

Paper

## The Standard Auger Electron Spectra in The AIST DIO-DB(111); Ge(111)

K. Goto,<sup>a,\*</sup> Adel Alkafri,<sup>b</sup> Y. Ichikawa,<sup>b</sup> A.Kurokawa,<sup>c</sup> and Y. Yamauchi<sup>a</sup>

<sup>a</sup>AIST-Chubu Center, Anagahora 2266, Moriyama-ku, Nagoya 463-8560, Japan

<sup>b</sup>Nagoya Institute of Technology, Gokiso-cho, Showa-ku, Nagoya 466-8555, Japan

<sup>c</sup>AIST-Tsukuba, Umezono 1-1-1, Tsukuba 305-8568, Japan

\*gotou.keisuke@aist.go.jp

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This paper is a brief review of our final absolute Auger electron spectroscopy which was presented in the work shop on “Electron Transport Parameters Applied in Surface Analysis” (Lake Balaton, Hungary, Sept. 14-17, 2008) as a poster and new data of Ge(111). Some modifications and corrections are made from the poster. This work has been continued in these 21 years and only the significant change was the transmission of the meshes in the analyzer (cylindrical mirror analyzer); *effective* transmission being 0.22sr and 0.199(9)sr for 42.3 ± 6 degrees of emission cone for the data before and after 2008, respectively. This change was due to the over-coating of soot on the former aquadag coating on the gold plated tungsten meshes to stabilize the work function and to reduce the scattered electrons and secondary electrons at the mesh. These Auger electron spectra can now be available on the web; <http://www.sasj/COMPRO> and <http://riodb.ibase.aist.go.jp/DB111/welcome.html>.

### 1. Introduction

In surface analysis Auger electron spectroscopy and photoelectron spectroscopy are now the most significant methods and the international standard (ISO) for the methods have already been determined and progressive; ISO 17973 “Surface chemical analysis –Medium-resolution Auger electron spectrometers- Calibration of energy scales for elemental analysis” [1], ISO 17974 “Surface chemical analysis –High-resolution Auger electron spectrometers- Calibration of energy scales for elemental and chemical-state analysis” [2], ISO 20903 “Surface chemical analysis –Auger electroscopy and X-ray photoelectron spectroscopy- Peak intensity determination” [3], ISO21270 “Surface chemical analysis- X-ray photoelectron and Auger electron spectroscopies- Linearity of intensity scale” [4], ISO 24236 “Surface chemical analysis –AES-repeatability and constancy of intensity scale” [5]. While very limited numbers of spectra have been given in the ISO standards and thus it is very inconvenient to analyze the surface without standard spectra. We have developed an absolute Auger electron spectroscopy since 1987, in which the

spectra are given for the whole range of energies from zero through the elastically backscattered primary electrons. Thermionic primary electrons were accelerated by precision voltage of 1V through 5kV. Experiments are now progressive and some spectra obtained can be available in the webs of <http://www.sasj.jp/COMPRO> and <http://riodb.ibase.aist.go.jp/DB111/welcome.html>. We review our final spectroscopy and a new set of spectra of Ge(111).

### 2. Basic design

This paper is an experimental study to obtain an absolute Auger spectra that can be SI traceable and can be available in the data base (DB) of Auger electron spectroscopy (AES) of AIST, *i.e.* DIO-DB (111) being prepared for everyone who would study AES. The basic design of the specific analyzer (cylindrical mirror analyzer; CMA) has been independently reported [6,7] and is shown in Fig. 1. This symmetrical simple design with field compensation gaps at both ends of the cylinder was determined by computer calculation. The outer diameter was maximized for the mounting flange of ICF203 mm

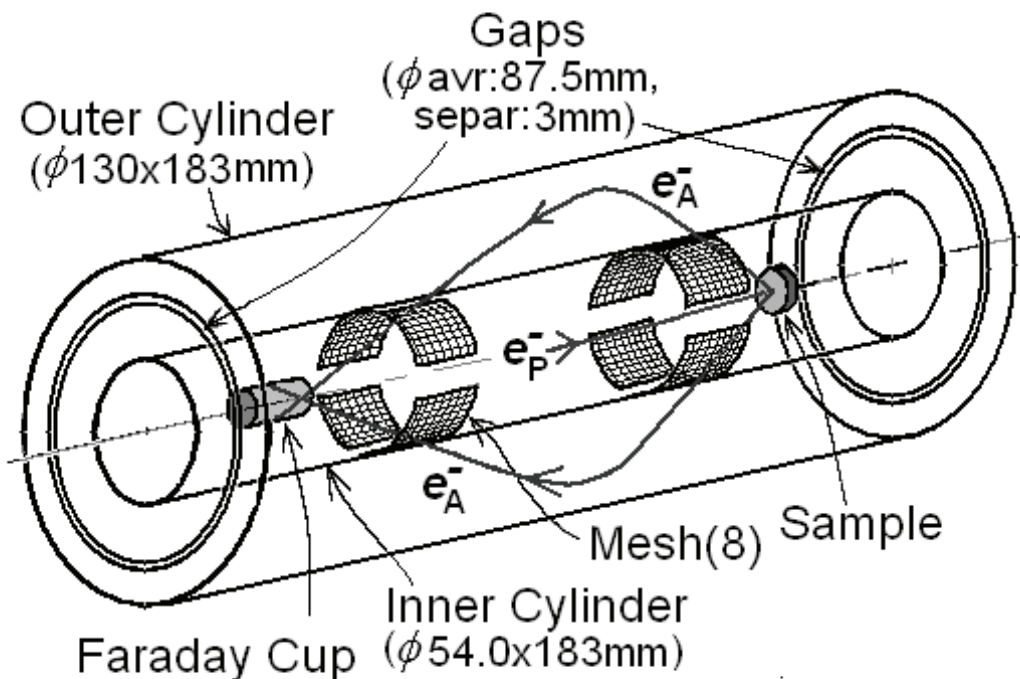


Fig. 1. The basic design of the CMA; symmetrical and the electrical field correction gaps at the both ends. Meshes gold plated tungsten (8 sheets; 100-mesh) were coated with soot and aquadag,

of diameter including a magnetic shield ( $\mu$ -metal cover). The basic design has not been changed [8].

### 3. Characteristics of the CMA

The whole system in the AIST-Chubu-Center is shown in Fig. 2 and the details of the CMA are shown in Fig. 3. The obtained characteristics of the CMA are follows:

1. Calculable property, energy to sweep-voltage, for the theory is 0.996; voltage and energy transfer coefficient,
2. the energy resolution 0.25% (FWHM) in  $\Delta E/E$ ; identical to the theoretical resolution,
3. transmission 0.177(9) sr for  $42.3^\circ \pm 6^\circ$ ,
4. primary electrons (probe) were accelerated 1 V through 5 kV (10 ppm),
5. primary probe current was detected with a Faraday cup (FC) just beside the sample holder; diameter 1.3 mm and the aspect ratio of 10 being capable of 99.9% of efficiency,
6. primary current was 1  $\mu$ A, with an extractor of 105 V with respect to the cathode, for the probe down to 30 V and decreased in the lower acceleration voltages down

to 1 V to be 12 nA, with simultaneous decreased extractor voltages,

7. probe current was stable to be 0.01%/h and thus the peak position of the spectrum can be determined to be 10 meV of resolution at 1 kV,
8. spectra were measured with another Faraday cup and electrometer (Keithley 642LN; noise levels of less than 1 fA) and ranged 0.00 eV through 5 keV, which is the whole range of the energy distribution, with the energy steps typically 0.01 eV through 1 eV depending on the structure.

### 4. Details of the CMA

The inner cylinder, Faraday-cup, slit, apertures to cut the extraneous electrons, electron gun, deflectors, secondary electron collector and sample are shown in Fig. 3. The magnetic field in the electron trajectories was far below 1 mG. An 4-wire twisted-pair connection to the ball pointed cathode (tungsten hairpin cathode, 0.3 mm diameter, 1/3 of surface magnetic field of the shank) can effectively reduce the magnetic field due to the heating

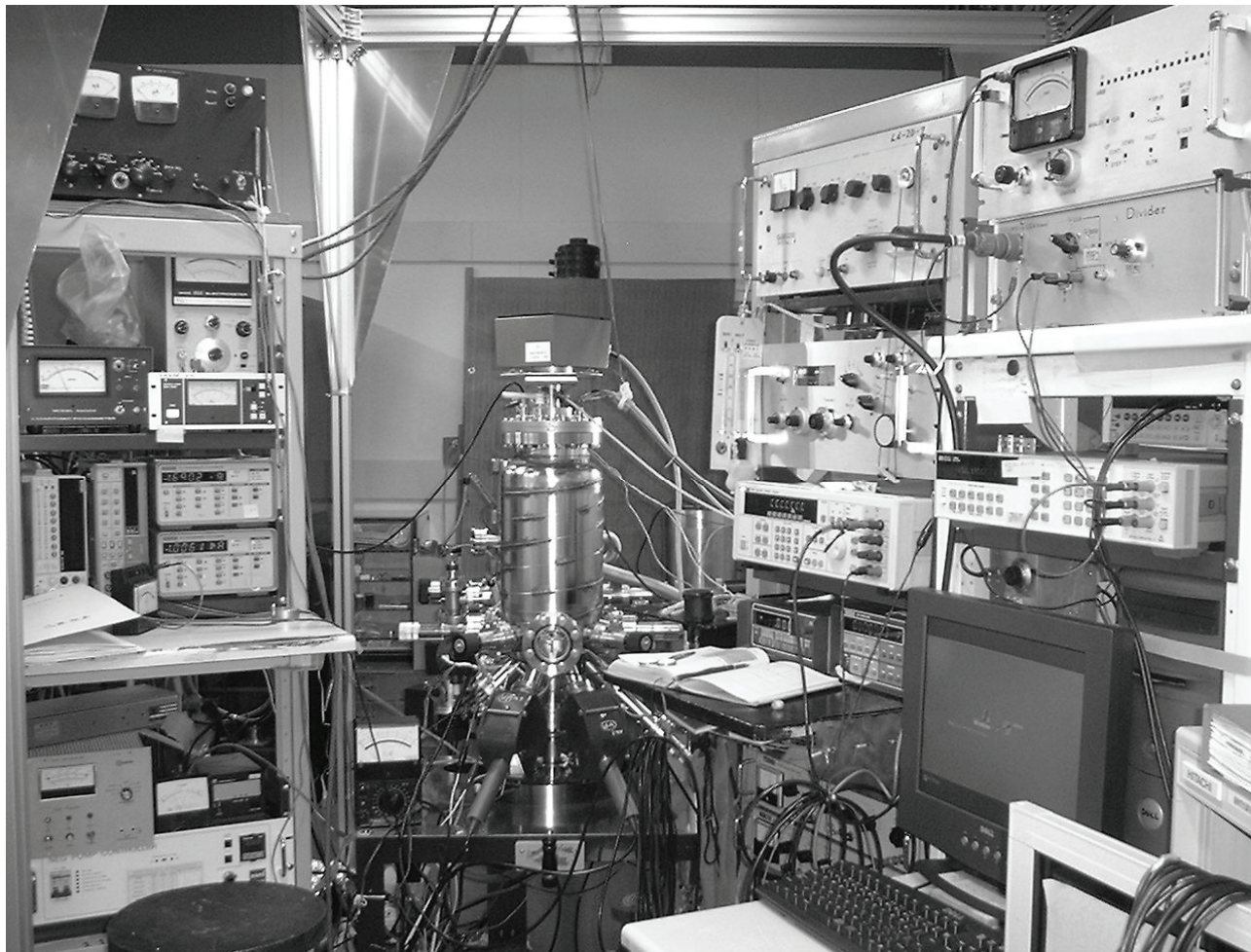


Fig. 2. The total view of experimental setup. The centers are electrometer (Keithley 642LN) and main chamber CMA being in it and sputter ion pump with magnetic shield.

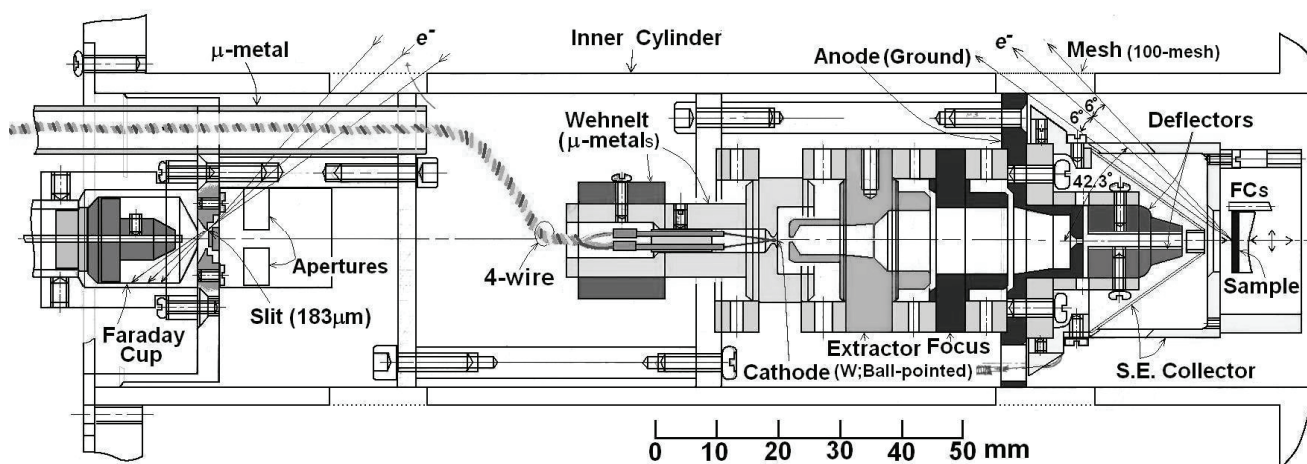


Fig. 3. Details of CMA; inner cylinder and F. Cup, electron gun, deflector, S.E. collector and sample with FCs parasitic on the sample holder to monitor the primary electron current.

current of about 2 A. Secondary electron (S.E.) collector was so sophisticated to observe the rise characteristics of the secondary electrons in connection with the work-functions by applying sample bias.

### 5. Measurements and calibration system

Measurement system is shown in Fig. 4. Signal currents were measured with electrometers (1,2, and 3). Primary acceleration voltages and sweep voltages were measured with metrological dividers (1 and 2): Each consisted of 100 pieces of precision wire-wound (Burster 1157, TC of 1pp, 2W) and sheet resistors ( $\alpha$ -Electronics MB100K, TC of 1ppm, 0.4W) of 100 k $\Omega$ . Each resistor was measured in absolute value and temperature coefficient (TC). Both dividers were compared by comparator (Keithley 155) and 20 ppm/-5 kV of differences were observed. Voltages were based on the standard voltage [10V; Fluke 732B] and the standard divider (Fluke, 752A/100V and 1000V) with care. We used elastically backscattered primary electrons in the energy calibration, in which the energy distribution of the thermionic emissions, accelerated 400 V through 1000 V, were corrected for its peak position, *i.e.* the off-

set- or iteration-method [9]. The relativistic effect was corrected as well. Electrometers were calibrated by the pA source (Keithley; 261) and this source can be calibrated by a calculable capacitance as an integrator.

### 6. Transmission measurements

Transmission of the CMA was measured carefully by setting a virtual electron source of mini-electron gun consisting of ball pointed tungsten hairpin cathode at the sample position [9]. The experiments were satisfactory enough, but we must be careful about the fact that the virtual source would not truly represent the sample. The obtained results are shown in Fig. 5. This is a raw transmission as a ratio of the input to output of the CMA. Fortunately, this results at the higher energies (about 35%) were quite identical to those of an optical simulation. Optical simulation may give better results, in general, but only applicable for the monochromatic cases.

### 7. Spectra of Ge(111)

The obtained whole spectra (0.1 eV through elastically backscattered primary electrons) for the acceleration voltages of 1 V through 5 kV are shown in Fig. 6 for a

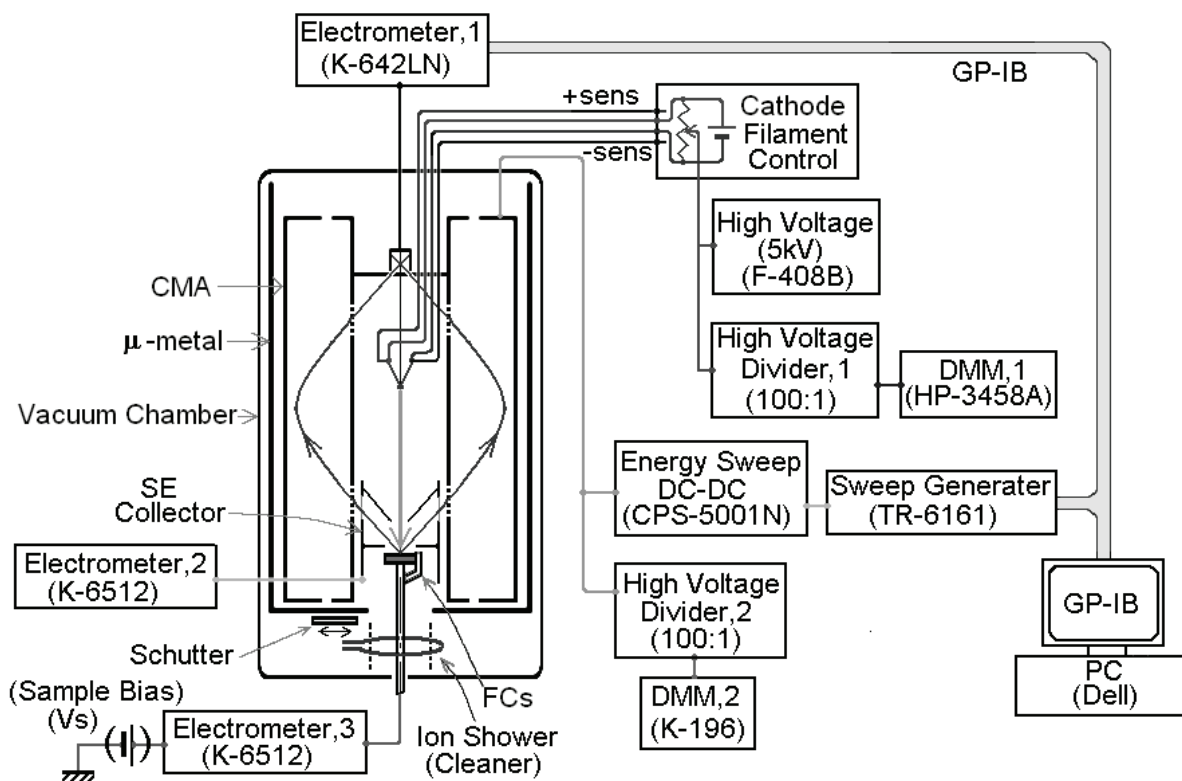


Fig. 4. Measurement system and ion shower to sputter/heat with shutter and PC with GP-IB to signal acquisition/energy sweep.



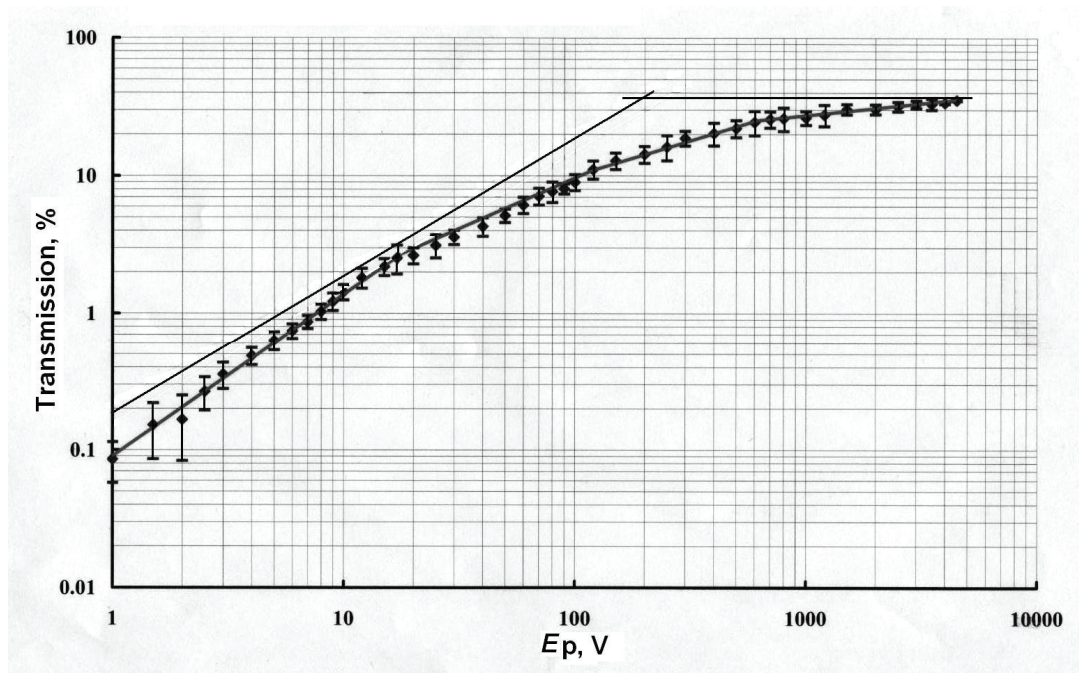


Fig. 5. Transmission measurements with virtual electron source at the sample position.

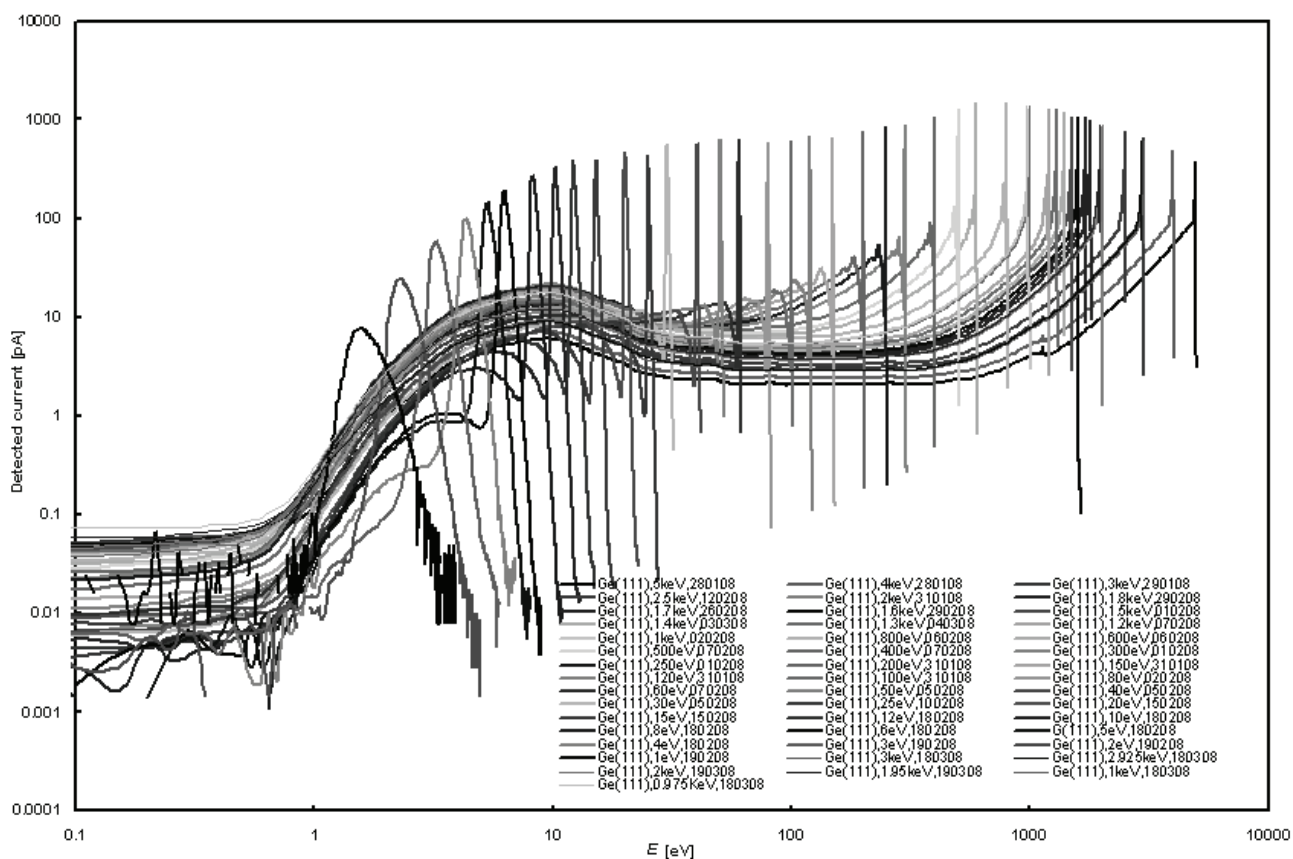


Fig. 6. A whole spectra for the primary acceleration voltages of 1 V through 5 kV; log-log plot.

Ge(111) in log-log plot. The spectra are normalized for the primary current of 1  $\mu$ A. The normalization was necessary for the acceleration of primary electrons lower than 40 V. Spectra are showing systematic changes for the energies. The shape of true secondary electrons (lower than 50 eV) present quite similar energy distributions. Practically no secondary electrons can be excited by the electrons of less than 5 eV. The ‘rise’ characteristics at about 1 eV are interesting, which may closely be related to the work function. It should be noticed that those tendencies of characteristics are quite similar for materials experienced. The ghost and scattered electrons in the analyzer and at the meshes were assessed to be typically  $10^{-5}$  of the concerned spectra [10]. The spectra are too much concentrated at a glance and the Auger spectra can hardly be seen, but in the webs. The predominant peaks of spectra (corrected for the relativity) were of for LMMs (eV); 1171.10(8), 1139.95(4), 1123.44(8), 1048.34(14), 1032.69(10), 948.16(20), and those of for LVVs (eV); 133.42(11), 103.82(19), 82.20(34).

## 8. Summary

We have attained a certain level of an absolute AES measurements but a study of work function is still remaining. Calibration of electrometer, pA, will soon be accomplished. There’s no end of final goal on this work and renewal and new data base will be given. At the end of this paper, we show our sincere thanks to Dr. T. Nagatomi for his labors in the poster session and arrangements of this paper.

## 9. References

- [1] ISO 17973: *Surface chemical analysis –Medium-resolution Auger electron spectrometers-Calibration of energy scales for elemental analysis.*
- [2] ISO 17974: *Surface chemical analysis –High-resolution Auger electron spectrometers-Calibration of energy scales for elemental and chemical-state analysis.*
- [3] ISO 20903: *Surface chemical analysis –Auger electron spectroscopy and X-ray photoelectron spectroscopy- Peak intensity determination.*
- [4] ISO 21270: *Surface chemical analysis –X-ray photoelectron and Auger electron spectroscopies –Linearity of intensity scale.*
- [5] ISO 24236: *Surface chemical analysis –AES-Repeatability and constancy of intensity scale.*
- [6] D. Varga, Á. Kövér, L. Kövér, and L. Redler, *Nuc. Instrum. Methods in Phys. Res. A* **238**, 393 (1985).
- [7] K. Goto, H. Iwata, and Y. Sakai, *J. Vac. Soc. Jpn.*, **31**, 906 (1988).
- [8] K. Goto, N. Sakakibara, and Y. Sakai, *Microbeam Anal.* **2**, 123 (1993).
- [9] A. Alkafri, Y. Ichikawa, R. Shimizu, and K. Goto, *J. Surface Anal.* **14**, 2 (2007).
- [10] A. Alkafri, K. Goto, Y. Ichikawa, and R. Shimizu, *J. Surface Anal.* **14**, 95 (2007).