Reference Auger Electron Spectra with Work Function Corrected

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An iteration method in the energy calibration of AES has been developed by using elastically backscattered primary electrons with possibility of 15 meV (σ) for the energy range from 10 eV to 1200 eV, in which our improved novel prototype cylindrical mirror analyzer (CMA) has been used. For the true kinetic energy calibration, the relative work function of sample to CMA was investigated by changing sample bias to CMA at ground level. It can be obtained by observing the onset of secondary electrons (SE) since SE are believed to begin just above "0" of vacuum level, although it is hardly possible to obtain a true spectrum.

1. Introduction

We have been studying absolute Auger electron spectroscopy (AES) to make an AES Reference Data Base (DB). The DB should be absolute, i.e., SI traceable both in intensity and energy. For this purpose, a novel prototype cylindrical mirror analyzer (CMA) [1] of almost calculable ($\Delta E/E=0.25\%$), which is quite similar to the design of Varga et.al. [2], has been developed and is shown schematically in Fig.1. Some improvements have been made recently and with our improved novel cylindrical mirror analyzer the intensity has been obtained. The intensity was measured by an electrometer (Keithley model 642) that is SI traceable. While the energy calibration was quite uncertain, because there would exist no reference being available. employed the acceleration voltage for primary electrons as a reference and developed an iteration method [3], similar to Siegbahn [4] and Lassetre et.al. [5], which is the difference method and was employed to obtain a transfer coefficient of CMA. This is a self-consistent with automatic corrections thermionic emission and work functions of the cathode and CMA. The results should be referenced to the work function of the CMA. The energy calibration can be made by this iteration method with an accuracy of 15 meV (σ) for the energy range 10 eV though 1200 eV. Fig. 2 shows the obtained energy calibration, in which the sample positions were

optimized for Ep=50 eV, 500 eV, and 1000 eV with corresponding transfer coefficient Ks 1.49918, 1.49688 and 1.49533, respectively. This fact represents the fairly large immunity of the transfer coefficient for CMA. The characteristics rapidly dropped for the energies higher than 1200 eV can be explained by the increased overlap of the spectra due to the poor energy resolution of CMA for the spectra. By introducing correction of the convolution, which was simulated using an experimental spectrum [3]. It seems that the possibility of energy calibration is up to 3keV.

Although the iteration method gives us satisfactory results for the energy calibration, it offers the energy being referenced to the vacuum level of CMA provided the work function of the whole CMA is to be uniform. If work function of CMA coated with soot/carbon and that of sample are different, then there will

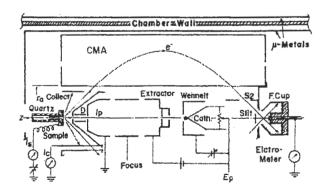


Fig. 1. The schematic picture of our CMA.

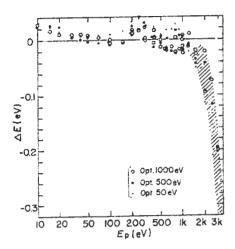


Fig. 2. The energy calibration by the iteration method.

be an ambiguous value. It is a quite common problem in AES and in other energy analysis as well. Thus the relative work function of sample to CMA needs to be known for a final kinetic energy calibration. Here, we will describe that the relative work function of sample to CMA can be examined and corrected by changing the sample bias.

2. Experiments

The experiments were carried out in our CMA, Fig. 1, of which the electron gun, the sample holder and the slit have been improved recently.

The electron gun with a special tungsten cathode of reduced magnetic field and unipotential property was operated with a constant emission current with an extractor (~80V). This mode would present well-defined characteristics of primary electrons. Thus energy distribution of primary electrons, i.e., the shape and the peak position, would be kept constant irrespective of the acceleration voltage of primary electrons. The quartz sample holder would keep sample position within a few micrometers. An extra baffle slit (S2) was set between the slit and the exit mesh of our CMA to cut down extraneous electrons after the Monte Carlo Simulation [6] and the reduced ghost spectrum of less than 104 was obtained [7]. To reduce the residual magnetic field the whole CMA is enclosed with dual μ -metals. The Wehnelt is made of μ -metal and the wiring of the cathode is twisted to decrease and cancel the generated magnetic field. The CMA

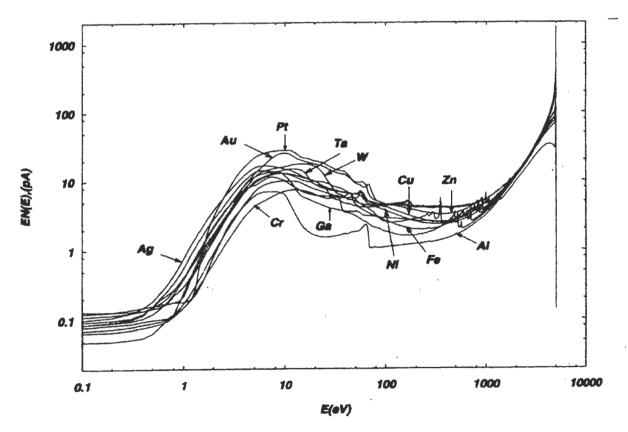


Fig.3. Typical spectra in "Advanced Sickafus" plot (log-log).

is coated with soot and/or aquadag to reduce the electron scattering and to obtain a stable and uniform work function.

To make effects of work function clearly, the relationship between the onset of secondary electrons and the sample bias voltage was investigated. The triaxial and guard method to apply the bias was employed. The sample used here was polycrystalline Mn.

3. Results and Discussion

A log-log plot that covers the whole range of energy distribution including Auger spectra in one sheet is shown in Fig. 3. We call it as "advanced Sickafus plot" [8] and it can be used to detect and compare the spectra. This plot is quite convenient in general use. It should be noted that the spectra ranging $0.1\sim10~{\rm eV}$ showed the work function difference, interband transitions, and structures due to the plasmon excitation [9] in the true secondary electron range easily.

Several typical spectra for primary electron energies 5 keV without work function corrected are shown in the advanced Sickafus plot in Fig. 3: C/graphite (~5), Al (4.3), Cu (4.7), Ag (4.3), Pt (5.7) and so on, the work functions in parentheses are in eV [10]. The almost flat characteristics in the range 0.1-1 eV are consisted of the rubbish electrons in CMA [1]. One of the reasons that the curves are not coincident in the onset of the true SE is due to the work function difference. Particularly, the sudden deflection observed in Pt just above 1 eV is caused by the acceleration of SE by the work function difference. It is because relative work function of the sample Pt is the largest value to the CMA.

By changing sample bias (Vs) from -1.0 V to 1.0 V in every 0.1 V at Ep=50 V, we obtained the energy distribution of SE. It is shown in Fig. 4. Fig. 4 reveals the relationship between the onset of SE and the sample bias voltage. In the bottom section, with the sample bias decreasing from 1.0 V to -1.0 V, the EN(E) simultaneously becomes less at the same kinetic energy. While in the top section it is contrary. It is because the energy distribution of true SE was artificially shifted with respect to the CMA. Thus deceleration and acceleration could occur and intermediate stage should exist between the two extremes. The intermediate stage would necessarily be the stage of the "0" of the work

function difference. We urged to find this "0-stage". In the intersection, for all sample bias voltages the all characteristics seem to cross at the EN(E) 1.83 pA and the kinetic energy about 3.35 eV.

To find the "0-stage", the bottom of Fig. 4, the almost the onset parts of SE are displayed in Fig. 5. As can be seen in Fig. 5, energy distributions faded into the background (BG) of CMA [7]. The characteristics begin to concentrate at Vs of about -0.1~-0.2 V, i.e., the border between the modes of acceleration (coarse) and the cut off (fine) for the SE by the retarding potentials. The fine area in the left part of the characteristics shows that relative work function (RWF) between CMA and sample Mn is plus, so the electrons can be detected with retardation. While the coarse area in the right part of the characteristics shows RWF between CMA and sample Mn is minus. Thus the critical border would exist which means RWF to be zero "0-stage". This critical

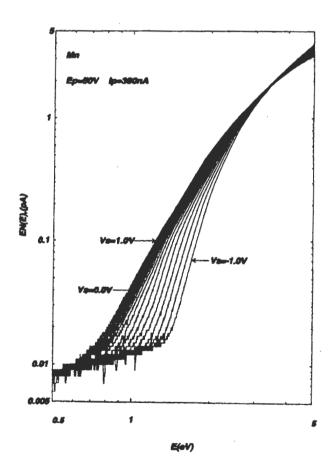


Fig. 4. Spectra of onset of SE as a function of sample bias.

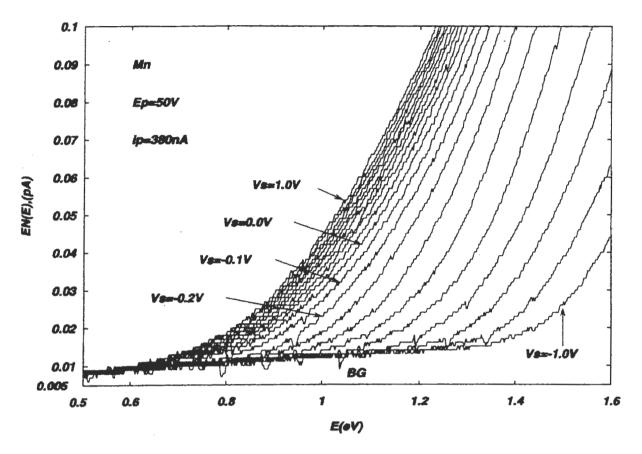


Fig. 5. Spectra of SE as a function of sample bias; lower section of Fig. 4.

voltage should correspond to RWF of sample Mn to CMA (~5eV: carbon) being null and can be used to correct work function difference. We appreciated the correction can be vicinity of -0.1 to -0.2 V. This method is quite self-consistent associated with the iteration method [3]. The latter is also a self-consistent method.4. Summary

The RWF of sample to CMA was examined by observing the onset of SE, since SE has been believed to start just above the "zero" eV of vacuum level, but it is hardly to obtain a true spectrum. It can be possible only with a well-defined system and careful experiments. A method of changing sample bias was introduced to observe the energy distribution of SE in low energy and the correction voltage for RWF can be obtained. From these results we conclude that RWF can be corrected by adjusting a sample bias. Experiments are continued further and the final kinetic energy calibration will be expected.

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6. References

- [1] K. Goto, N. Sakakibara, and Y. Sak Microbeam Anal. 2, 123 (1993).
- [2] D. Varga, A, Kövér, L. Kövér, and Redler, Nuc. Instrum. Meth. Phys. R A238, 393 (1985).
- [3] K. Goto, N. Nissa. Rahman, Y. Z. Jiang, Asano, and R. Shimizu, Surf. Interfa Anal., (to be published).
- [4] K. Siegbahn, Ark, Mat. Astr. Fys. 3 (No.20), (1944).
- [5] E. N. Lassettre, A. Skerbele, M. A. Dille and K. J. Ross, J. Chem. Phys. 48, 50 (1968).

- [6] M. M. El Gomati and T. A. El Bakush, Surf. Interface Anal. 24, 152 (1996).
- [7] K. Goto, N. Nissa Rahman, and R. Shimizu, 3rd Korean-Japan International Symposium on Surface Analysis (Kyong Ju, Korea, Nov. 2-3, 2000).
- [8] Y. Z. Jiang, K. Goto, R. Shimizu, J. Surf. Anal. 8, 147 (2001).
- [9] F. Pellerin, et.al, Surf. Sci. 103, 510 (1981).
- [10] R. W. Strayer, W. Mackie and L. W. Swanson, Surf. Sci., 34, 225 (1973).