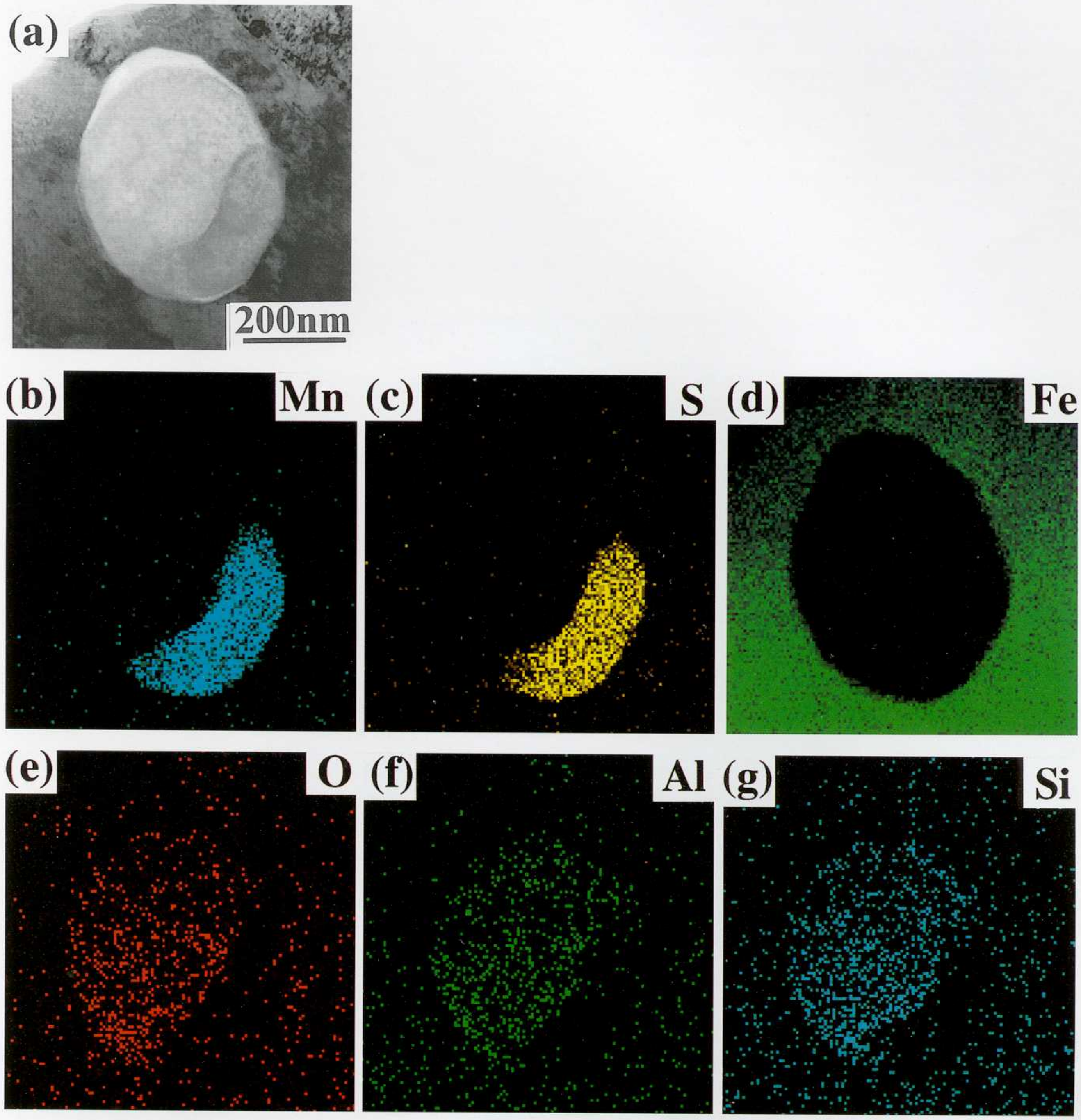

Fig. 7 X-ray mapping images of a precipitate in steel specimen

Accelerating voltage: 200 kV
Acquisition time: About 7 minutes

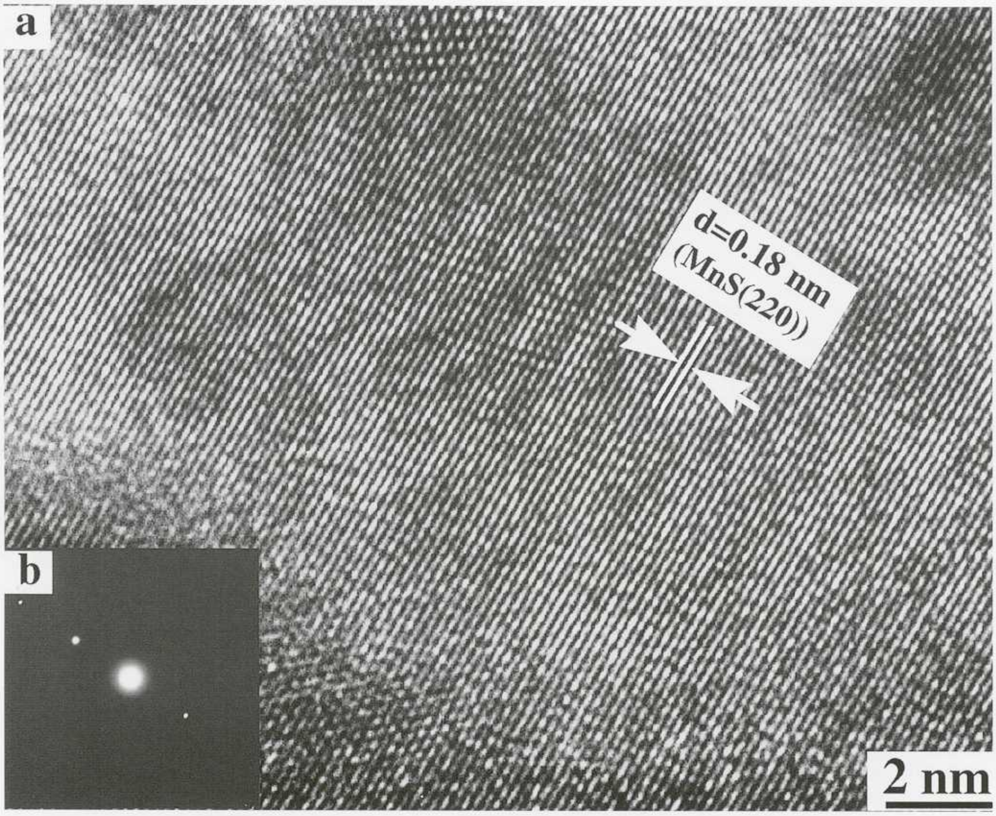

Fig. 8 A high resolution image (a) and a corresponding electron diffraction pattern (b) of a precipitate in steel specimen
Accelerating voltage: 200 kV

5. CLOSING REMARKS

We have introduced a cross-sectional TEM specimen preparation technique using an FIB/TEM system and an example of a structure analysis of a precipitate in steel. The FIB system allows preparation of uniform thin sections even from a specimen which is composed of a heavy material such as Fe for matrix and a light element of precipitates. The high voltage SEM/STEM imaging allows monitoring of the specimen milling conditions which result in thinning of a pin-pointed specific area of interest at a high accuracy. We trust that this technique will be found useful for fine structure analyses of a specific area or object of interest of compound materials or any other advanced materials.

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For further information, please contact your nearest sales representative.

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