Legacy of Tetsu Sekine in AES

A. Mogami
JEOL Ltd., 3-1-2 Musashino Akishima, Tokyo 196-8558, Japan
mogami@jeol.co.jp

Received 31 January 2003

In January 31, 2002,Tetsu Sekine left the surface analysis world, his wife and many intimates, for the world of stars. Tetsu Sekine had a very good personality. No one would forget the working/ drinking time with him, and they may remember his vivid, honest, challengeable attitude toward his targets. In this paper, I would like to evoke Sekine's memory through some of his achievements at our company, JEOL, from the joining time to our AES team to the receiving the doctorate. His thesis was almost centered on the surface analytical developments, i.e. AES instrumentations, data-handlings, and micro-area/depth/ quantitative analyses. His activity in the subsequent days would be written by the other suitable persons.

BBM (BEAM BRIGHTNESS MODULATION) METHOD

It would be worth relating the story of the development of the BBM in some detail. Sekine joined-in our AES team in 1974 after one year training at one of production lines. It was just the time that JEOL announced the scanning AES JAMP-3, say α-version, as the first commercial model in the world. This instrument brought sub-μm-area analysis capability in AES[1].

We, the AES team, had a plan to promote the features of this instrument. Taking many market needs into considerations, we thought, that the analytical capability with the least primary electron beam current should be most important in AES. The smaller beam current intensity, the more easily we could realize the following significant features; (1) Avoidance of charge-up phenomena on insulator materials, (2) Elimination of elemental/chemical damages due to electron dose, and (3) Reduction of the diameter of primary electron beam for improvement of spatial resolution.

About the reduction of the beam current necessary to get spectrum, we discussed very deeply, and studied Seah's experiments and Le Gressus' suggestions. We had finally adopted a system capable of SN ratio enhancement on the spectra by the use of modulation technique, in which the primary electron beam was modulated in its intensity, and the detected signal was de-modulated by a lock-in amplifier to provide E*N(E). Moreover, this method made it possible to selectively obtain only the beam-induced Auger electron spectrum without any bad influences, for example, ion-induced electrons during ion sputtering. Such a modulation/de-modulation technique was well known as AM (amplitude modulation) in radio and telecommunication fields.

For designing of this method, we adopted the BBM, in which a small beam-chopping coil was put as the modulator just beneath the electron gun the outside of vacuum. So no re-designing was necessary for the high voltage supply as well as the lock-in amplifier system.

Now, the first job of Sekine was the assembling and functional testing of the BBM. Before starting this job, he had to learn whole of the instrument JAMP-3. He did it so eagerly. Using a piece of pure Ag plate as a standard specimen, he could confirm that (1) AES spectra could be obtained under the beam current of 1 nA with the low-pass filter (in lock-in amplifier) of 1 sec, (2) beam diameter was 50 nm under that condition, and (3) remained slight tail of the electron beam was caused by some hysteresis of chopper, which seemed to be smaller than the beam diameter[2, 3]. Then, Sekine applied this high spatial resolution instrument to industrial material analysis, such as testing of LSI, inclusion control in steels, characterizing of insulator catalyzers.

Here, I would like to add one point. Each member of our AES team was acting in those days as a "Jack-of-all-trades"; say, a sales-engineer, repairman, petit-scientist at conference halls, engineer making electronics/mechanical components in our dirty laboratory. Although each of them had one's own specialty and/or background, of course, every one had to be such a Jack-of-all-trades. If not so, our small team could never achieve its own functions. Sekine was really a bright Jack. No one could believe that Sekine had the master degree in theoretical-colored polymers science.

DATA HANDLINGS AND BBM STAGE-2

Next subject in the instrumentation was the computerization of the JAMP-3. Sekine strongly recommended himself to try the system developments. Data acquisition from the instrument to CPU, we thought, was a more urgent item to be developed than the machine control itself.

Firstly, he did compare the SN ratio on final AES spectrum data ($E*N(E)$ type) obtained by pulse counting to that obtained by the lock-in amplifier (analogues type). He found that the pulse-counting method had an unacceptable weak point, which was the fact that usable (maximum) primary electron beam current was limited to the range less than 10 nA in the pulse counting, which the spectra having equivalent SN ratio could be obtained by both the acquisition methods under the beam current of nA range. Those results were the fruits derived from co-operations with users. Based on the above fact, Sekine succeeded in designing the acquisition system so smoothly, and he got friends at the same time. A high speed VF converter was built-in to the JAMP-3 as an ITF, through which output data from the lock-in amplifier were transferred to the CPU system without any big modification in its main console. In this system, no limitation was existent in principle to the beam current range, that is, the full range of current of 1 nA~ 10 μA could be used for AES. Accordingly, the system provided a quick operation by high-speed energy sweep capability as well.

Further, Sekine proposed a new idea for data handling. This was the use of digital filter to provide a general energy-differential AES spectrum $dE*N(E)/dE$ from the acquired $E*N(E)$ spectrum data shown above. The digital filter technique was familiar at that time, mainly in quantitative analysis by solid-detector-type X-ray spectroscopy (EDS), where the X-ray spectra having broad peaks width and poor PB ratios. The filter-fit method was one of the most powerful tools to make quantitative EDS analysis with high accuracy by suppressing the background due to continuous X-rays as well as to improve peak separation.

He proposed to transplant the filter-fit technologies to AES system, where the spectrum has also a huge background due to inelastic scattered electrons. Also, he brought a special filter, Schamber's "Top-hat filter" having operational-ease. He confirmed that the Top-hat filter had following features; (1) $dE*N(E)/dE$ and $d^2E*N(E)/dE^2$ spectra could be obtained with the least increase of random noise accompanying with the differentiations, (2) good background rejection could be obtained over a wide energy range of spectrum, and (3) resultantly, good fitting could be made at quantitative calculation stages. Sekine applied the digital filter method to the quantitative AES analysis of TiC_xN_{1-x} (x=0, 0.3, 0.5, 0.7, and 1.0 by chemical analysis) compounds, with the use of pure TiC and TiN as the reference materials. Although Ti-MNNs and N-KLLs peaks overlapped each other and the peak separation was very hard, the results obtained by this filter-fit method showed a good agreements (a few % error) with by chemical analysis data[4].

Accordingly, the basic frame of instrument was established as one of commercial model. Sekine did contribute strongly to the development of the key functions of this instrument. After the early-stage CPU system where the total memory size was only 2 kB(not MB), the system was expanded rapidly in its size, year by year, with the continuous CPU advancements. In those periods, a small/charming system was also born under the collaboration with Geller. This system was just similar to the current EDS syatem.

We, products maker JEOL, have been responding to various needs from the science/industry fields. At the time of developing new additive functions, it is liable that the new components part will be designed one-by-one from zero! But Sekine and/or our team members usually kept in mind to make proper functions with the least change in the design of main instrument console. As descrived in the above, when we made the basic frame of instrument JAMP-3 on the base of its α-version, newly additive parts/components were so limited. Those were a beam-chopping coil for BBM, a VF-converter as ITF for data acquisition, and drivers. Anyhow we took such way. Even today the author likes such a designing philosophy. Important is to know neighbor's technologies.

MATRIX CORRECTION METHOD FOR QUANTI-

TATIVE AES ANALYSIS

In 1970s, many attempts were carried out for quantitative surface analysis by AES. One of the easiest ways was so-called relative sensitivity correction. Because the Auger electron peak varied in its intensity so widely among elements, the observed peak intensity of the object element in a sample had to be corrected with the relative sensitivity factor in order to obtain its elemental concentration (generally atomic %). Most people were never satisfied by such an elementary method. A new improved correction method was desired very strongly at that time in many fields.

Sekine proposed a new model for quantitative Auger analysis in 1983. The model was developed on an analogy with ZAF theory used in EPMA and was applied to general cases including alloys with non-linear matrix dependent characteristics. He made it under the guide of Hirata and Shimizu. The new model was consisted of three procedures; (1) A procedure to obtain the normalized Auger current intensity, (2) A procedure to correct for the matrix effects by the use of the correction function β, and (3) A procedure to calculate the value of β, which comprises atomic density, electron backscattering, and electron escape depth factors, and is a function of the concentration of constituent elements. The concentration is figured out by iterative calculation of procedures (2) and (3) carried out alternately until it approaches a certain constant value. The effectiveness of this model was confirmed by the quantification of Ag-Pd and Ni-Pd alloys. The results corrected by this method were close to chemical analysis values, although the chemical analysis data might not be adequate as a reference for surface analysis [5, 6].

In the same year (1982), JEOL published a "*Handbook of Auger Electron Spectroscopy*" for the purpose of quantitative AES analysis. Without help/encouragements from Shimizu and Sekine's hardwork, we could not carried out the publication. The Handbook represents the both type of Auger spectra ($E*N(E)$ and $dE*N(E)/dE$) over 50 elements, peak energy value table, correction factor tables, together with physically important contents [7, 8].

AFTER THE EARLY STAGE INSTRUMENTATIONS

VAMAS (Versailles Project on Advanced Materials and Standards) project started to make the standards of surface analyses in 1982. Sekine joined in the SC (scientific committee) as its member, where his sphere of activity would be expanded rapidly; he could also get many intimates.

In 1989, Sekine could get the doctorate at Osaka University, under the kind direction of Prof. Shimizu. The title of his doctoral thesis was: "Practice in Research of Surface Analyses on Auger Electron Spectroscopy"[9].

Sekine belonged to the AES development group until 1994, and he moved to the focused ion beam FIB group in the semiconductor development division. When he fell in ill in 2001, he was a manager bearing total responsibility of the group. As for Sekine and his achievements, you could find in this issue papers or essays written by the other suitable persons.

At the end, I would like to say that I was very happy to work together with Sekine for the first 10 years of the development of JAMP series. Thank you, Sekine-san. I would never forget you.

References

[1] A. Mogami, M. Hotta, and H. Hashimoto, *8th International Congress on Electron Microscopy, Canberra* **1**, 60 (1974).

[2] A. Mogami and T. Sekine, *6th European Congress on Electron Microscopy, Jerusalem*, 422 (1976).

[3] A. Mogami and T. Sekine, *9th International Congress on Electron Microscopy, Toronto* **1**, 6 (1978).

[4] T. Sekine and A. Mogami, *International Congress of Surface Science ICSS-4 and ECOSS-3, Paris*, 1283-1286 (1980).

[5] T. Sekine, K. Hirata, and A. Mogami, *Surf. Sci.* **125**, 565 (1983).

[6] T. Sekine and A. Mogami, *Surf. Interface Anal.* **7**, 289 (1985).

[7] T. Sekine, Y. Nagasawa, M. Kudoh, Y. Sakai, A.S. Parks, J.D. Geller, A. Mogami, and K. Hirata, *Handbook of Auger Electron Spectroscopy*, JEOL, Tokyo, (1982).

[8] T. Sekine, A. Mogami, M. Kudoh, and K. Hirata, *Vacuum* **34**, 631 (1984).

[9] T. Sekine, doctoral thesis, "Practice in Research of Surface Analyses on Auger Electron Spectroscopy", Osaka University (1989).