HITACHI SCIENTIFIC INSTRUMENT

# TECHNICAL DATA

SEM

**SHEET NO.** 90

**SUBJECT:** A NEW EVALUATION TECHNIQUE FOR BULKY COMPOUND MATERIALS

**INSTRUMENT:** THE S-4700 FIELD EMISSION SEM
THE FB-2000A FOCUSED ION BEAM SYSTEM

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## 1. INTRODUCTION

Compound materials have been used in various fields of science and industry. For evaluation of these materials, SEMs and FIB systems have been playing major roles. The SEM and FIB system allow preparation of site-specific cross-sections of a sample quickly and permit examination of inner structures as well as compositions without difficulty.

The compound materials have been moving toward finer structures and multiple functions, making the required analysis areas smaller or submicron sizes for evaluation. It requires a better spatial resolution for EDX analysis coupled with SEMs. In addition, it is difficult to understand distribution of dispersion materials by a 2-D analysis using cleaved or FIB prepared samples. A 3-D analysis has been required to resolve these requirements.

We have used the S-4700 field emission SEM (Fig. 1) and the FB-2000A focused ion beam system (Fig. 2) to analyze thinned submicron areas and 3-D reconstruction of inner structures of compound materials. We report the details.

Fig. 1 The S-4700 field emission SEM

Fig. 2 The FB-2000A focused ion beam system

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## 2. HIGH RESOLUTION EDX ANALYSIS OF SITE-SPECIFIC THINNED AREAS

### 2.1 Analysis of sintered nickel/alumina

Compositions and dispersion conditions of matrix and dispersion materials are examined by BSE images or EDX analysis. Fig. 3(a) shows an SE image of a cleaved specimen of sintered nickel/alumina. Fig. 3(b) shows an EDX result of the same specimen on aluminum. It is difficult to record X-ray mapping images reflecting compositions due to topographic conditions of the specimen. Fig. 3(c) is an X-ray mapping image showing nickel in red color.

Fig. 4(a) shows a BSE image of the same sintered specimen. The specimen was milled by FIB to get analytical face. It allows EDX mapping without problems of topographic conditions of the specimen as shown in Fig. 4(b). It is difficult, however, to locate nickel at the same clarity as seen in Fig. 3(c). We have studied a good technique which allows EDX analysis of these specimens at a high spatial resolution. Fig. 5 shows a technique which we found useful. First, the specimen is milled by FIB to be flat. It is then box-milled at the back of the area of an analysis point. Then, the analysis point is thinned as 100nm, approximately. Fig. 6 shows EDX mapping images of the sintered nickel/alumina specimen prepared by the method of Fig.5. Distribution of nickel dispersion material of about 200 nm is clearly shown. This allows EDX analysis of submicron areas at a high spatial resolution.

Effects of specimen topography

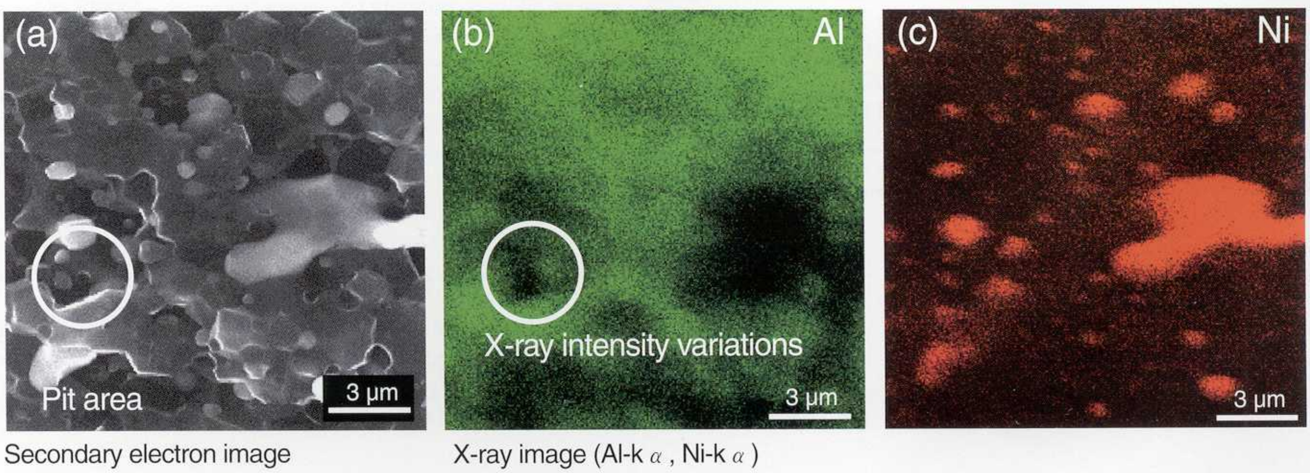



Fig. 3 EDX analysis of a cleaved specimen of nickel/alumina sintered material

Plane specimen: No effect of specimen topography → Mixed inner information

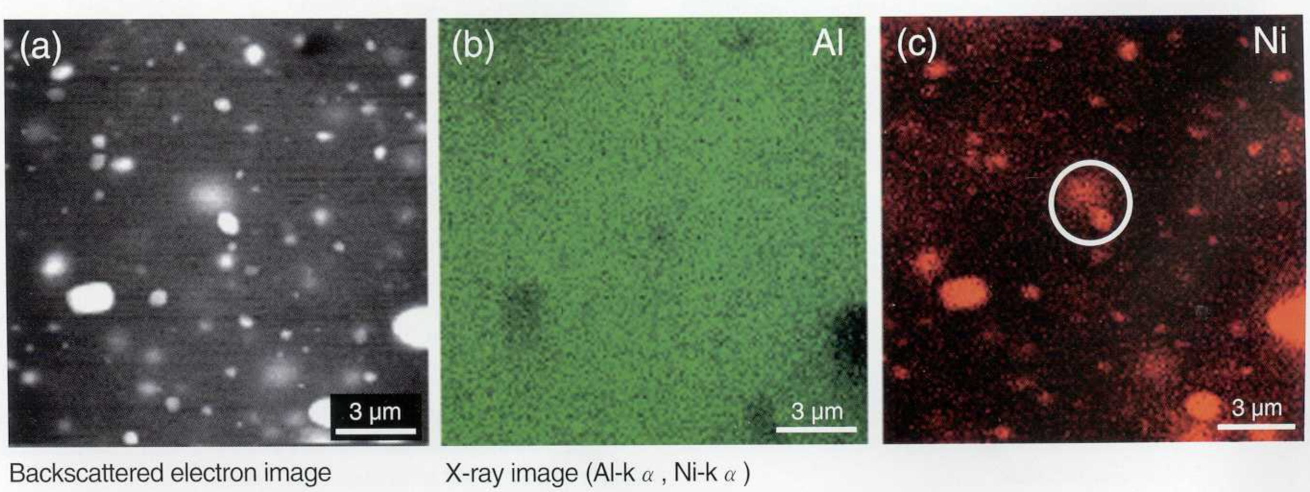



Fig. 4 EDX analysis of FIB milled specimen of nickel/alumina sintered material

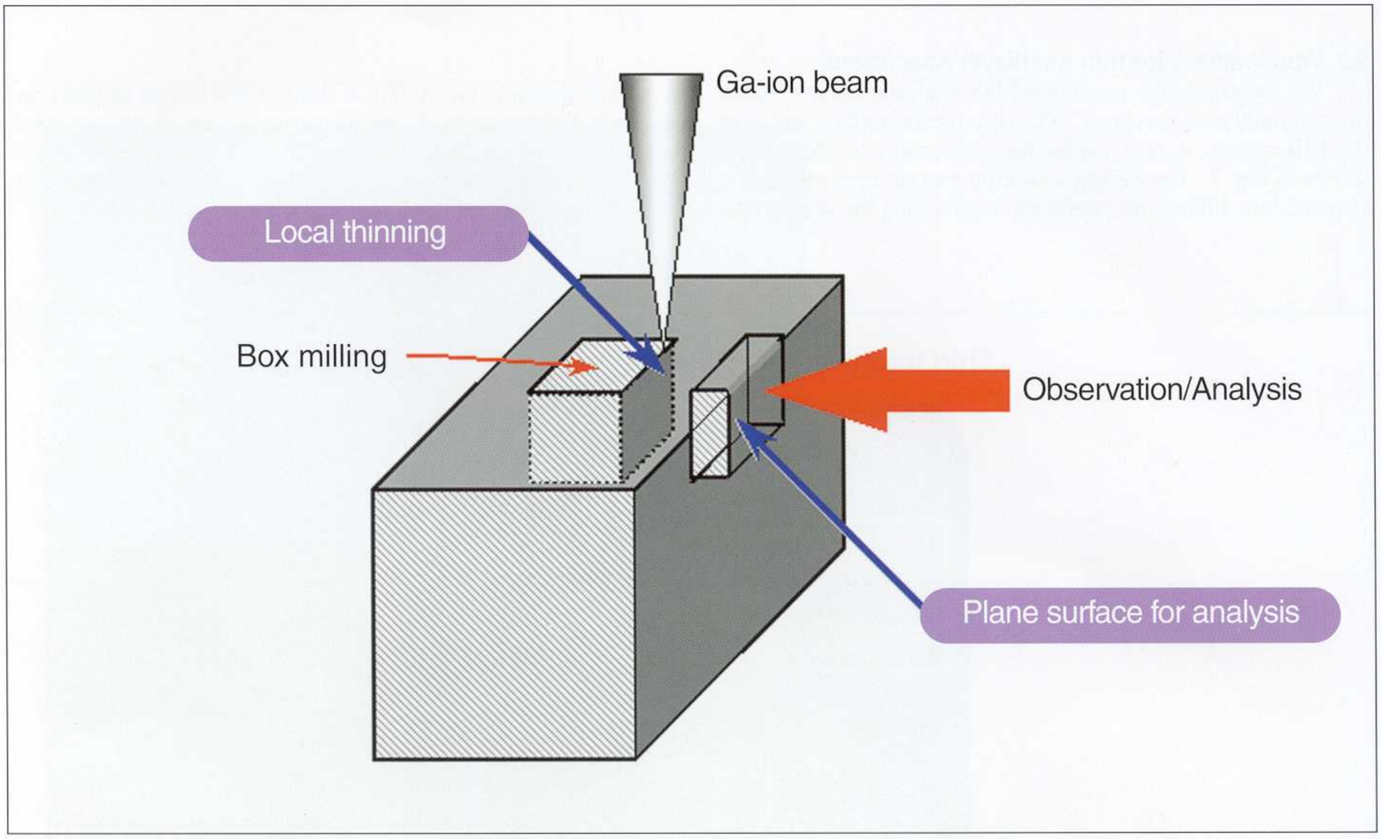



Fig. 5 Local thinning of a specimen using the FIB system

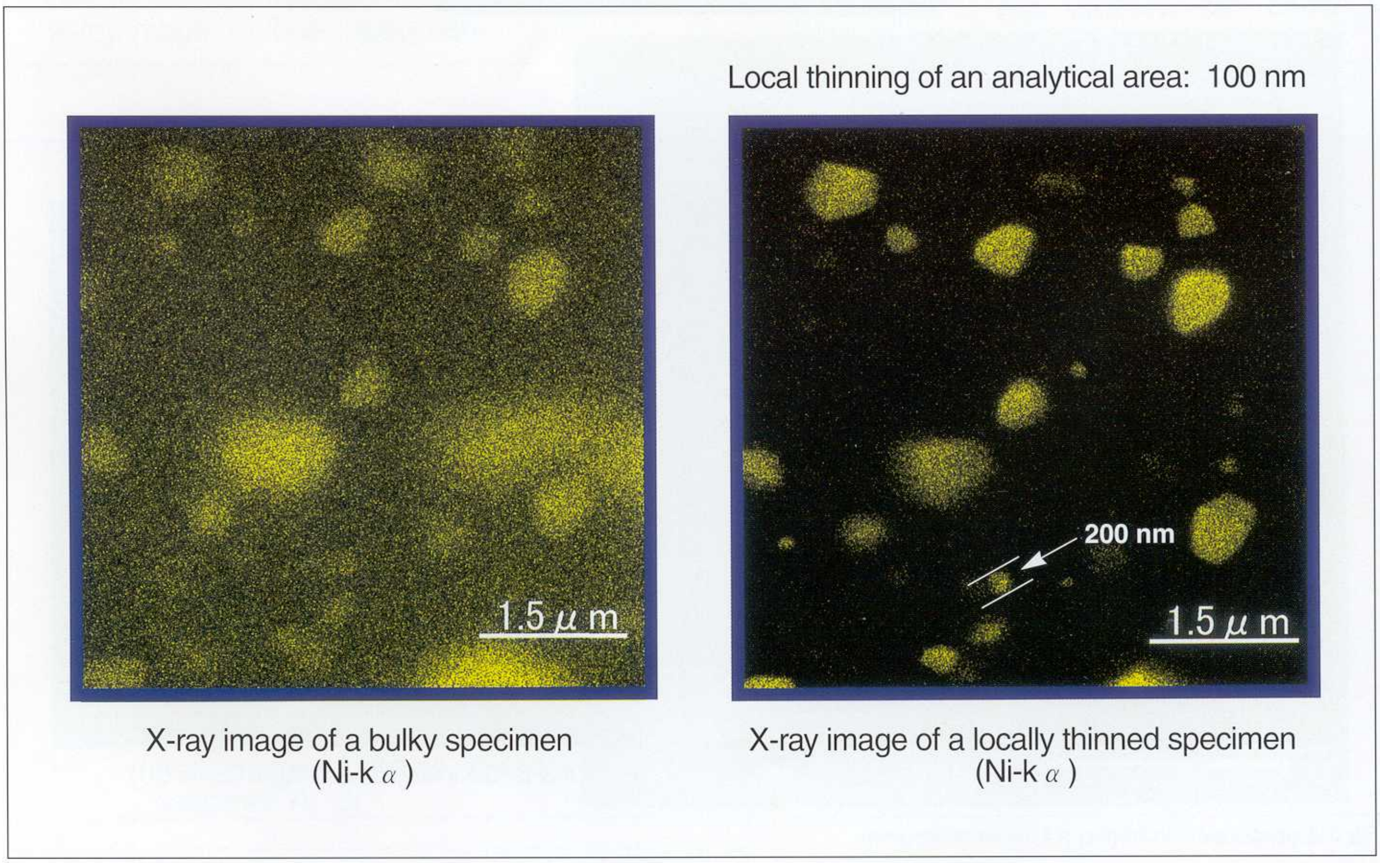



Fig. 6 A comparison of X-ray images using bulky and locally thinned specimens

## 2.2 Applications for thin multilayer specimens

We report on high resolution EDX analyses using a locally thinned multilayer specimen. The site-specific milling ability of the FIB system is very useful for semiconductor devices as shown in Fig. 7. For milling a specific area or areas of interest, step and box millings are useful for approaching the site-specific area as shown in Fig. 8. Fig. 9 shows EDX images of bulky and locally thinned areas of a multilayer specimen. A thinned specimen shows on the EDX mapping image with thinned film, aluminum and chromium layers are clearly defined in comparison with that of bulky specimen.

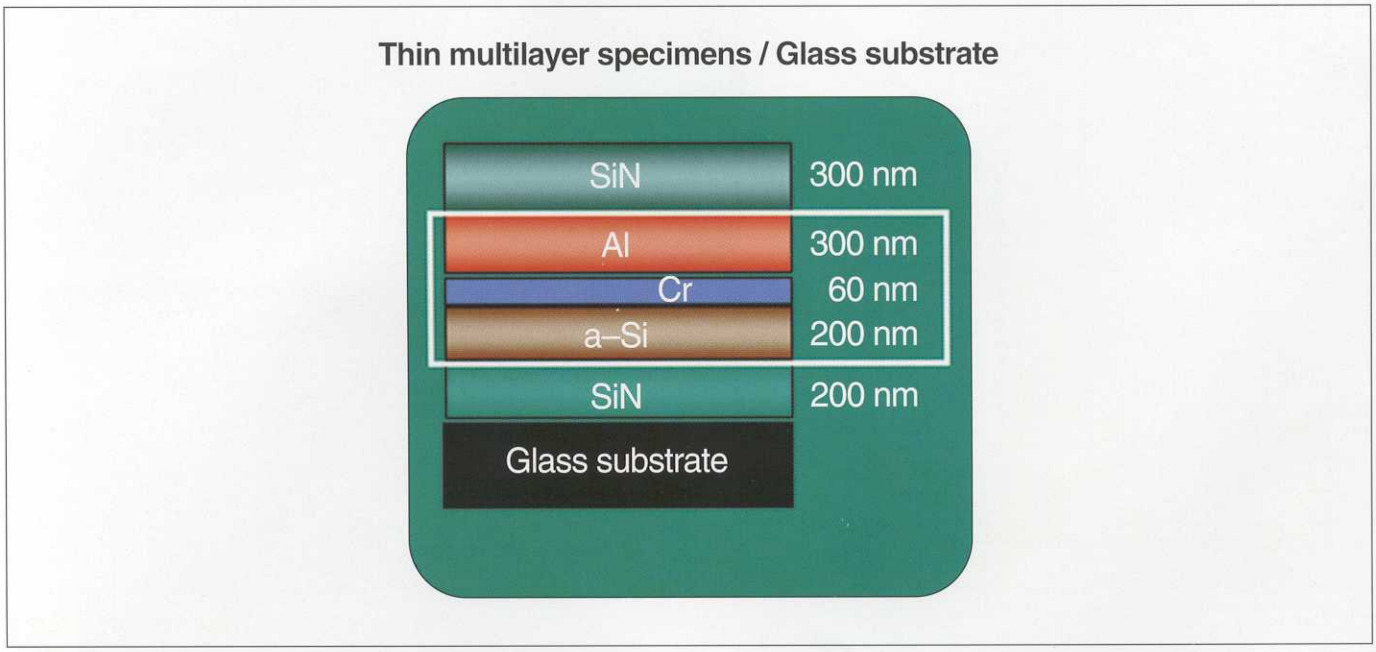



Fig. 7 An example of observation and analysis areas of a multilayer specimen

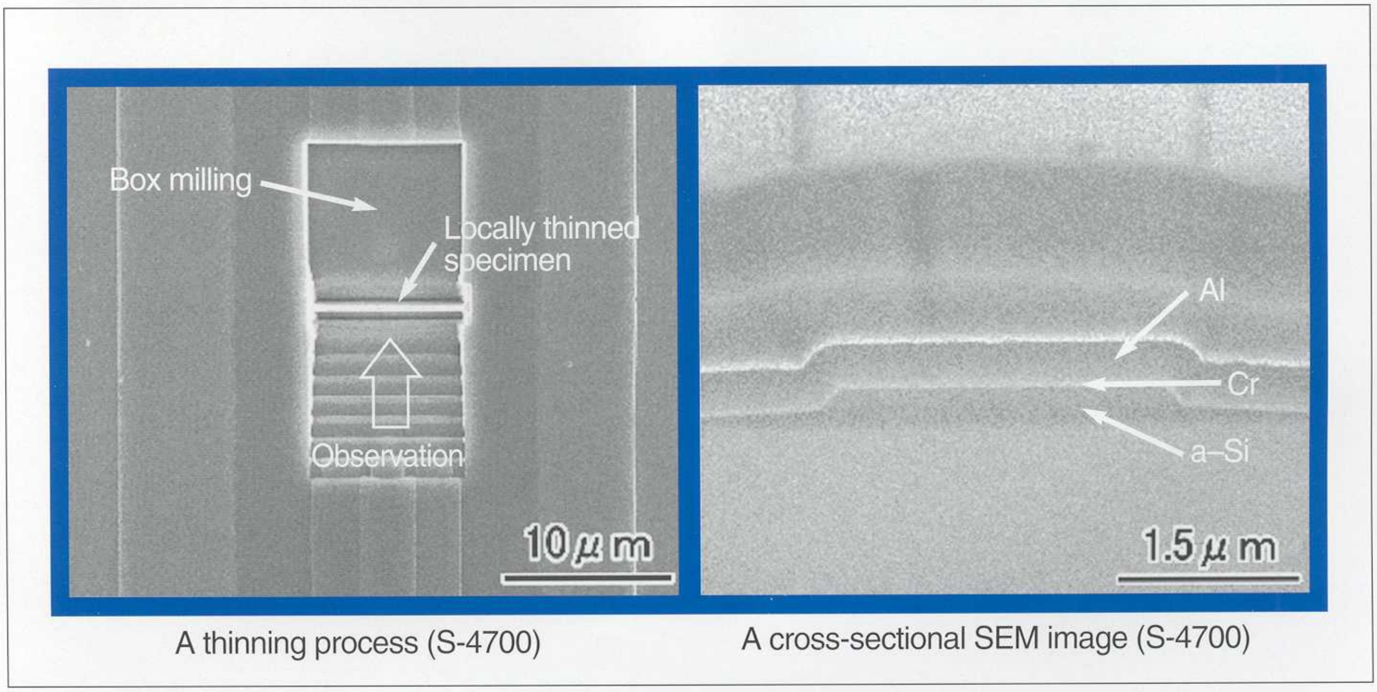
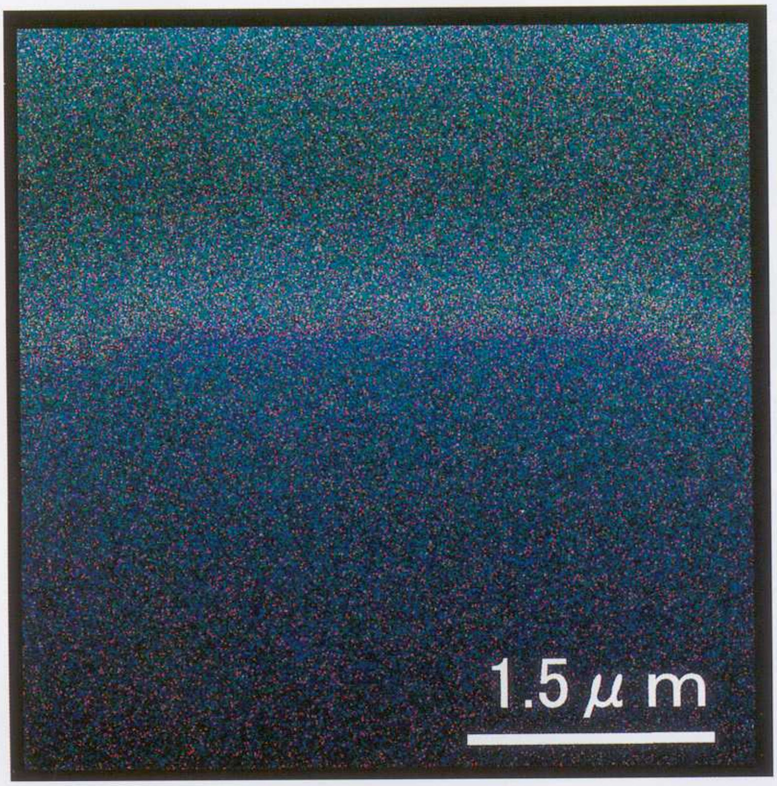
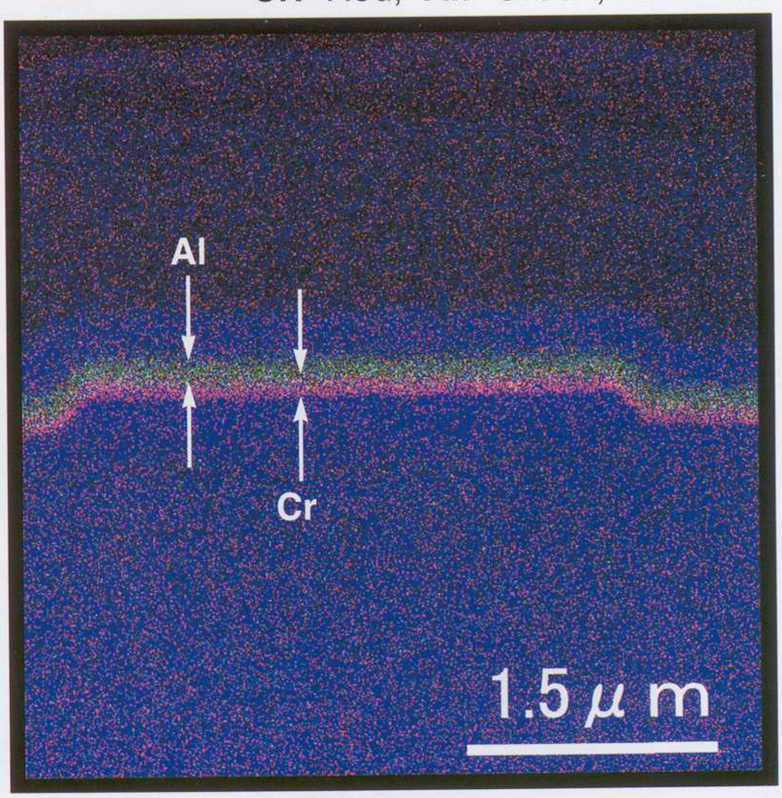



Fig. 8 A typical local area thinning of a multilayer specimen




X-ray image of a bulky specimen

**Cr:** Red, **Al:** Green, **Si:** Blue




X-ray image of a locally thinned specimen Al Cr

Fig. 9 A comparison of X-ray images using bulky and locally thinned specimens

## 3. 3-D RECONSTRUCTION OF DISPERSION MATERIALS IN A COMPOUND MATERIAL

For evaluation of dispersion conditions of matrix and dispersion materials, secondary electron and backscattered electron images have been used up to this time. Conventional 2-dimensional analyses of cleaved or FIB milled specimens, however, do not provide dispersion conditions of inner direction. Fig. 10 shows a BSE image of compound materials sintered nickel/alumina the same as shown in 2.1. These materials may be milled and observed using SEMs and FIBs repeatedly to get grain information of inner direction. This technique allows examination of dispersion conditions of the materials.

Fig. 11 illustrates this technique. After milling the specimen using an FIB, the specimen image is recorded in backscattered electron mode. After the image recording, the same specimen is milled by the FIB once again to a small thickness of about 0.1 µm. By repeating the operation, we recorded about 30 images which were processed in 3-D reconstruction software (Vox Blast; Image Analysis Facility) for obtaining a 3-D image of dispersion materials. Fig. 12 shows a 3-D image of nickel particles. Fig. 13 shows a cross-section of the 3-D image.

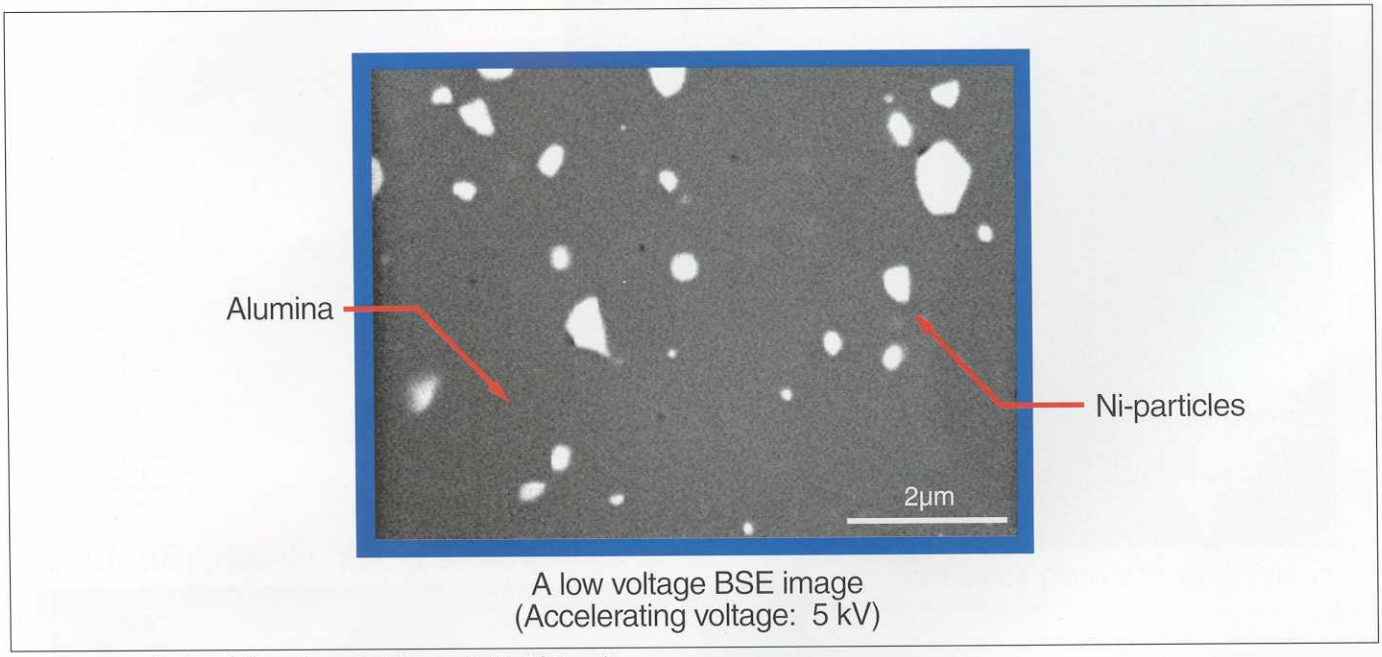



Fig. 10 2-D distribution of dispersion materials recorded in BSE mode

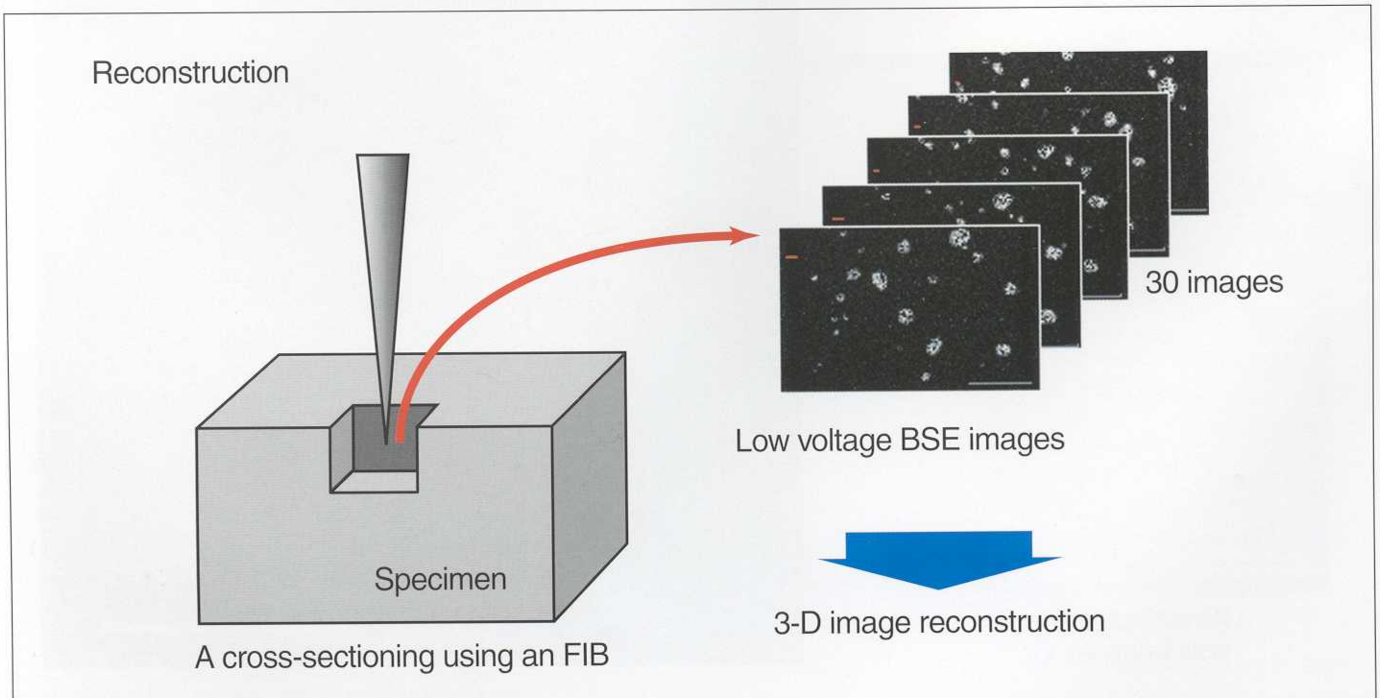



Fig. 11 A technique for 3-D reconstruction

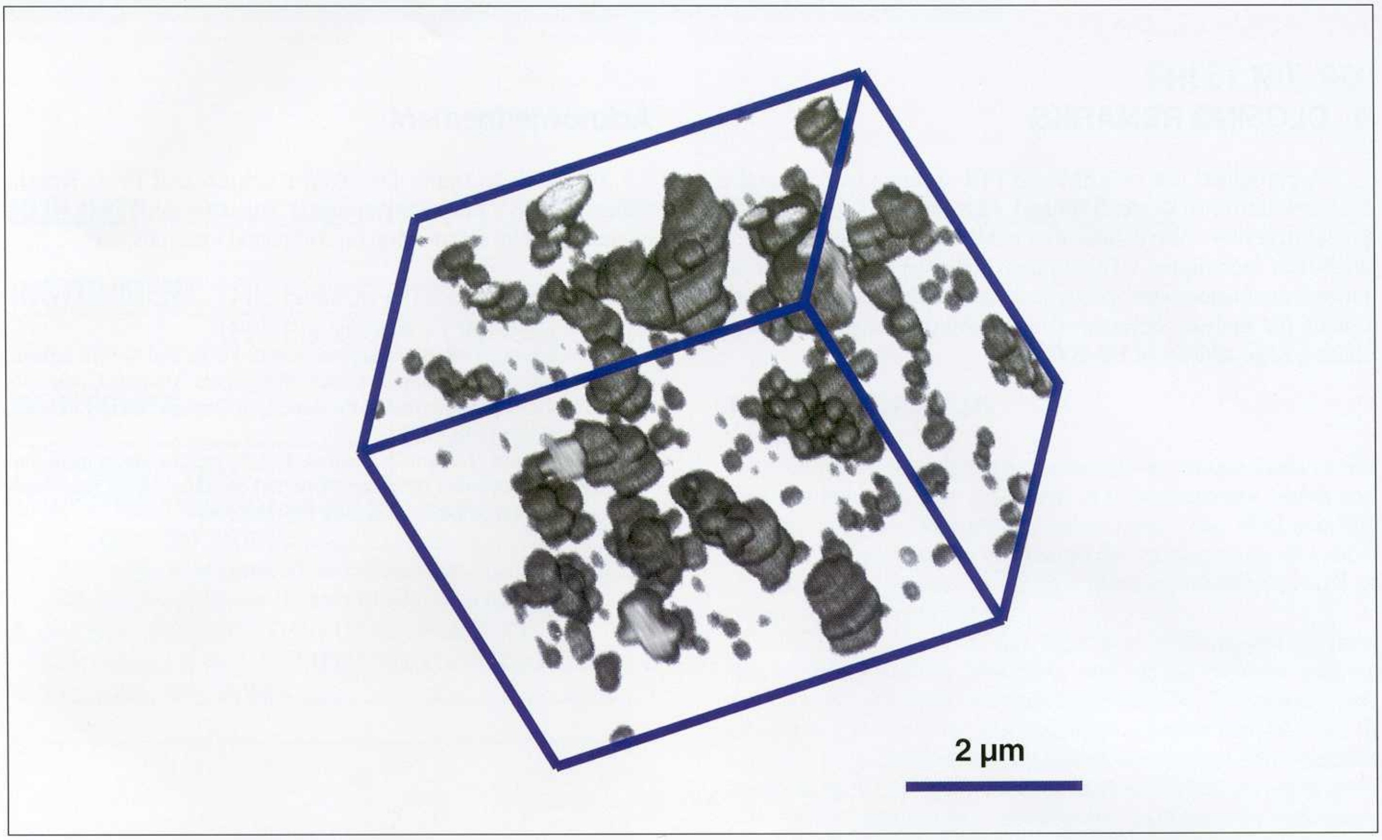



Fig. 12 3-D distribution of nickel/alumina particles

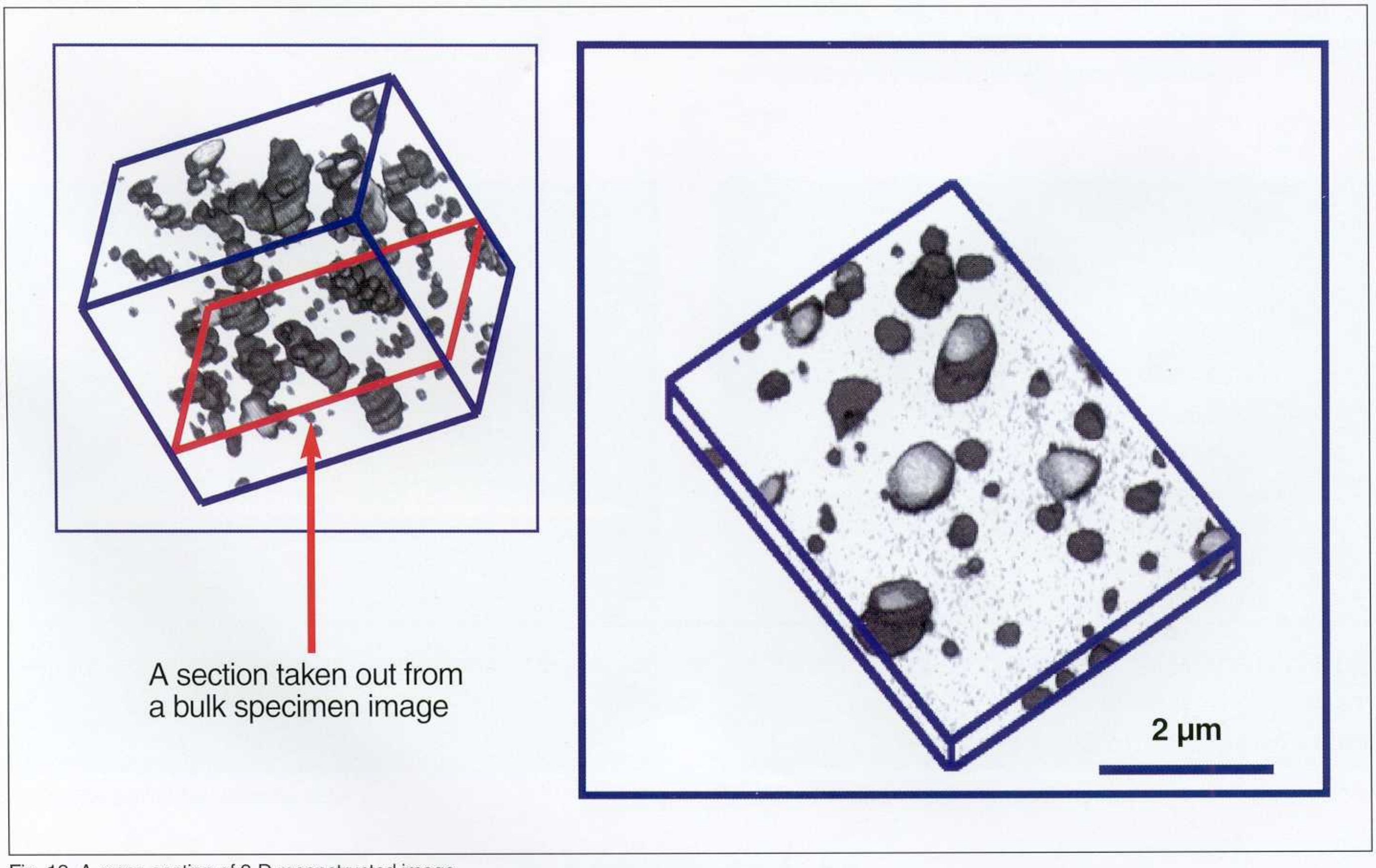



Fig. 13 A cross-section of 3-D reconstructed image

## 4. CLOSING REMARKS

A combined use of SEM and FIB systems has allowed a high resolution EDX analysis and 3-D reconstruction of compound materials. We believe that it will become one of the new analytical techniques. This system will contribute to various process evaluation with speedy analysis and high accuracy positioning for analysis, because of its capability of step milling and sharing stage address of FB-2000A.

## Acknowledgement

We wish to thank Dr. Tohru Sekino and Prof. Koichi Niihara, ISIR, Osaka University in Japan for providing specimens and useful information on compound materials.

### References

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