Report to AOARD/AFOSR

Secondary Electron Emission (SEE) Measurements on Materials under Stress
(Preliminary measurements of SEE under high temperature condition)

May 23, 2003

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ABSTRACT (Maximum 200 words)

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Results can be summarized as follows;

1. The SEE coefficients of silicon and sapphire could be measured under room and high temperature.

2. In relative high temperature (200 °C), an appreciable reduction in the SEE coefficient was confirmed for both silicon and sapphire.

3. Position dependence (not only temperature dependence) of the SEE coefficient was observed in sapphire.

4. The peak of energy distribution for secondary electrons was in about 8 eV, and the width spreaded over about 3.4 eV. The temperature rise caused decrease of energy about 0.7 eV, and did not affect the spread.

5. The peak height of the distribution decreased with temperature.

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Summary

Secondary electron emission (SEE) coefficient of sapphire and Si and energy distributions of secondary electrons from aluminas were measured under high temperature at about 400 ℃. These measurements intended to establish measurement systems of SEE coefficient and energy analysis of secondary electrons under high temperature conditions. Sample stages of scanning electron microscope (SEM) and X-ray photoelectron spectroscope (XPS) were modified to enable a sample to heat upto a temperature. SEE coefficients were measured by the modified SEM with pulsed electron beam. The modified XPS system was used to analyze energies of secondary electrons.

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1. Introduction

Klystrons, the high power microwave generator, are used for particle acceleration energy sources in the high energy accelerator. In the klystron, there is a rf window which made of an insulator. Surface flashover along the surface of rf window insulator is a present problem in the klystron applications for high power use. Therefore, it is necessary to understand the surface flashover phenomena and thus improve transmission capability of high power microwave [1,2].

The important points in the breakdown mechanism along the rf window insulator are multiplication of secondary electrons emitted from the insulator surface and the temperature rise caused by microwaves which pass through the insulator. In the previous interim report, SEE, CL, effect of X-ray irradiation, and tentative results of SEE energy distribution were reported [3]. These results, however, were measured under room temperature condition except the last one. An rf-window of a klystron under operation may be heated by loss caused by the microwave transmission. Therefore, study of temperature effect on secondary electron emission (SEE) characteristic is required to understand the breakdown process in the rf window.

Some theories have proposed to explain the SEE characteristic under high temperature conditions [4,5,6,7]. However, there are still only few data of the SEE under high temperature conditions, especially for technical material. This study aims to measure the SEE coefficient under high temperature conditions of technical materials insulators, such as alumina. At present, the samples measured were silicon and sapphire. The measurement of silicon was carried out because of two reasons; (a) the electronic state of silicon looks like that of insulator, (b) there is no charge up on this sample surface when an electron beam is injected. Therefore, the silicon is a good sample to practice measurement skill at beginning of this study. The SEE coefficient of alumina will be measured in the next experiments.

This time our experimental systems have been modified to enable measurements under high temperature conditions. In this interim report, firstly experimental systems prepared for the SEE coefficient measurements and secondary electron energy distribution analysis system under high temperature conditions have been described, then temperature dependence of the SEE coefficient and secondary electron energy distributions have been reported and discussed, and finally results have been summarized.

2. Experimental

2-1 SEE measurement system and sample heating unit

In this study, the secondary electron emission coefficients (δ) were measured under room and high temperature conditions by using Scanning Electron Microscope (SEM). A heater circuit was used to observe SEE coefficients (δ) under high temperature conditions. Measurements of SEE coefficients could be carried out by two methods: (i) measuring the absorption currents, or (ii) measuring the secondary electron currents.

Schematic diagram of the absorption current measurement method is shown in Fig. 1. The absorption currents were measured by applying a negative bias voltage to insure that the secondary electron emissions were not absorbed into the sample. In order to measure primary currents, a faraday cup was installed on the sample holder. A sapphire insulator (0.3 mm thickness) was used to prevent currents flowing to ground. Fig. 2 shows photograph of sample holder used in this experiment.

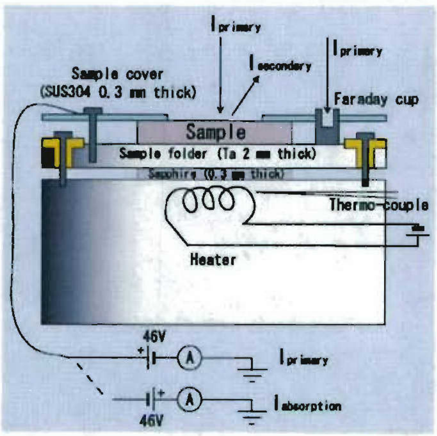

Fig. 1 Schematic diagram of absorption current measurements

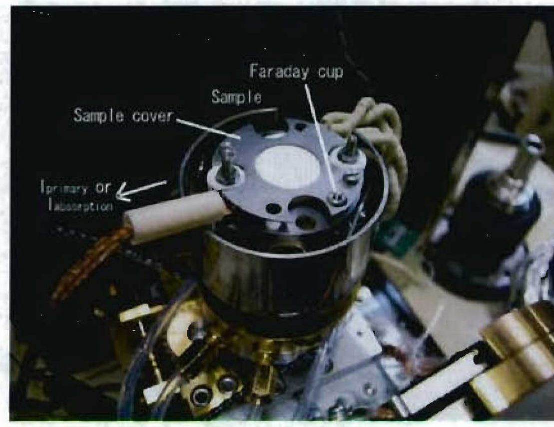

Fig. 2 Picture of sample holder used in this experiment.

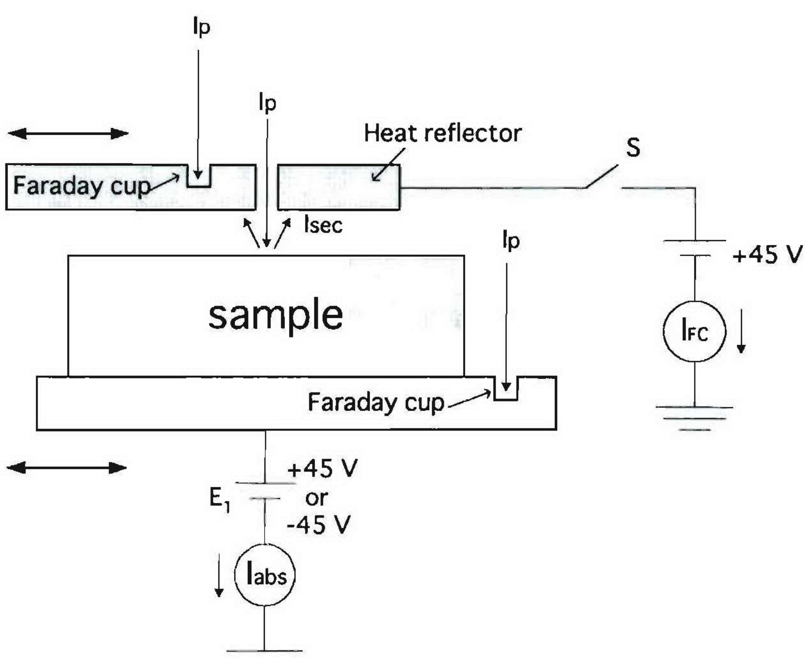

Fig. 3 A new configuration of SEE coefficient measurements.

Schematic diagram of the secondary electron current measurement method is shown in Fig. 3. The secondary electron currents were measured using faraday cup installed above the sample. A positive bias voltage was applied to the faraday cup to ensure all of the secondary electrons were measured (switch S was ON). A negative bias voltage was applied to the sample holder to insure that the secondary electron emissions were not absorbed into the sample.

2-2 Sample and procedure

Samples used in this experiment were silicon (semiconductor), sapphire and alumina KP990 (insulator). For silicon sample, the primary current was one shot pulsed electron beam with 400 pA amplitude for 1 ms duration. There is no charge up on the silicon sample, so the measurements of SEE coefficient (δ)can be carried out at the same site of the sample.

The insulator samples were annealed at 1400℃ in air for 4 hours before measurements. This annealing process affects the insulator properties concerning the trapped or stored charges in the vacancies [2].

The primary current used for the insulator samples was one shot pulsed electron beam with 100 pA amplitude for 1 ms duration. For insulator, only once measurement at one site of the sample was carried out. Then, the insulator sample was traveled to the next measurement site by moving 2 mm for x direction and or 1 mm for y direction, as shown in Fig. 4. This procedure reduces the influence of surface charging on the measurements.

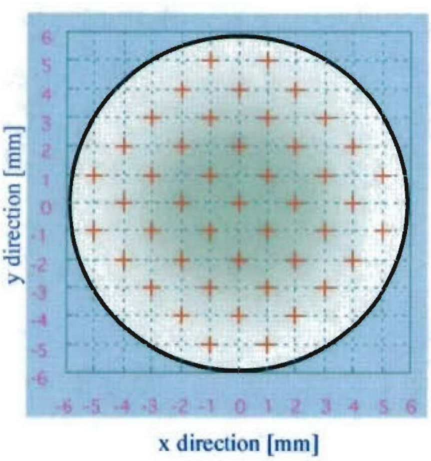

Fig. 4 Positions of measurement sites for sapphire.

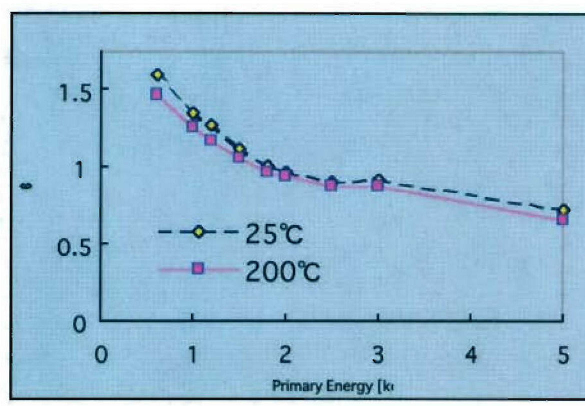

Fig. 5 SEE coefficient (δ) of silicon.

2-3 Energy distribution measurement system

Energy distributions of secondary electrons from samples were analyzed by an XPS/AES system. Schematic diagram of electron energy measurement with sample heating circuit is shown in Fig. 6. A cylindrical mirror analyzes (CMA; PHI 15-255G, energy resolution $\Delta E=0.15$ eV) with a coaxial electron gun is used. Primary electrons (Acceleration voltage:1500 V, current:2 μA) from the coaxial gun excite sample surface to emit secondary electrons. Then energies of emitted secondary electrons are analyzed by CMA operated under the XPS mode. The picture of the total system is shown in Fig. 7.

A W-Re heater mounted behind the sample holder heated the sample. Electron bombardment heating using thermionic emission from the heater is possible as well. The maximum temperature achieved was about 400 ℃, and the pressure of the vacuum chamber was in 10^{-5} Pa orders. A picture of the sample holder is shown in Fig. 8. The detail of the sample holder part is shown in Fig. 9. In this system primary electrons impinge on the sample surface continuously, then it is inevitable to suffer some influences on the energy distribution from surface charging. Even so, some valuable information may be obtained, since auger electron spectroscopy is useful to analyze insulator surface.

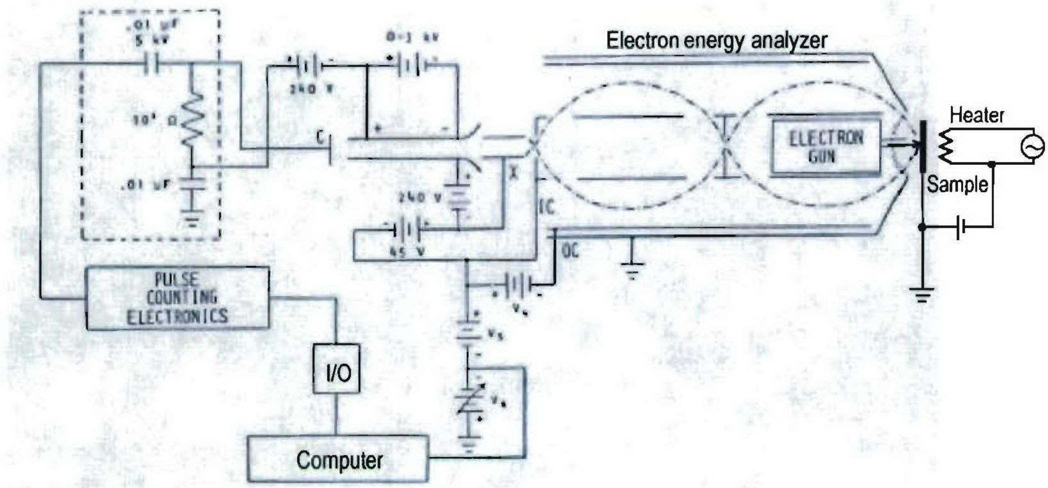

Fig. 6 Electron energy analyzer with sample heating circuit.

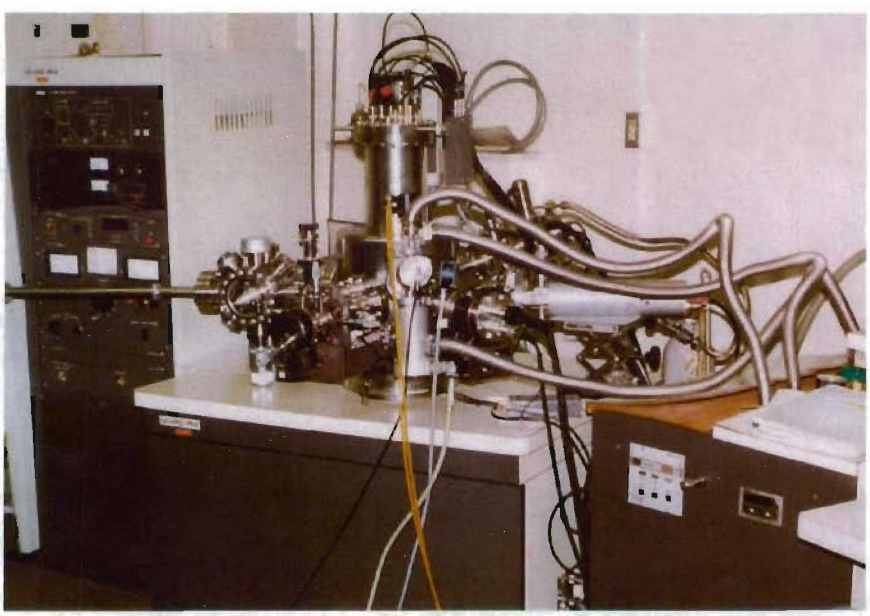

Fig. 7 A picture of electron energy analysis system having cylindrical energy analyzer.

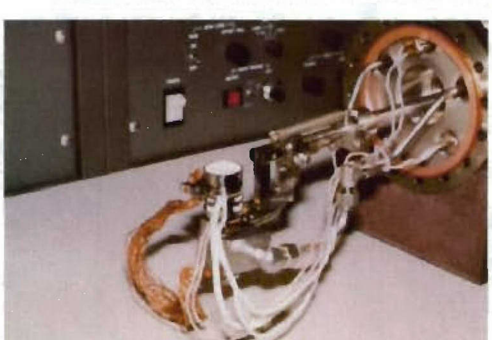

Fig. 8 Picture of sample stage.

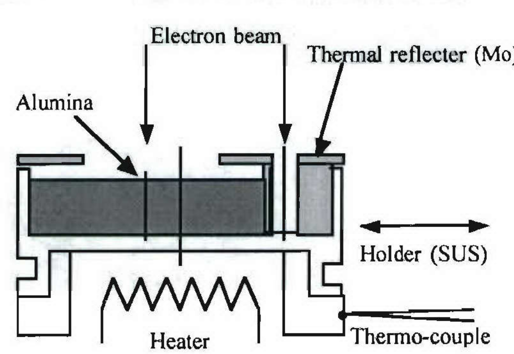

Fig. 9 Detail of the sample stage

2-4 Measurement procedure

Measurements were carried out under the following procedure

1. A test sample was set in the vacuum chamber under the pressure of $2X10^{-7}$ Pa, then it was tilted to 30° and located at the analyzing point.

2. The coaxial gun was switched on to emit primary electrons, and the primary electron current was measured at the hole made in the sample holder shown in Fig. 9.

3. The sample holder was moved to permit electrons to irradiate the sample surface.

4. CMA under the XPS-mode analyzed energy of emitted electrons.

5. After the energy analysis we started to heat the sample to about 400 ℃.

6. When the sample temperature reached the desired temperature, energy analysis of secondary electrons was carried out again by the same way as that in room temperature.

3. Results and discussion

3.1 SEE coefficients of silicon

Measurements of SEE coefficients (δ) of silicon sample were carried out by using the absorption current method under room and relative high temperature (200℃). Results of the measurements are shown in Fig. 5. The peak of the SEE coefficient curve could not be measured, because measurement of SEE coefficient using the SEM at low primary energy was very difficult.

At 200℃, the SEE coefficients of silicon are lower than that room temperature condition. The decrease of SEE coefficient of silicon at higher temperature is caused by the increased interaction between the internal secondary electron and the augmented density of free electrons in the conduction band [6].

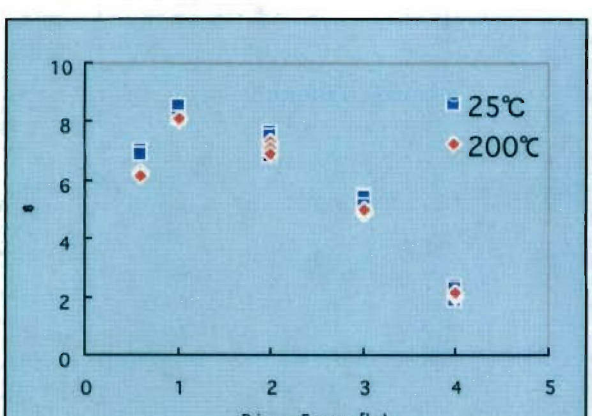

Fig.10 SEE coefficient of sapphire (sample A).

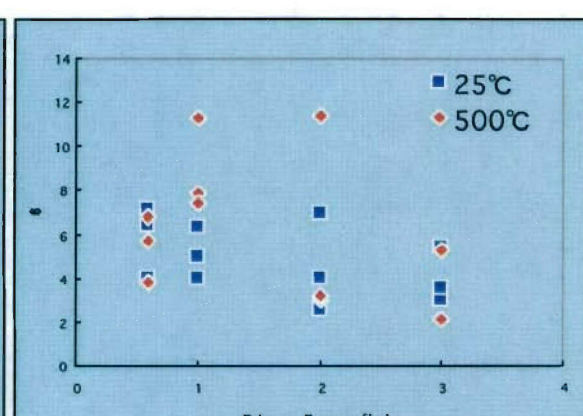

Fig.11 SEE coefficient of sapphire (sample B).

3-2 SEE coefficients of sapphire

The SEE coefficients of sapphire (sample A) under room temperature and 200℃ are shown in Fig. 10. At 200℃, the SEE coefficients of sapphire are lower than that obtained under room temperature condition. Fig. 11 shows SEE coefficients of another sapphire sample (sample B) under room temperature and 500℃. For the sample B of sapphire, the SEE coefficient data have high standard deviation. It shows position dependence possibility of the SEE coefficient.

At present, there are still only few theories of the SEE under high temperature conditions, especially for insulator material. However according to empirical data and references, it is considered that the decrease of SEE coefficients of sapphire when temperature increases may be caused by phonons and electrons scattering in the bulk before escaping from the surface. Therefore, under high temperature conditions, the secondary electron emitted to the surface is less than that of in room temperature.

The SEE coefficients of sapphire in Fig. 10 and Fig. 11 are larger than those of silicon shown in Fig. 5. This difference may be ascribed primarily to two sources; those are (a) the energy required for a free electron to escape from the solid, (b) the rate of energy loss of internal secondaries as they approach the surface [6]. These two factors do not act independently, for, if the rate of energy loss is high for all energies of the internal secondaries which lie between their initial energy and that required for escape, the coefficient should be low. This represents the situation in semiconductor where there are more electrons with which the secondaries can make allowable collisions involving either large or small transfers of energy.

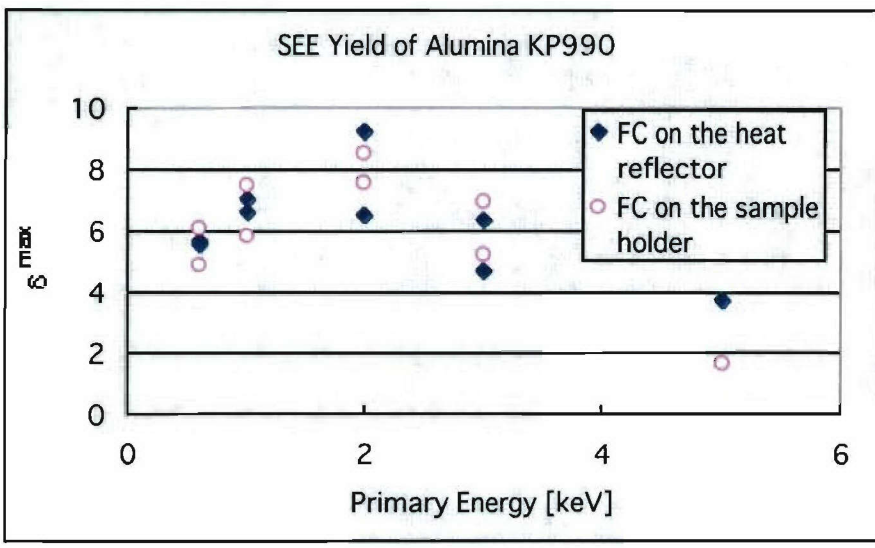

Fig. 12 SEE coefficients of alumina KP990

- Faraday cup (FC) on the heat reflector:

Ip : E_1=+45V, S=ON

Isec: E_1= −45V, S=ON

- Faraday cup (FC) on the sample holder:

Ip : E_1=+45V, S=OFF

Isec: E_1= −45V, S=OFF

3-3 SEE coefficients of alumina

The SEE coefficients of alumina KP990 (Alumina purity : 99.5 %) under room temperature measured by absorption and secondary current method is shown in Fig. 12. SEE coefficients of the sample measured by both of the methods were almost same. The SEE coefficients of the sample under high temperature conditions will be measured in the next experiments.

3-4 Energy distribution

3-4-1 SEE energy distribution

Energy distributions of secondary electrons measured at room temperature (22 ℃) and high temperature (429 ℃) for alumina HA95 (Alumina purity : 95 %) are shown in Fig.13. At room temperature energy at peak position locates at about 7.6 eV, and its full width at half maximum is about 3.4 eV.

The peak height decreases with temperature. In addition the peak of the spectrum shifts towads lower energy side with temperature. In this case its shift is can be estimated as 0.7 eV. The decrease of peak height with temperature is reasonable [4]. The rate of decrease, however, seems fairly large, since secondary electron yield δ at 700 ℃ and 1.0 keV primary electron energy decreases to 90 % of the value at 25 ℃ for Mg single crystal [8].

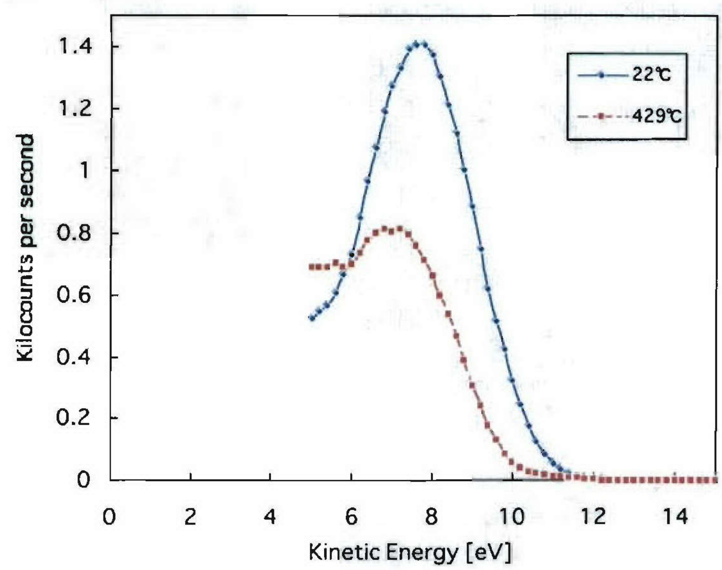

Fig. 13 Secondary electron energy distributions at room temperature and 429 ℃ (Sample:Alumina HA95)

Further measurements were carried out for another type of aluminas ; named KP95 (Alumina purity : 95 %), and KP990. Results are shown in Fig.14 for KP95 and Fig. 15 for KP990.

Both Fig.s show decrease of secondary electrons with temperature. Shift of spectrum to lower energy side is significant for alumina KP990, while it is not obvious for alumina KP95. At room temperature the peak of the energy distribution locates at the energy of 8.5 eV. This value is 0.9 eV higher than that of HA95. This difference may come from not only intrinsic characteristics of material but also many factors surface charging, surface contamination, etc.. The full width at half maximum of the spectrum for KP990 at room temperature can be estimated as 3.3 eV. This value almost coincides with that for HA95. Peak shift of the spectrum at high temperature for KP990 is about 0.93 eV.

For KP95 the full width at half maximum could not be estimated, since the slope of lower side of energy spectra were eliminated. This is because the lower limit of energy that the energy analyzer is capable to analyze energy is 5 eV. When the sample surface is positively charged, emitted electrons decelerated to less than 5 eV can not be analyzed. To eliminate this defect in analyzer behavior, it is effective to bias sample negatively. In the future experiment program we will prepare a power supply to apply bias voltage.

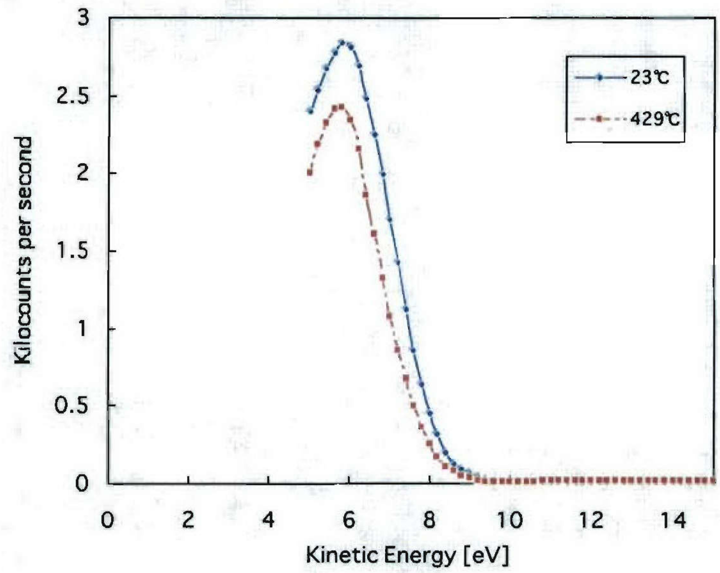

Fig. 14 Electron energy distributions at room temperature and high temperature.
(Sample: Alumina KP95)

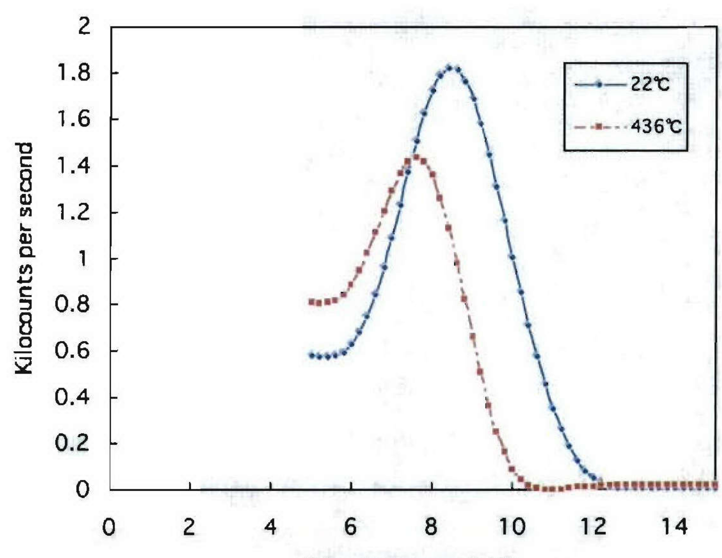

Fig. 15 Electron energy distributions at room temperature and high temperature.
(Sample: Alumina KP990)

3-4-2 Elastic peak

Elastic peak was analyzed as well, since the energy of the elastically scattered electrons is independent of surface charging. Example of elastic peak is shown in Fig. 16. This figure. compares elastic peaks obtained for alumina HA95 at room temperature with that at high temperature. While shift of the peak is not found, intensity decreases at high temperature.

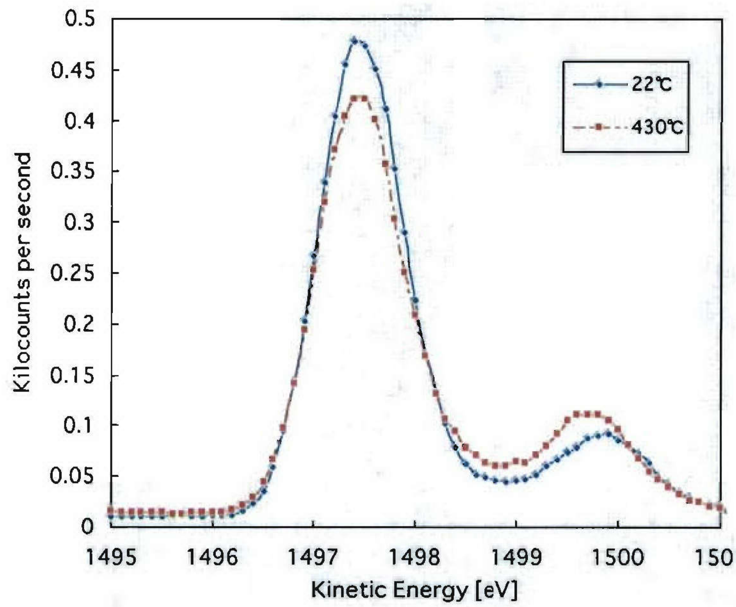

Fig. 16 Elastic peak of scattered electrons.
(Sample: Alumina HA95)

Electron emission is very sensitive to surface conditions. Electron energy spectra shown in Fig. 13 to 16 are influenced surface conditions of samples. To canvass electron energy distribution, it is to analyze surface condition of samples.

4. Conclusion

According to results of this study, it can be conclude that:

a. SEE coefficients of silicon and sapphire could be measured under room and high temperature. SEE coefficients of alumina also were measured under room temperature by absorption and secondary current method.

b. SEE coefficients of alumina KP990 measured by absorption and secondary current method were almost same.

c. In relative high temperature (200℃), an appreciable reduction of SEE coefficient was confirmed for both silicon and sapphire.

d. Position dependence possibility (not only temperature dependence) of SEE coefficient was observed in sapphire (sample B).

e. Electron energy distribution showed that energies of emitted electrons are in about 8 eV at room temperature and its full width at half maximum is in about 3.4 eV at room temprature. These values are independent of samples measured.

f. At high temperature (about 430 ℃) energies of emitted electrons decreases about 0.7 eV, and the number of electrons decreased.

g. To canvass electron emission characteristics described above, it is necessary to analyze surface condition, since sample surface conditions strongly influence on electron emission characteristics.

5. Further experiments planned

In the future, this study will be continued to:

a. Improve the sample holder in order to carry out more high temperature measurement.

b. Investigate the position dependence possibility of secondary electron emission (SEE) coefficient of the insulators.

c. Carry out SEE coefficient measurements under room and high temperature conditions for technical materials, such as alumina.

d. Analyze sample surface condition by for example XPS or AES, in conjunction with electron energy distribution measurements.

e. Process sample surface in vacuum.

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