A Site Specific Characterization Technique and Its Application

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Abstract

A technique to characterize specific site of materials using a combination of a dedicated focused ion beam system(FIB), and Intermediate-voltage scanning transmission electron microscope(STEM) or transmission electron microscope(TEM) equipped with a scanning electron microscope(SEM) unit has been developed. The FIB system is used for preparation of electron transparent thin samples, while STEM or TEM is used for localization of a specific site to be milled in the FIB system. An FIB-STEM(TEM) compatible sample holder has been developed to facilitate thin sample preparation with high positional accuracy. Positional accuracy of 0.1㎛ or better can be achieved by the technique. In addition, an FIB micro-sampling technique has been developed to extract a small sample directly from a bulk sample in a FIB system. These newly developed techniques were applied for the analysis of specific failure in Si devices and also for characterization of a specific precipitate in a metal sample.

Introduction

Although FIB technique is one of the most reliable method to prepare the sample from the area of interest, required accuracy in localization of a specific site for TEM sample preparation of electronic devices, often beyond the capability of a standard FIB technique. As a solution of this problem, a technique to prepare for TEM sample preparation from the specific site with high positional accuracy has been developed[1),2)]. In this technique, a dedicated FIB system equipped with a side entry sample holder and an intermediate-voltage STEM(TEM) equipped with SEM unit are used. We have also developed an FIB micro-sampling technique to extract small sample directly from bulk sample. Although the

FIB micro-sampling technique resembles the FIB lift-out technique[3)-4)] in the respect that bulk sample remain intact, the method to lift out a micro-sample and the thickness of the lifted out sample are different. The details of these technique and some applications are discussed.

Instruments

An FIB-STEM system employed in the study is shown in Figure 1. Hitachi FB-2000A FIB system is used for the sample preparation. Ion source of the instrument is liquid Ga metal and accelerating voltage of the ion gun is 30kV. Localization of a site to be characterized and characterization of the sample after thinning are carried out with Hitachi HD-2000 STEM. Electron gun of the microscope is cold FE type and accelerating voltage is 200kV. An FIB-STEM compatible sample holder is used for sample preparation.

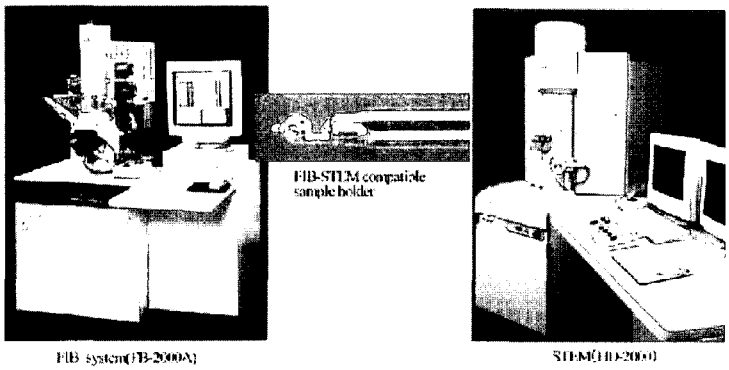

Figure1. The FIB-STEM system developed for site specific characterization

Method and experimental results

A method for site specific TEM sample preparation using an FIB-STEM system is shown in Figure 2. First, the sample is FIB milled to the thickness of 3 to 5μm remaining the site to be characterized in the center of the remained area(Fig. 2a). Then, the sample is transferred to a STEM to observe the STEM and SEM images from both sides(Fig. 2b-c). In this technique, both STEM and SEM images play important role in localization of a site to be prepared for the observation. A structure of the defect or a site to be characterized may by confirmed by STEM observation. The distance from the cross sectional surfaces to the failure or site can be determined by SEM observation. After the STEM and SEM observation, the sample is transferred back to the FIB system again for further milling.

Thus, FIB milling and STEM and SEM observation are repeatedly carried out until a thin sample with the thickness of about 0.1㎛ is obtained from the site to be characterized(Fig. 2d).

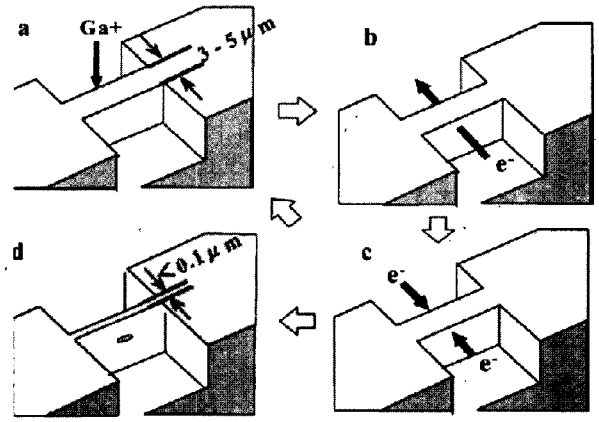

Figure 2. Procedure for site specific TEM specimen preparation using an FIB-TEM system

As an example to show the steps to prepare thin sample from a specific site, STEM and SEM images of an FIB milled steel sample are shown in Figure 3 and Figure 4. Thickness of the sample for Figure 3 and Figure 4 are 1㎛ and 0.5㎛ respectively. The precipitate to be characterized is indicated by arrows in the figures. The precipitate is observed on both cross sectional surfaces in 0.5㎛ thick sample (see SEM images shown in Fig. 4b and c).

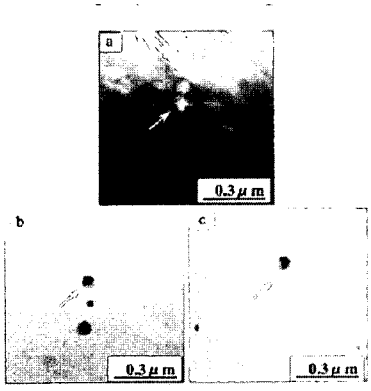

Figure3. STEM(a) and SEM images(b:front, c:back) of a steel(sample thickness : 1 μm.)

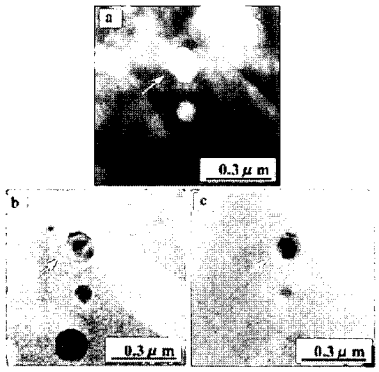

Figure4. STEM(a) and SEM images(b:front, c:back) of a steel(sample thickness : 0.5 μm.)

An FIB micro-sampling technique has been developed to extract a small sample from a bulk sample. A procedure for the FIB micro-sampling technique is shown in Figure 5. The area of interest is covered with a protection layer of a metal deposition. Then the sample is deep trenched by FIB milling to dig out a micro-sample from the area of interest. Next, the micro-sample is separated from the bulk sample and lifted out using a micromanipulator.

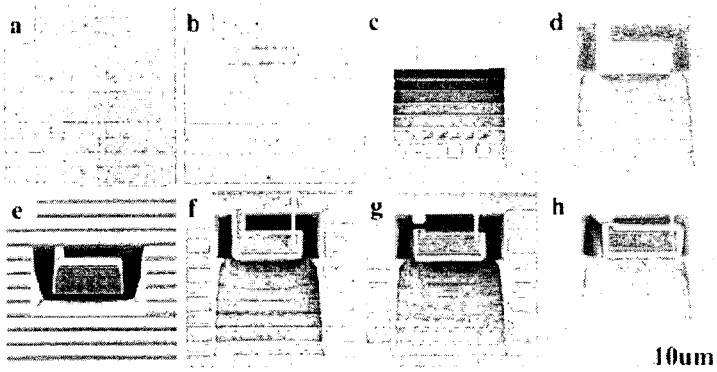

Figure5. A procedure for an FIB micro-sampling technique

Application of the FIB micro-sampling technique for three-dimensional structural characterization of the crystal lattice defects in a Si device is shown in Figure 6. Both cross-sectional and plan-view TEM samples were prepared from the site. The crystal lattice defect is characterized at near atomic resolution. The high resolution TEM revealed that the crystal lattice defect occurred along Si(111) plane.

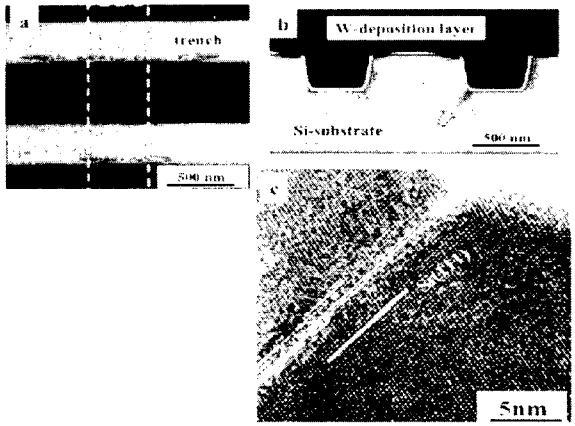

Figure6. Three-dimensional structural characterization of a failure in a Si device by plan-view TEM and cross-sectional TEM observation

References

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