Current State of Scanning Auger Microscopy Using FIB Sample Preparation

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The FIB is recently used for the sample preparation of TEM and SEM. Furthermore, when it would be used as the preparation for Auger analysis, Auger analysis of the cross section and the insulation material for a semiconductor device can be performed. This report is the application technology of FIB offered to SAM and the joint research with Dr. Tetsu Sekine was responsible for the development of SAM and FIB.

INTRODUCTION

An Ar ion gun is being installed in SAM (scanning Auger microscopy) to clean the surface and analyze the depth profiling. The ionization type of the Ar ion gun is an electron bombardment type. Since the brightness of it is about 0.01 μA/str・cm² and its source diameter is several 10 μm, the beam size of the focusing Ar ion beam is about 50 μm～100 μm on the sample surface. The Ar ion etching can not be used for micro areas etching smaller than several 10 μm. A duo-plasma ion gun with 1 μm beam diameter or FIB (focused ion beam) with several 10 nm beam diameter are used for the micro area etching. The FIB is recently used mainly for the cross sectional imaging on semiconductor device and the sample preparation of TEM (transmission electron microscopy) and SEM (scanning electron microscope). The reason why the FIB is used widely is because the high space resolution of the FIB has remarkably improved due to the technical development of the FIB and the cross section in the depth direction is made easily by the ion sputtering phenomenon. The metal of low melting point and low vapor pressure such as Ga (gallium) is usually used for a source of ion in the FIB. The mechanical structure of Ga ion source consists of a Ga reservoir tank and a tungsten tip. The ion of Ga is emitted from the top of the tungsten tip by heating the reservoir and supplying it with a strong electrostatic field. This Ga source would make the micro ion beam by its high brightness as several μA/str・cm² and small source size as several 10 nm. The FIB system is constructed with the FIB source, the ion beam scanning unit, and the function of the observation for secondary electron image. The FIB system is used to process the semiconductor materials and observe the shapes by the secondary electron image, which is specially known as SIM (scanning ion microscopy) image by exciting the ion beam. The processing precision of the FIB micro cutting depends on the etching conditions as ion beam diameter and ion beam current density. The etching rate of Cr is about 0.1 μm³/s with the acceleration voltage of 30 kV, the beam diameter of 0.2 μm and the beam current of 0.1 nA . As for the etching speed, the irradiation ion and target element have a dependence on each other, and the adsorption molecule on the target also have an influence on the etching rate. The FIB provides the selective gas assisted chemical etching or selective deposition at flooding the variety of gases on the sample. The mechanism of the reaction and adsorption between the primary ion beam and the gases are still not made fully clear. But, there are many experiment data and these processes are put to practical use. The analysis of this mechanism is a necessary subject to solve problems such as the character or reproducibility of the material.

Since the FIB comes to be the milling machine for the semiconductor manufactures, a practical FIB system comes into widely use for many applications[1, 2]. The main application is used for cutting cross section, observation of micro areas and analysis of the microstructure for the electrical routing on the semiconductor device. Other applications are used for sample preparation for TEM and SEM[3]. Recently, the high resolution observations of TEM and SEM are necessary for the FIB sample preparation. This FIB system would make a high capability developed as a special system. And the FIB system is produced

commercially to two types of instruments as a dedicated FIB and a combination of FIB and SEM. The many applications of the sample preparation are carried out by the dedicated FIB. The combination of FIB and SEM provides a better image resolution than that of SIM image and observation of fine structure. The main application of the combination system is the foreign particles defective analysis of the semiconductor material. In the combined system, the FIB is used to process a semiconductor material and the SEM is used observe the shape of the processed material and the particles.

Furthermore, it would be used as the cross section preparation device for Auger analysis. Recently, Auger spatial resolution has improved by using the FEG (field emission gun) as the electron gun, and observed a cross sectional structure in the IC (integrated circuit)[4]. Yamada and Sekine made the cross section of IC by using FIB, and observed Si oxidized gate structure with about 40 nm space resolution[5]. For Auger analysis, the cross section sample made by the FIB is necessary for surface cleaning with the Ar ion gun, because there are contaminations such as C and O by exposing in the atmosphere at the sample transfer from the FIB to SAM. The influences of charging from the insulation material inside the cross section layer would decrease by widely cutting the neighborhood area of the cross section.

When the FIB is employed to SAM's sample preparation, Auger analysis of the insulation material can be performed. Auger analysis of the insulator needs the experimental conditions with decreasing the influence of charging from the insulator. Several methods are proposed for Auger insulator analysis[6]. When the specimen is tilting up to 60 or 70 degrees, the secondary electron emission is increased, and the charging is decreased. At other methods, the charging is compensated by the irradiating low energy ion, and Auger analysis is performed on the thinning insulator by the primary electrons transmitting the thin material and the charging not stay on the surface. Furthermore, the FIB deposition function is used for Auger analysis of the insulator. The FIB deposition provides the conductive material is deposited on the micro area. There are reports that the Auger measurement of the insulator was possible by the conductive material being deposited around the insulator by the FIB, and the charging on the insulator being moved easily to the conductive plate[7, 8]. By using the FIB system for the Auger sample preparation, the Auger application for the insulator will increase more and more. In general, these specimen treatments are made by using the dedicated FIB system, and introduced to SAM. In special cases, when the FIB is combined with SAM, Auger analysis and depth analysis are performed without exposing the sample in the atmosphere after a specimen treatment by the FIB. It becomes characteristic that the analysis and treatment for the interesting sample can be done at the same vacuum condition in the case of the combination of FIB and SAM. There are only few reports from the combined FIB/SAM device, because there are still few machines, we want to expect a peculiar result from the combined FIB/SAM machine.

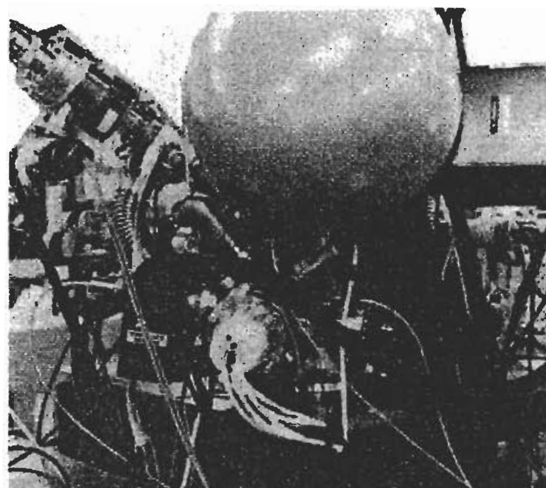

Fig.1. Combination of FIB and SAM.

This report is specifically limited to the use of application technology of FIB offered to SAM and the application with the joint research with Dr. Sekine.

EXPERIMENTAL

The combination of FIB and SAM

The instrument that FIB (FEI: 2 Lens Ion Column) was installed into the extra port of SAM (JEOL: JAMP-7800F) is shown in Fig. 1[9]. The experimental geometry of the sample and the ion beam irradiation for this device are shown in Fig. 2. The angle with HSA (hemi spherical analyzer) and EOS (electron optical system) is 55 degrees. The constructing arrangement of the FIB was chosen as that the FIB would be irradiated on the surface normal to the specimen and the top of the FIB would not be touched on the specimen by moving stage as sample tilting. The FIB was installed at the 100 degrees angle to EOS under HSA, and 50 mm WD (working distance) to specimen. By this arrangement, if the stage is moved in any direction, there is never a stage interference with the FIB. The FIB could get the resolution of 20 nm at 25 keV as the beam

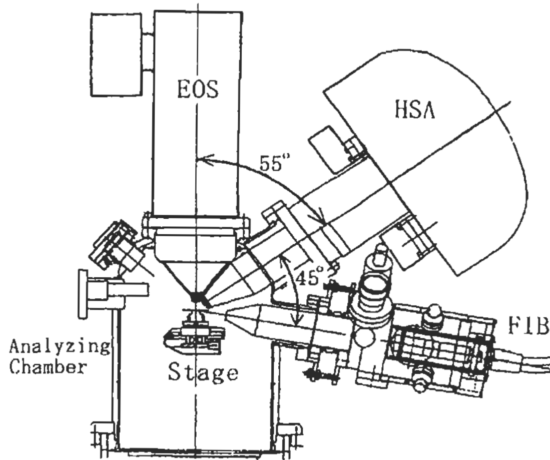

Fig.2. Schematic diagram of combination FIB/SAM.

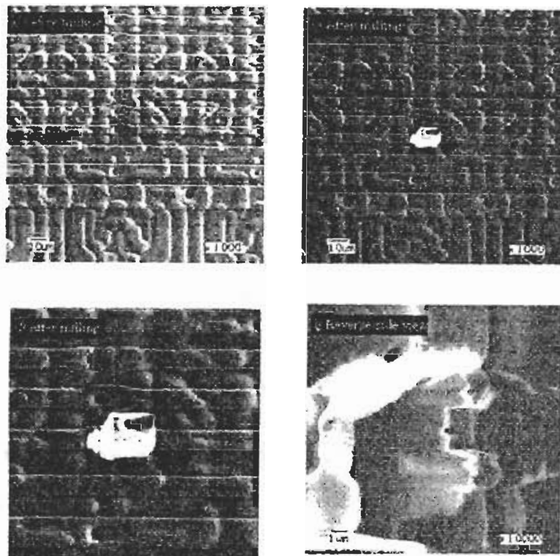

Fig.3. SEM images of IC cross section by FIB, a) Before milling, b), c) After milling, X1000, X3000 Magnification respectively, d) reverse side view, X10000 Magnification.

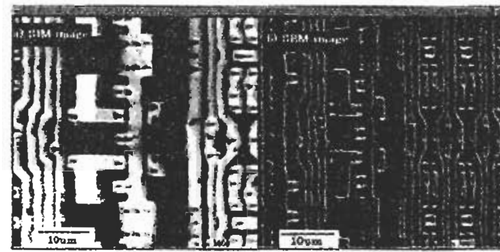

Fig.4. SIM and SEM images were measured simultaneously at specimen. tilted 45 degrees. At this condition, the ion and electron beam irradiated to specimen at 55 and 45 degrees restrictively.

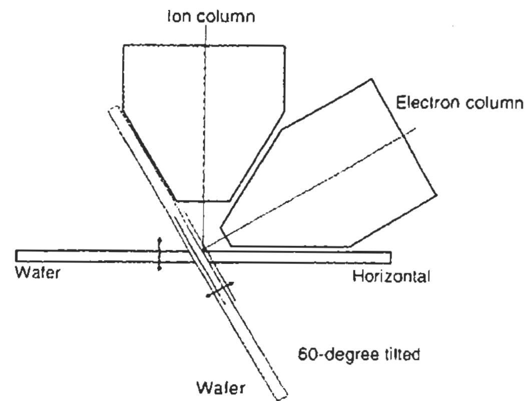

Fig.5. Dual beam FIB system.

diameter in this experiment. A milling process of the cross section is made as following. First, the IC sample was mounted on the sample holder with a 45 degrees slope. The FIB becomes the condition of normal incidence on the specimen surface when this sample holder is installed in the stage and is tilted at 55 degrees. The cross section was made by FIB on this condition. After ion milling, the sample holder is returned by the negative 55 degrees tilting. The cross section is turned to the direction of the Ar ion beam by the sample stage rotation, and the cross section was finished by the Ar ion etching. The section was turned in the direction of HSA after the Ar etching, and the analysis can be performed. The test data of IC pattern processed by FIB are shown in Fig. 3. SEM image of the IC pattern can be observed by SAM, and the cross section was milling by FIB on the same position of the SEM observation, and the analysis by SAM becomes possible. For the combined machine of FIB/SAM or FIB/SEM, the SEM image by electron excitation and the SIM image by ion excitation are observed as shown in Fig. 4. When the contrast of these images are compared, the contrast of each image is turned over. We are analyzing why this contrast is different between SEM image and SIM image, and it has been known that the contrast between electron and ion excitation is shown a reversed contrast for the atomic number dependence[10, 11].

Dual beam FIB

In the manufacturing process of the semiconductor device, the defect can occur inside the multiple layer structure, which may not be able to be identified by the observation of SEM image. In such a case, a dual beam FIB combined with FIB and SEM is very effective for defect analysis. The exclusive machine that installs FIB into SEM is used with the semiconductor application that does the

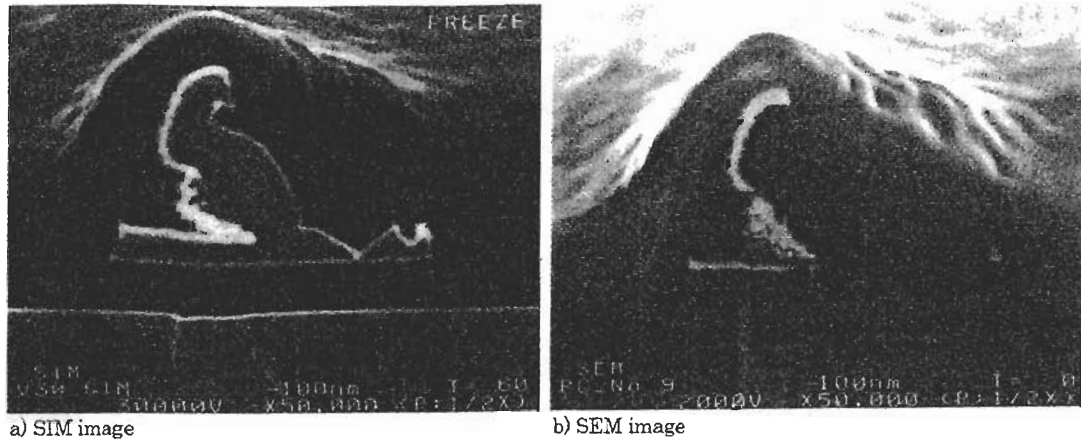

Fig.6. Observation of the defect by the dual beam FIB. a) SIM image, b) SEM image of the cross section

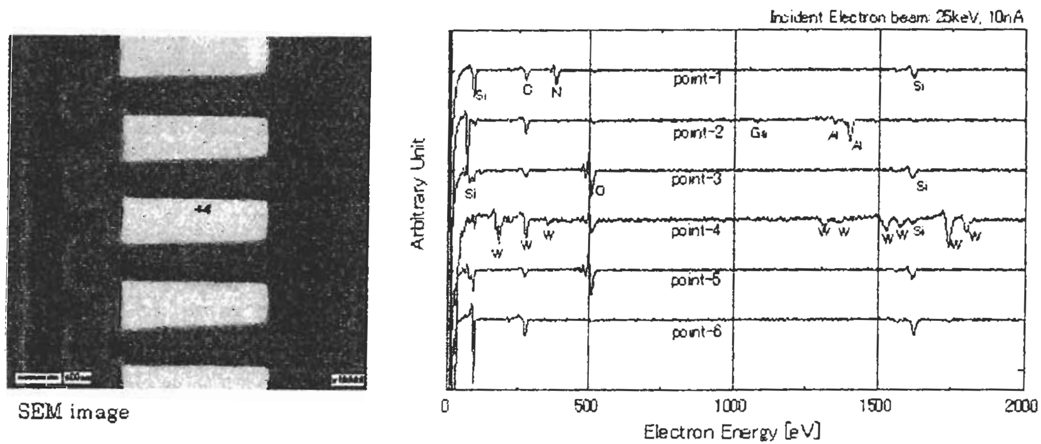

Fig.7. SEM image and Auger spectra at each point on SEM of the semiconductor specimen made by the thin film method.

etching of micro area, cross section preparation and observation at the same time. When EDS (energy dispersion X-ray spectroscopy) is installed in this system, an elemental analysis may be performed. A group of Sekine developed the dual beam FIB system shown in Fig. 5 for defect analysis[12, 13]. The defect was milled to the cross section and observed the fine structure with SIM and SEM as shown in Fig. 6. The particle causing the defect can be found out and analyzed by this dual beam FIB. This instrument is used as an analytical tool for the exclusive use of the 200 mm or 300 mm wafer. The instrument such as a combined SEM/FIB is expected to observe defect review and analysis of defect on the wafer. The main reason for the combination of SEM and FIB is that the resolution of SEM image is better than that of SIM image, and because the FIB system used the electro static lens for the focusing lens system. If the new FIB system with the aberration corrected lens system is developed in the future, the combined system with FIB and SEM will change to the stand alone FIB system[14].

APPLICATION

A cross section analysis by SAM

The application of the sample treatment by the dedicated FIB is shown here. Yamada and Sekine had already observed Si oxidized gate structure in the IC with about 40 nm space resolution[5]. At this experiment, there was the charging phenomenon. By widely etching, the neighborhood area of the section was cutting at about 4 μm x 8 μm, the influences of charging from the insulator material inside the cross section layer would decrease, but it is very difficult the influence from insulator would be removed completely.

Recently, a method of thin film pick up was proposed for making a section sample of the TEM application[15]. The thin film of section structure is cut by the FIB, the cut sample is picked out from IC by the special manipulator, and is put on the sample holder. Tsutsumi reported that a

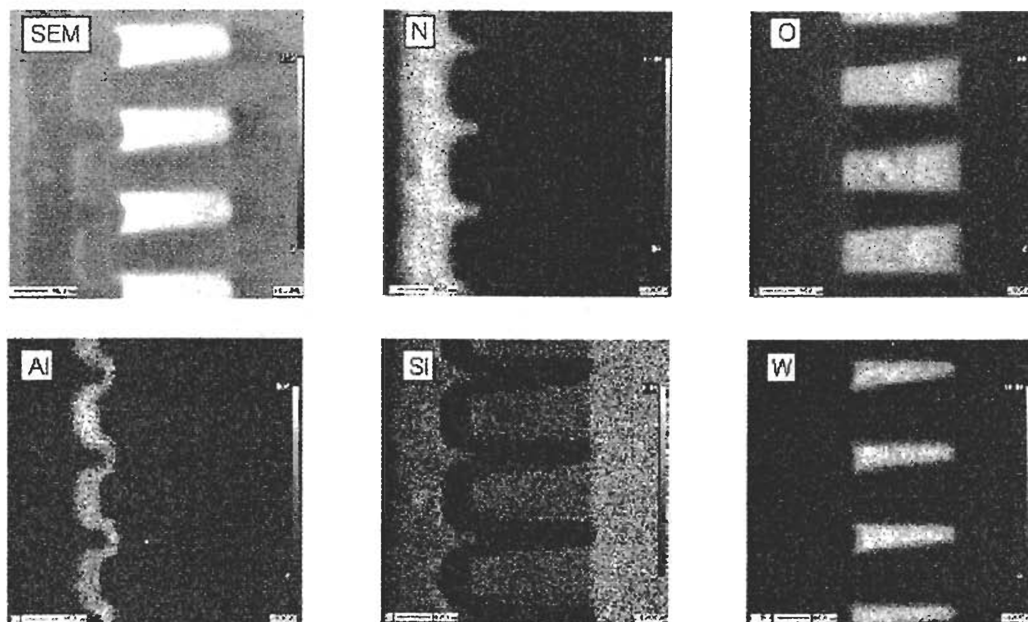

Fig. 8. SEM and N, O, Al, Si and W Auger images of the semiconductor specimen made by the thin film method.

thin film insulating sample was made from the IC by the FIB, and that the AES analysis would be possible with the reduction of influences from the insulator[16, 17]. They made the thin film by cutting it to the size of 10 μm x10 μm and thickness of 0.1 μm from the section part of the IC test pattern. The thin film sample was picked out by the static electricity power of the rounded glass probe and made as the sample, which was put on the conductive material graphite, and analyzed by SAM. By making the thin film, Auger measurement of insulator can be performed by decreasing the influence of the insulation such as SiO_2 and Si_3N_4. Auger spectrums for each point on the thin film of the IC test pattern were shown in Fig. 7. The observed SEM image and N, O, Al, Si and W Auger images are shown in Fig. 8. This thin film method shows that the influences of the insulation inside the cross section were reduced, and Auger image resolution was improved to about 10 nm.

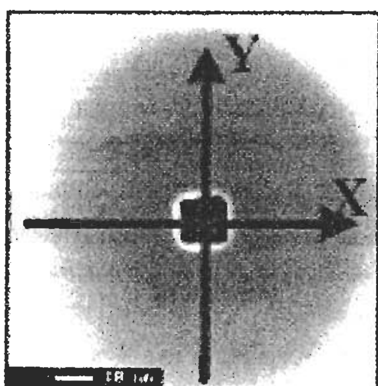

Fig.9. SEM image of crater on Si by 30 keV and 1 nA Ga ion beam during 20 minutes irradiation.

Analysis of Ga contamination by FIB

When the sample was irradiated with the Ga through the milling process, the Ga contamination on the sample must be examined. Sakata and Sekine had analyzed the Ga contamination test on a Si wafer irradiated by the dual beam FIB[18, 19]. In this experiment, the Ga contamination was analyzed by several techniques of TXRF (total reflection X-ray fluorescence), TRXPS (total reflection X-ray photo electron spectroscopy) and ICP-MS (inductively coupled plasma mass spectroscopy). The results of TXRF and TRXPS indicated that the deposited Ga species distributed within the area of 10 to 20 mmϕ, and also that Ga contamination level around the irradiated spot was 2 - 10 x 10^{10} atoms/cm^2 for Ga dosages of 30 kV accelerating voltages and 1 nA beam currents during 113 minutes. The results of the SAM analysis are shown in Fig. 9 and Fig. 10. After the Ga ion with accelerating voltage of 30kV and beam current of 1 nA was irradiated in a Si wafer during 20 minutes, a crater of 10x10 μm^2 was made, and the

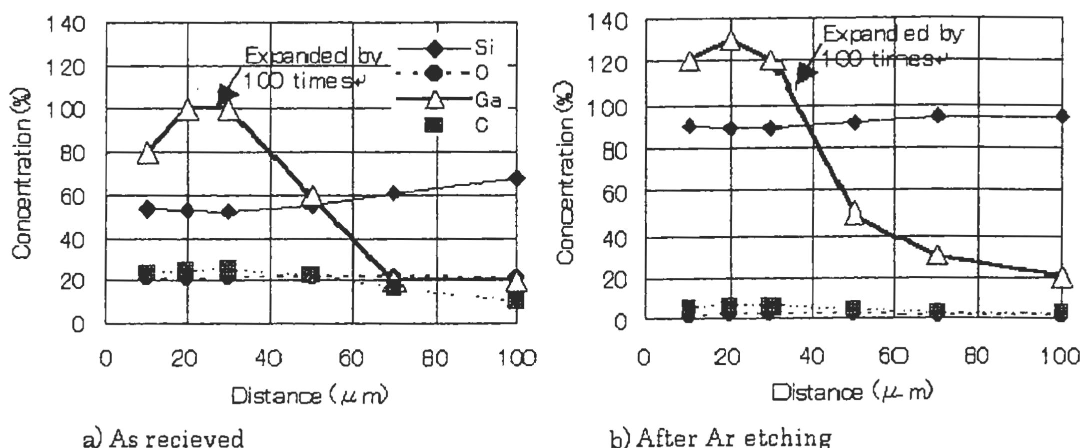

Fig.10. Ga distribution measured by AES. The vertical scale of Ga is expanded by 100 times. a) as received, b) after Ar etching.

area of 90 μm from the end of the crater was measured by SAM. In Fig. 10, the vertical scale of Ga is expanded by 100 times. The Ga contamination is 4.5×10^7 atoms/cm^2 within the area of 200 μmϕ. Taking into account the results for the Ga lateral distribution by the AES and TRXPS, we supposed the contaminated area was limited to be 20 mm in diameter. Auger analysis would have considered this influence from this result as well.

SUMMARY

It was shown that the Auger analysis was performed on the insulator material by the FIB sample preparation. When the insulator was made to the thinning film, the following process of the FIB cutting, picking up by the glass probe, and contacting on the conductive plate, Auger analysis is progressed for the thinning film without charging. For the specimen made by the FIB preparation, the Ga contamination was remained. The highest Ga concentration was 4.5×10^7 atoms/cm^2 within the area of 200 μmϕ, and the maximum Ga contamination area was 20 mmϕ. Auger analysis would have carefully considered this contamination. The dedicated FIB is used for the sample preparation with effective and high working machine efficiency. But, when FIB and SAM are included at the same time, the combination of FIB and SAM makes use for the characteristics of milling and observation inside the same vacuum, it is not only a sample preparation for the cross section observation, but also it is favorable for the observation of deposition treatment process by FIB. As reported in this, the sample preparation processed by FIB is expected to extend the application range of SAM greatly.

ACKNOWLEDGEMENT

The author would like to acknowledge Dr. Tetsu Sekine, passed away on 31 January 2002. Most of these reports are the works from the cooperation with Sekine and JEOL Auger group. There were many Sekine's idea which were always newly proposed for the development of Auger microscope and SEM/FIB, and the application of its. His main works were the development of a hemispherical electron analyzer[20 - 22], Auger chemical analysis[23 - 25] and Auger polymer analysis[26, 27]. Sekine had always worked with his best and challengeable sprit. We had the useful memory the discussions on the development of SAM with Sekine. Sekine moved from Auger group to FIB group by the company requirement and developed the dual beam FIB. The many ideas proposed by Sekine are used in the development of these instruments, and are the big future of these instruments. These ideas of instruments will live and succeed to future.

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