Development of Natural SEM and some applications

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ABSTRACT

Recently, study of insulators and water containig specimens has increased with wide spread use of SEMs. Authors developed Natural SEM operating at a low vacuum (1~270Pa). A stable low vacuum condition which is not influenced by external factors such as outgassing from the specimen and a simple changeover function to and from high vacuum mode were realized by a real time low vacuum control function and indpendent low vacuum evacuation system.

Results confirmed that a charging phenomenon can be reduced through applications for insulators, biological and food specimens. Furthermore, X-ray generation area can be reduced to 10 μm or less at 100Pa. It is expected that a capability of non-destrucive observation and analysis will contribute to expanding application fields of SEMs.

KEYWORDS

Low vacuum specimen chamber, Backscattered electron, Digital SEM, vapor pressure, Real time scan convesion, Realtime vacuum control.

INTRODUCTION

Scanning Electron Microscopes (SEM) have been used as indispensable observation tools for microscopic surface morphology in various fields of science such as electronics, materials science and biology since they were commercialized more than twenty years ago. Recently, observation of specimens containig water, oil and the like without preparation has increased. Authors have developed "Natural SEM" providing a low vacuum specimen chamber (1~270Pa) in order to allow microscopy of such specimens.

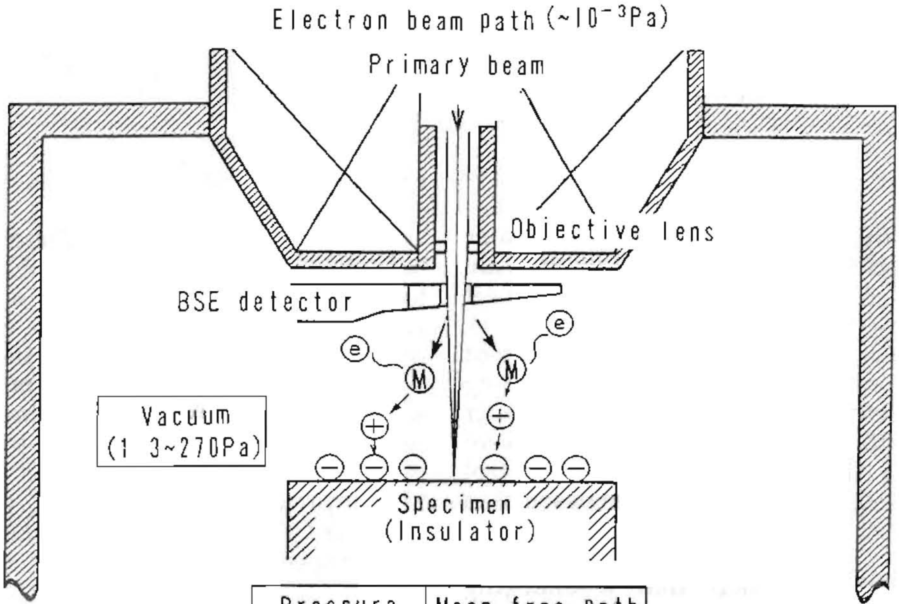
The features of Natural SEM are (1) to reduce a charging phenomenon, (2) to minimize shrinkage or deformation of hydrated specimens like biological tissues and (3) to allow microscopy of specimen without preparation. In the low vacuum condition, primary and backscattered electrons ionize gas molecules by mutual collisions as shown in Fig. 1. Positive ions generated here may reduce a charging since these ions neutralize negative charges on the specimen surface [1] [2]. Fluid in the specimen vaporizes rapidly in the conventional SEM specimen chamber due to high vapor pressure. The low vacuum condition protects the specimen becuse it regulates vaporization of fluid and temperature-rise on the specimen surface [3]. Authors have developed 3 types of Natural SEMs, S-2250N, S-2360N, S-2460N and will report here instrumentaion and some applications. Table 1 shows comparison of each instrument.

DEVELOPMENT OF NATURAL SEM

1. Low vacuum system

Authors have designed the system to allow a stable observation and an easy operation without influence of outgassing from the specimen. The system also allows a high vacuum mode for conventional scanning microscopy.

1) Vacuum conditions in the specimen chamber can be controlled 1~270 Pa.
2) An automatic adjustment is provided to allow stable vacuum condition.
3) The operation modes include high and low vacuum modes with a touch of a button.
4) An independent evacuating system permits use of one oil rotary pump exclusive for low vacuum evacuation.



Pressure	Mean free path
10^{-3}Pa	100m
13Pa	10 mm
270Pa	0 5mm

Fig. 1 Reduction of charging phenomenon in Low Vacuum conditon

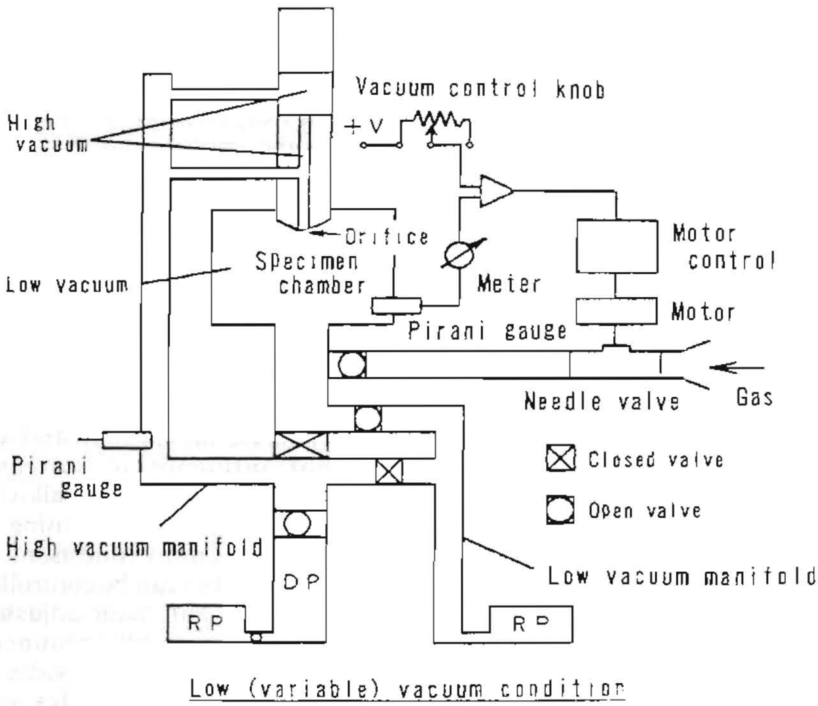


Fig. 2 Vacuum system of Natural SEM

Table 1 Specificational comparison of Natural SEMs

	S-2250N	S-2360N	S-2460N
Resolution (High Vac.)	4.5nm	4.0nm	—
Resolution (Low Vac.)	8.0nm	6.0nm	—
Magnification Range	20~200k	—	—
Accelerating Voltage	0.5~25kV	—	—
Display Monitor	9 inch CRT	—	12 inch CRT x 2
Photo Recorder	Common Use With Monitor	Exclusive 9 inch CRT	—
Image Memory	Option	—	512 (Buffer) 1024 x 2
Vacuum Range	1~270Pa	—	—
Vacuum Indication	Analogue Meter	—	Digital Display
Vacuum Control	Knob Operation	—	Menu Operation

The vacuum system of Natural SEM is shown in Fig. 2. The column and specimen chamber are separated by a small aperture (orifice) to facilitate differential pumping. In the high vacuum mode, the column and specimen chamber are evacuated with an oil diffusion pump assisted by an independent oil rotary pump. Clean vacuum is available without back stream of oil mist. A changeover operation for high/low vacuum modes is performed with ease by electromagnetic valve design. A fine vacuum adjustment in the low vacuum mode is carried out by a motor driven needle valve. In practical opration, a desired vacuum condition is easily set with a simple adjustment of the control knob (Fig. 3 a) (S2250N, S-2360N) or a menu selection on the screen (Fig. 3 b) (S-2460N). In addition, the vacuum in specimen chamber is monitored by a pirani guage at all times and it is kept constant by a real time vacuum feedback system even with heavy outgassing specimen.

2. Backscattered electron detector

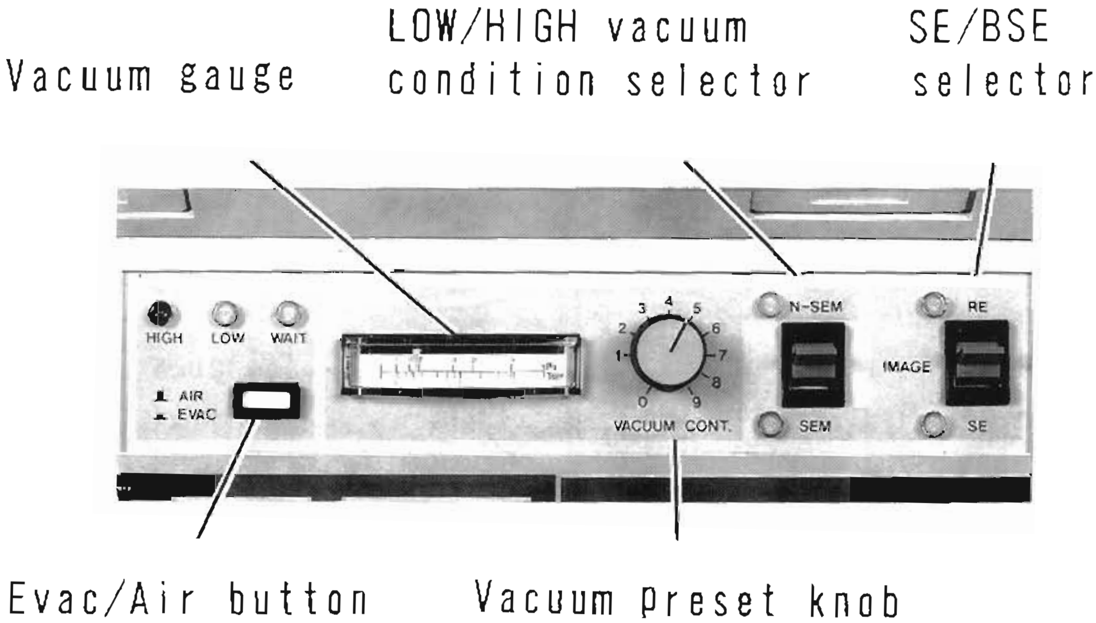
Secondary electrons which are ordinarily used for SEMs are unable to reach detector in the low vacuum conditions due to loss of energy by collisons with gas molecules. Natural SEM forms images by detecting backscattered electrons (BSE) which have almost the same energy as primary electrons. A scintillator type detector (Robinson Detector) or a semiconductor solid-state detector shown in Fig. 4 is commonly utilized for detection of BSEs. The former has advantages for high sensitivity and faster response time. The latter has a capability of displaying atomic contrast image or topographic image by manipulating signals. The Robinson Detector has been employed for Natural SEM. It enables easy focusing, brightness and contrast controls. Both of these detectors can be accomodated simultaneously when necessary.

3. Image display system

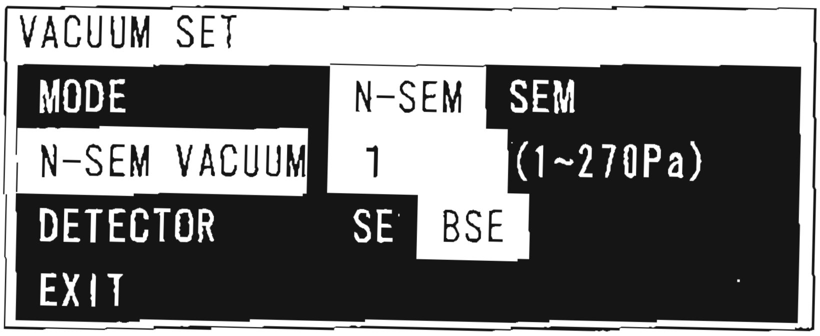
A flicker-free TV-scan display associated with a realtime slow to TV scan conversion system using frame memories are adopted for S-2460N. Since conventional SEM depends on an after-glow image on viewing CRT, image brightness is insufficient for observation in normally lit room as shown in Fig. 5 a. The frame memory-based real time scan conversion function provides quality images as shown in Fig. 5 b. This design allows microscopy without a need of dark room facility. There are three set of frame memory systems including one 512x512 pixel buffer and two 1024x1024 pixel precision memories as shown in Fig. 6. The system allows versatile image recording techniques in adition to conventional photographic cameras. Besides, image processing functions such as contrast manipulation, split screen display, image integration are also incorporated.

4. X-ray microanalysis

X-ray analysis in low vacuum conditions will result in loss of spatial resolution due to



a) Vacuum preset with control knob
(S-2250N, S-2360N)



b)Menu operation (S-2460N)

Fig. 3 Vacuum preset and monitoring in low vacuum condition

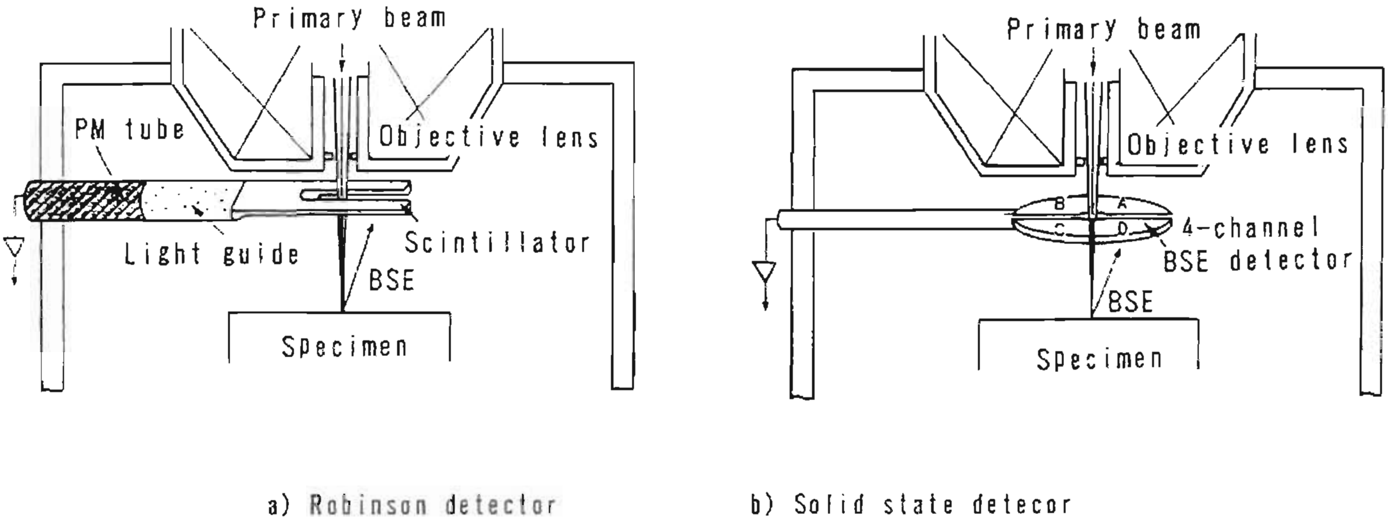


Fig. 4 BSE detectors for Natural SEM

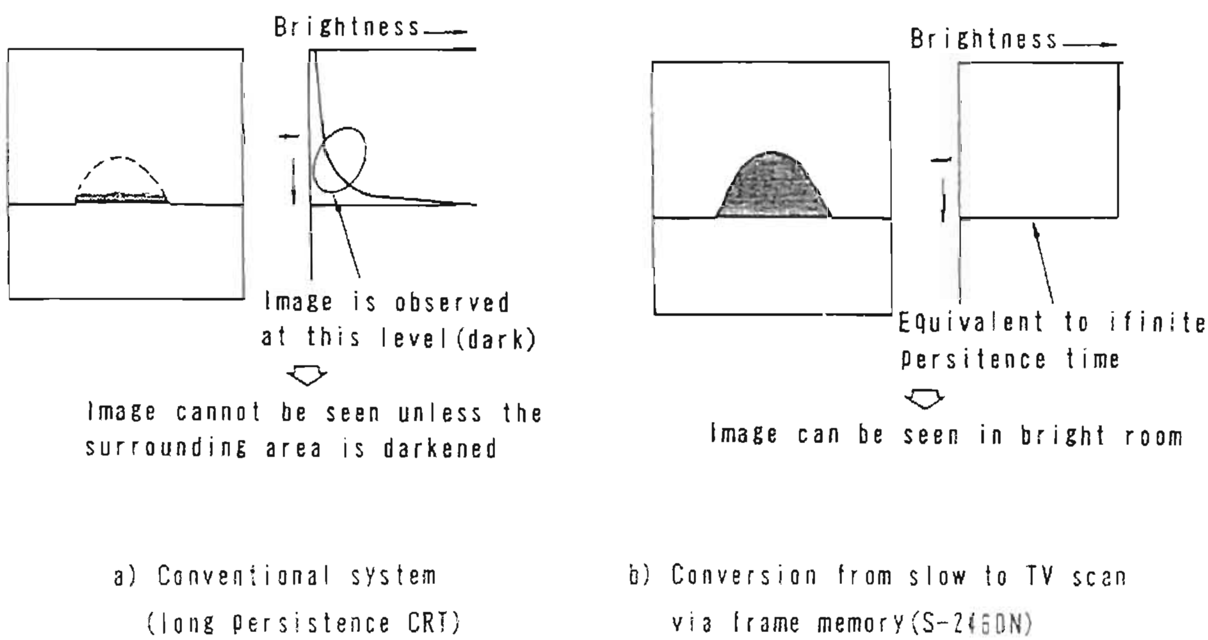


Fig. 5 A real time Slow - TV scan conversion function (S-2460N)

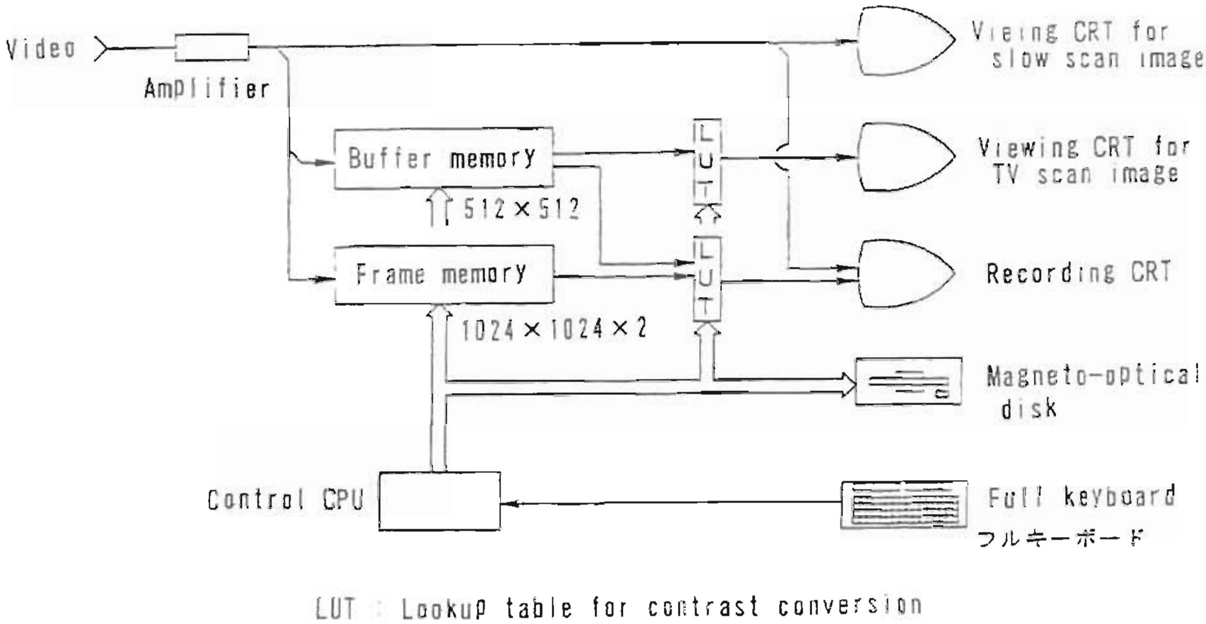


Fig. 6 Integrated frame memory system for S-2460N

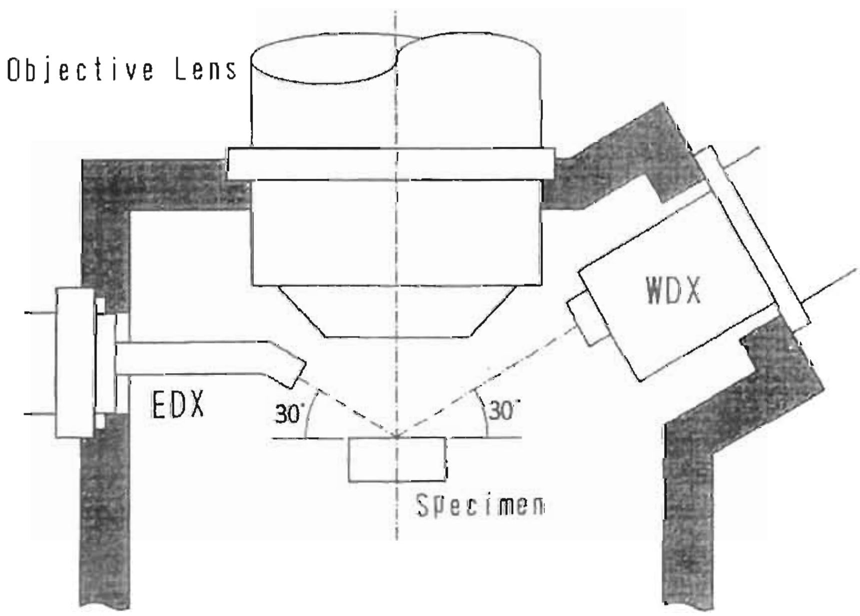
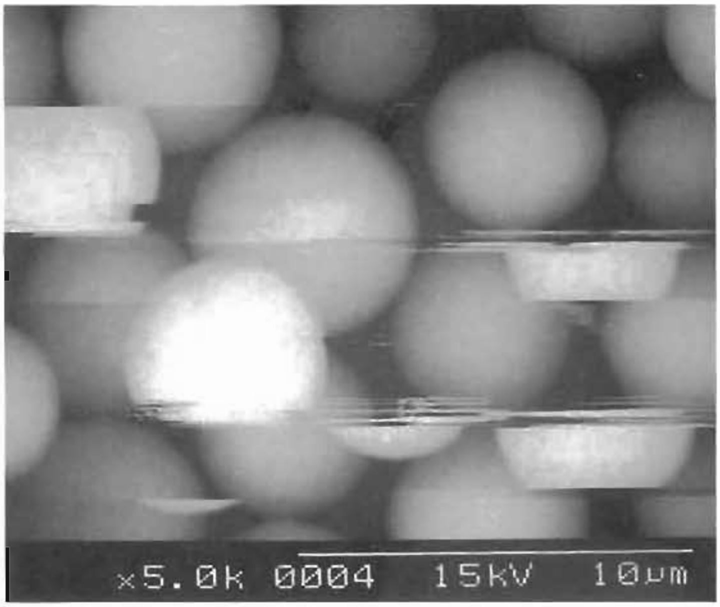
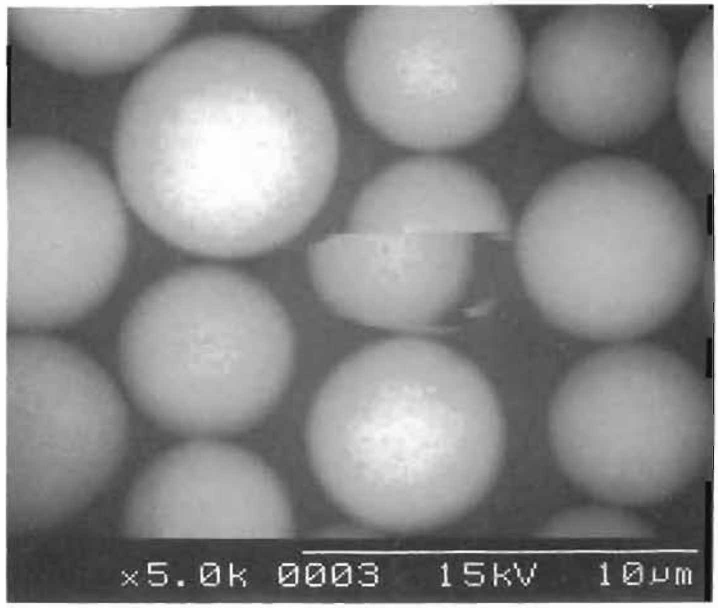


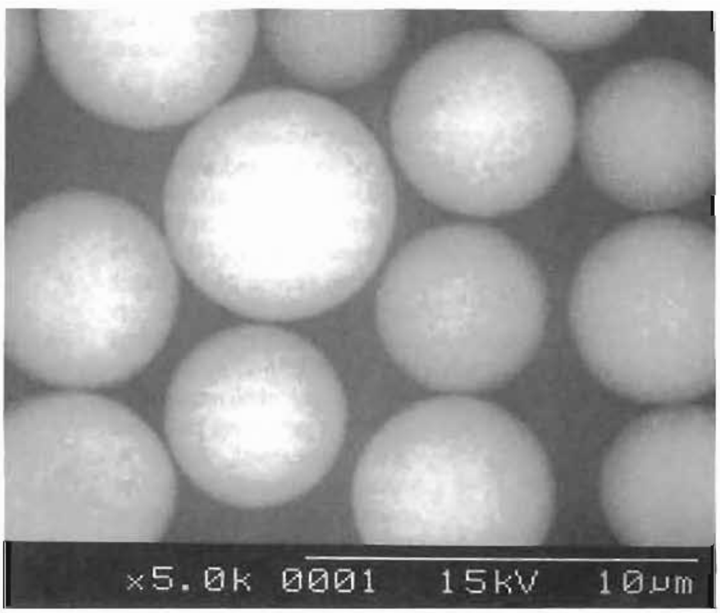
Fig. 7 Accomodation of EDX and WDX Spectrometers



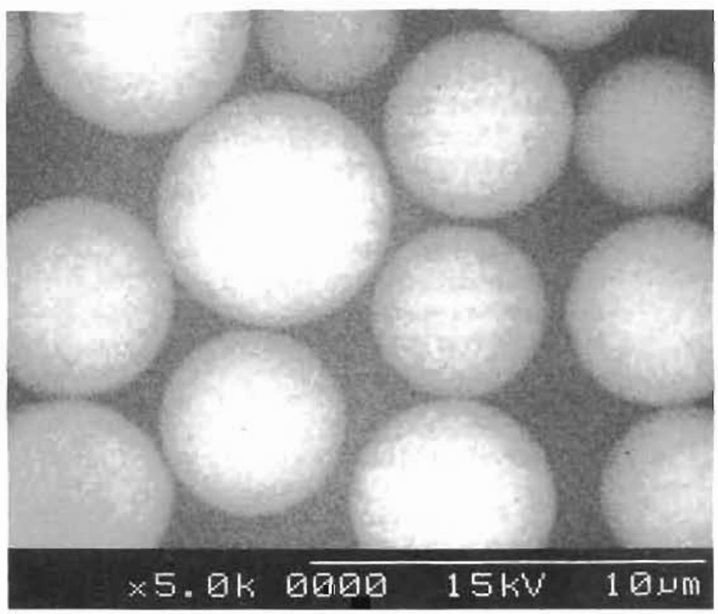
a) Vacuum 1.3Pa



b) Vacuum 13Pa



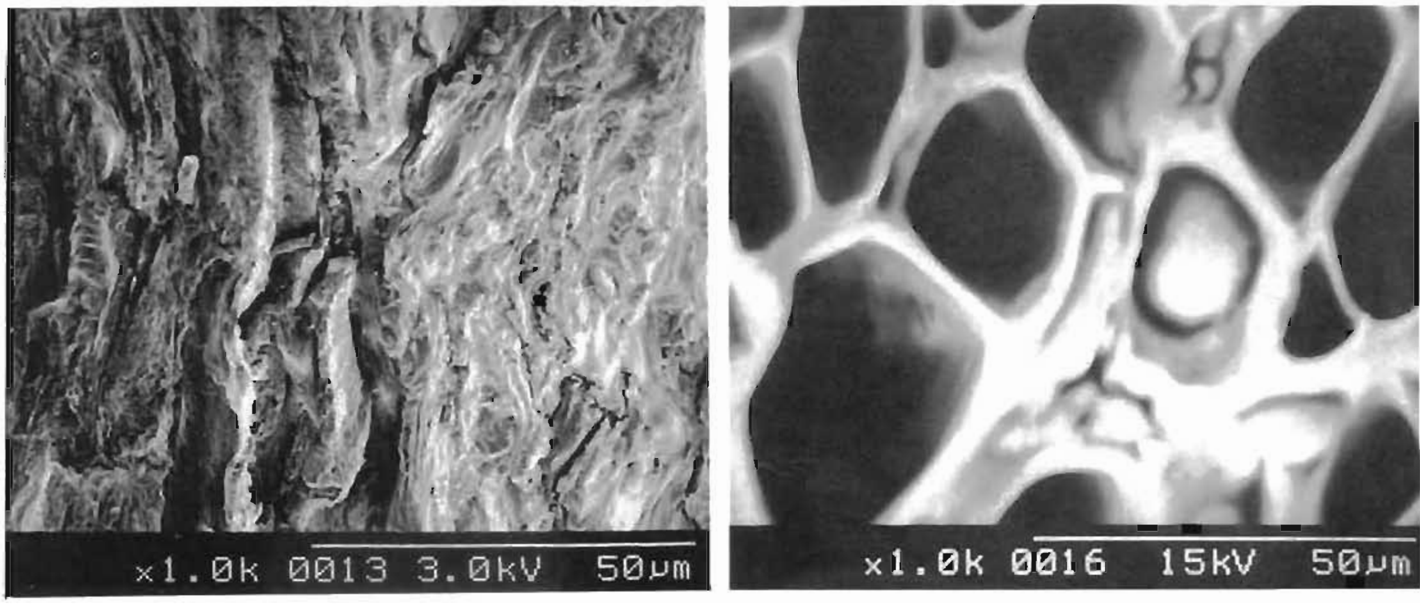
c) Vacuum 130Pa



d) Vacuum 270Pa

Specimen : Packing material (silica gel) for HPLC

Fig. 8 Observation of insulating material



a) Dried seaweed — vacuum

b) Seaweed soaked in water for 30minutes — 13 Pa

Fig. 9 Observation of water contained specimen

electron scattering by gas molecules [4] [5]. But, it is useful from a view point of nondestructive analysis for insulators in spite of some restrictions [6]. Natural SEMs allow accommodation of Energy Dispersive X-ray spectrometers (EDX) and Wavelength Dispersive X-ray spectrometers (WDX) as shown in Fig. 7. An X-Ray take off angle of 30 degrees is available for WDX and mostly for EDX. However, the use of WDX is recommended only in the high vacuum mode because the system use a gas flow detector, which is usually energized at a high voltage.

APPLICATIONS

1. Observation of insulating materials

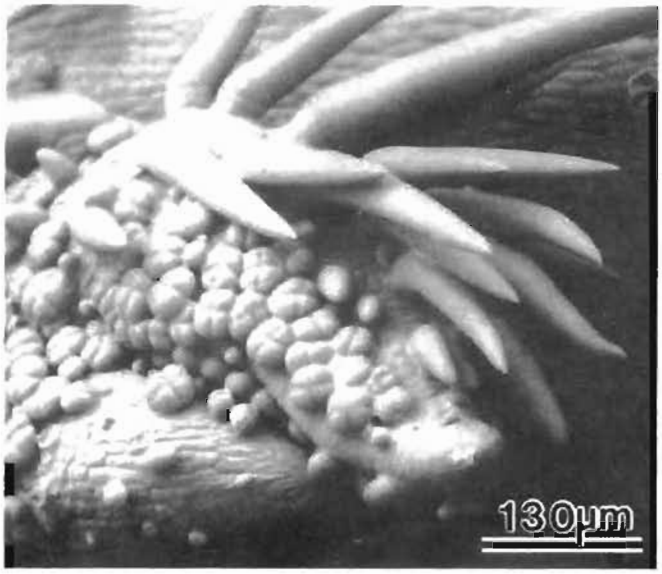
Photographs in Fig. 8 show uncoated SiO_2 powder (about 5 μm in diameter) taken with the S-2250N at 1.3 ~ 270Pa. Defective images with unusual contrast and disorder of image caused by charging phenomenon are visible in vacuum less than 13 Pa (Fig. 8 a, b). However, charging was remarkably reduced at 130Pa as shown in Fig. 8 c and a stable observation was possible. Although any defective image by charging was not observed in 270Pa (Fig. 8d), S/N ratio of the image was slightly dropped. This problem was caused by scattering of primary and backscattered electrons because of a short mean free path.

2. Study of specimens including water

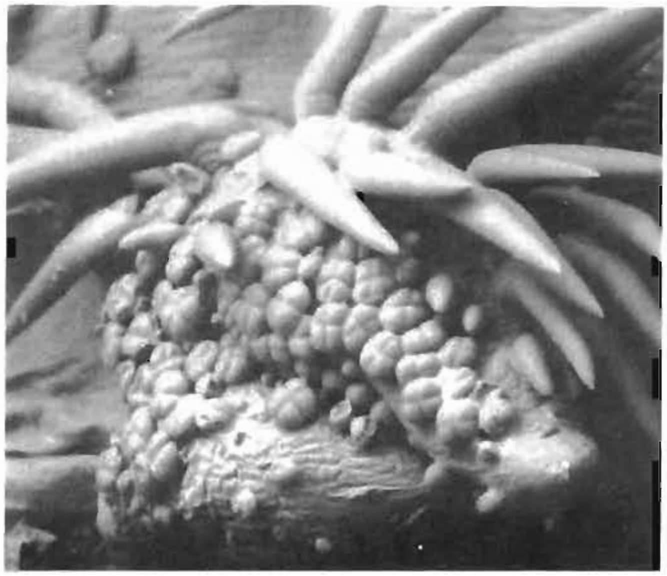
Micrographs taken with a dried seaweed and the same soaked in water for 30 minutes at 13Pa are shown in Fig. 9. An observation under saturated water vapor pressure (about 2700Pa) is impossible since the operating range of Natural SEM is 1~270Pa. Although water in the water-soaked specimen was evaported, some pits which were supposed to have water contained were observed without shrinkage or deformation. Fig. 10 shows microsgraphs of a growing point of morning-glory continuously observed at 130Pa. Authors confirmed no major damage to the specimen for the first 5 minutes. The observation of mold in Fig. 11 was stably practiced without specimen preparation.

3. X-ray microanalysis in the low vacuum condition

Fig. 12 shows an experimental result of relative intensity for low energy X-rays. Intensities of O-K and Ti-L lines declined at 50 Pa or higher. The X-ray absorption by gas molecules was increased as the pressure rises. On the other



a) 2 minuts after started to observe



b) 5minuts

c) 20minuts

Specimen : Growing point of morning glory

Vacuum : 130Pa

Fig. 10 Deformation of biological specimen in low vacuum condition

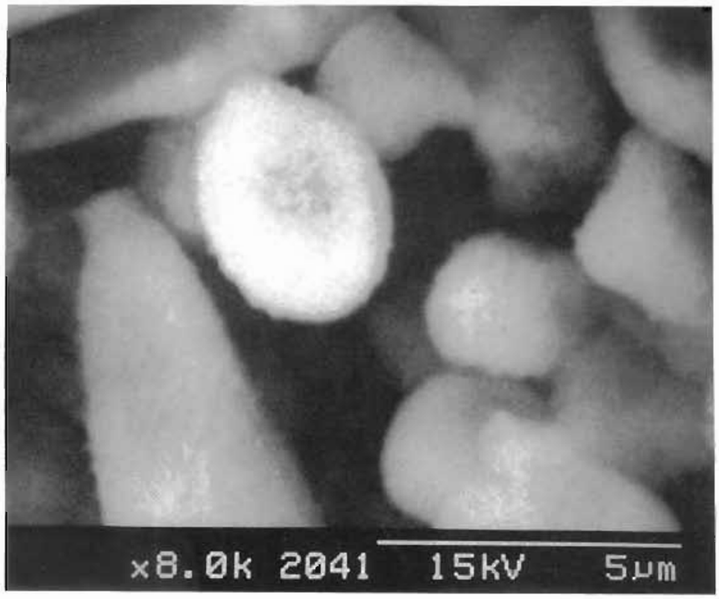


Fig. 11 Observation of mold (at 13Pa)

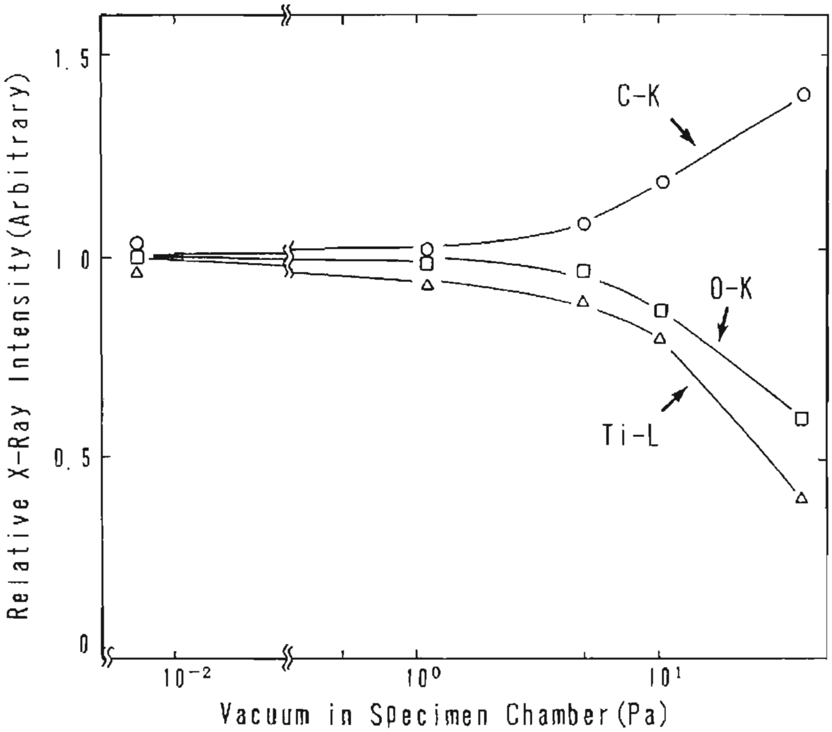


Fig. 12 Relative intensity of low energy X-ray as a function of Vacuum

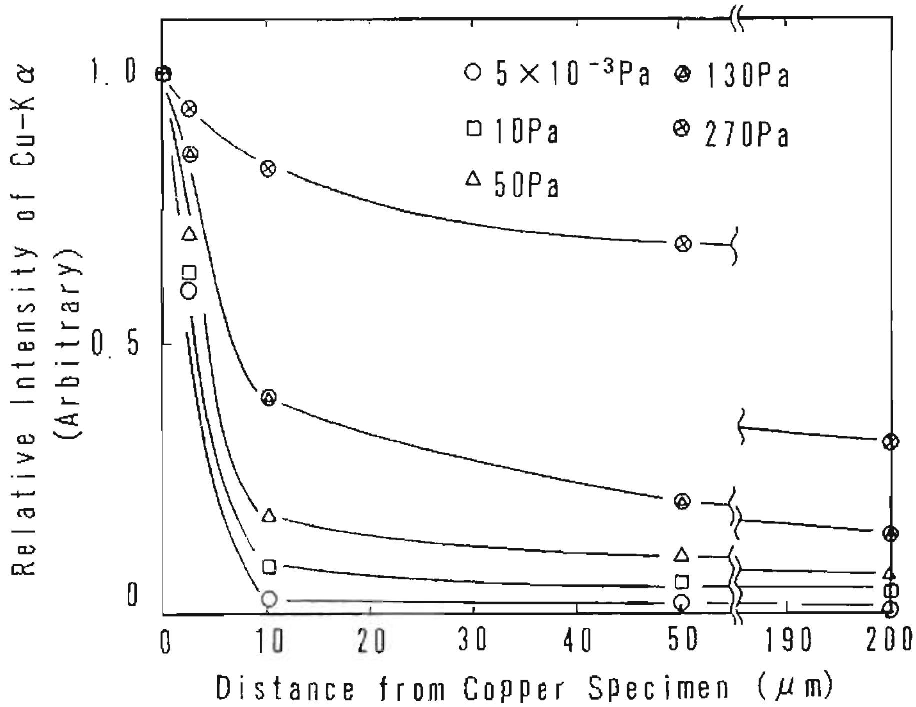


Fig. 13 Plot of relative intensity (Cu Kα) as a function of distance from copper specimen

hand, C-K line increased in intensity, but the reason has not been cleared yet.

Change of relative intensity of Cu-kα line depend on the vacuum condition and the distance between specimen and primay beam as shown in Fig. 13 (The intensity when the beam irradiated on the specimen was defined as 1). From this result, it is assured that undesired X-rays were generated when the pressure exceeded 100Pa even at a distant position of 10 μm from the specimen. Fig. 14 displays an analytical result of processed cheese at 100Pa for an additive "A" and a fat globule "B".

CONCLUSIONS

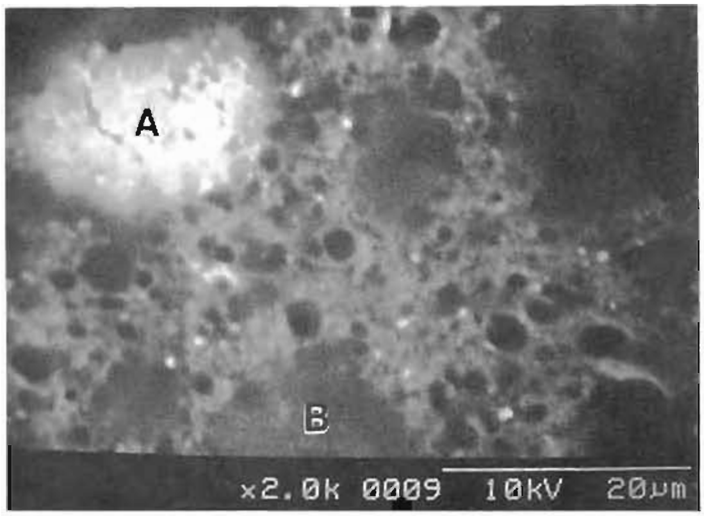
A charging phenomenon was reduced at vacuum condition over 100Pa. It was comfirmed that image quality, analytical accuracy and vacuum condition are closely related. Therefore, a stable low vacuum system is desired. Natural SEM have met these requirements by the real time vacuum control mechanism and exlusive evacuating system for low vacuum. Natural SEMs do not allow observation and analysis under saturated vapor pressure of water, but it is quite significant that a nondestructive study of wet specimens and insulators are perfomed without pretreatment. It is expected that these new SEMs will make contributions for expansion of application fields for SEMs.

RESUMEN

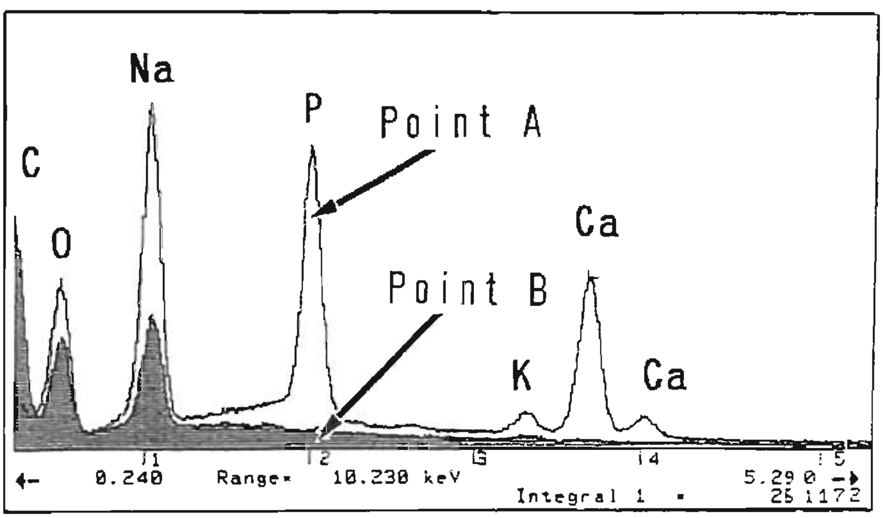
Recientemente, los estudios de los especímenes de tipo aislantes o los que contienen agua, se han incrementado debido al amplio uso de los microscópios de barrido ambientales.

Los autores han desarrollado técnicas que operan a bajo vacío (1 - 270 Pa). Una condición estable de bajo vacío se logra debido al control en tiempo real de la función de bajo vacío y a la creación de un sistema de evacuación independiente. De esta forma, se evita la influencia de factores externos como la degasificación de la muestra y de la función de cambio típica al funcionamiento en condición de alto vacío.

Los resultados han confirmado que el fenómeno de carga se puede reducir cuando se estudia (caracterizan) muestras aislantes o muestras biológicas ó de alimentos. Aún más, el área de generación de rayos X se puede reducir a 10 μm para 100 Pa. Se espera, de esta forma, un aumento en el campo de aplicación de la técnica de microscopía electrónica de barrido que permitirá la observación y el análisis de las muestras en una forma no destructiva.



a) BSE image



b) EDX spectra of point A and B

Vacuum 130Pa

Specimen Processed cheese

Fig. 14 An example of EDX analysis in low vacuum atmosphere.

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