Auger electron emission from metals under gallium focused ion beam bombardment

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(Received October 12 1998; accepted January 18 1999)

When light elements, *e.g.*, Mg and Al, are bombarded by energetic ions, Auger electrons are emitted from their surface. Ion-induced inner-shell excitations, which result in Auger emission, are generally explained by the formation of instantaneous quasi-molecules consisting of a target atom and a projectile or target atom. Through crossing of the molecular orbitals, an inner-shell electron is promoted to a higher orbital and leaves an inner-shell vacancy after they separate. An ion-induced Auger electron (IAE) spectrum comprises a continuous background due to the decay of excited atoms or ions under the target surface (bulk-like peak) and discrete sharp peaks from excited species which are sputtered into vacuum (atomic-like peak) during ion bombardment.

In order to evaluate the applicability of IAE to practical surface microanalysis, we studied gallium-focused-ion-beam-induced IAE (Ga FIB IAE) emission from a few metals. Ga-FIB-induced main Al_{LMM} atomic-like peak showed higher Auger yield and had much higher signal-to-background (S/B) ratio than its EB induced Al_{LVV} peak. The IAE yields decreased with increasing atomic number of the target atoms. By comparing the intensity of Ga FIB IAE from Al to the EAE one, the sensitivity of atomic-like Al_{LMM} emission per Ga ion was evaluated.

1. Introduction

Impact of accelerated heavy ions on solids of light elements, *e.g.*, Mg, Al, gives rise to electron spectra with sharp atomic-like features on continuous backgrounds. The continuum is related to the decays of excited atoms or ions in the solid while the sharp line comes from decays of sputtered atoms or ions in the vacuum. Ion-induced Auger electron (IAE) was first observed in 1965 by Snoeck *et al.* [1] for Ar^+ impact on Cu. After that, most of the works intended to clarify the difference between IAE and the electron-induced Auger electron (EAE) emission. It is now generally accepted that the inner shell excitations in IAE emission originate from violent binary atomic collisions, which promote core electrons through crossing of quasi-molecular orbitals formed by projectiles and target atoms. The atomic-like peaks on IAE spectra have much higher signal-to-background (S/B) ratios than the corresponding EAE peaks. The peak widths are typically a few eV and much narrower than those of the EAE peaks. The dependence of IAE intensities and the S/B ratios of atomic-like peaks on projectile energy E_p has also been studied. It has been found that the S/B ratio increases linearly with E_p when E_p is greater than 5 keV [2]. Behaviors of IAE emission from some alloys and compounds have also been studied in an effort to correlate the IAE intensities to the surface compositions [3,4]. However, surface analysis by means of IAE spectroscopy (IAES) is yet to be developed for practical use. It is because not only many fundamental aspects of IAE emission concerning to the quantification of IAES, *e.g.*, the matrix effect of IAE emission of an element under different combinations with other elements, have not been elucidated, but also the extension of its applicability, although IAE emission from many elements have been reported [5]. By now only IAE from limited elements have been intensively studied and intense atomic-like emission from heavy elements has not been observed [6]. Furthermore, noble gas ions have been used in most IAE studies. Such ion beams can not be finely focused, which restricts its application in surface microanalysis of solid materials. However, as light elements play an important role in determining properties of materials and are extensively used, it is still of significance to study the applicability of IAES to surface microanalysis.

Recently gallium focused ion beam (Ga

FIB) technology has been extensively applied both in surface analysis and microprocessing of solid materials. Since Ga FIB can be easily focused to less than $0.1\ \mu m\phi$ with current density of a few A/cm^2, it is applied, for example, to secondary ion mass spectrometry (SIMS) to perform two-dimensional surface elemental analysis with sub-μm lateral resolution [7].

The purpose of this study is to combine the characteristics of IAE emission and Ga FIB mentioned above to develop a novel surface analysis method with sub-μm lateral resolution. Intense IAE emission from Al and Si has been studied using Ga FIB bombardment by our group [8,9]. Very sharp LMM atomic-like peaks with much lower background than the LVV EAE peaks have been observed. From these atomic-like peaks, it is expected to perform high contrast two-dimensional elemental mapping with sub-μm lateral resolution. The authors will present another paper on the elemental mapping over an integrated circuit (IC) surface using Ga FIB IAE in this volume. In this paper, Ga FIB IAE emission from Al, Si and Ti, which are extensively used in semiconductor industry, is studied. The dependence of the IAE intensity on atomic number is discussed. The L-shell electron excitation probability of Al under 20 keV Ga FIB bombardment is also evaluated.

2. Experimental

Si (110) and polycrystalline Al, Ti were used in the experiment. Ga FIB was accelerated to 20 keV with a beam current of 1 nA. The incident angle of the Ga FIB was 60 degrees from the surface normal. In order to calculate the L-shell excitation probability of Al, spectra of Ga FIB IAE and EAE in normal incidence were also obtained. The electron beam (EB) was accelerated to 4 keV with a current of 1 nA. The spectra were recorded in pulse counting mode using a cylindrical mirror analyzer (CMA). The CMA is coaxial to the EB. The energy resolution of the CMA was set to 1.2%. Details on the apparatus can be found elsewhere [8]. Pre-sputtering using Ga FIB has been carried out on all samples before the experiments.

3. Results and Discussion

In Figs. 1-3, $E \times N(E)$ (solid lines) and numerically differentiated $d[E \times N(E)]/dE$ (dashed lines) spectra of Ga FIB IAE from Al, Si and Ti, respectively, are shown. In Fig. 1 and Fig. 2, very sharp atomic-like Al$_{LMM}$ and Si$_{LMM}$ peaks can be observed on their $E \times N(E)$ spectra. All of the numbered peaks result from Al$_{LMM}$ and Si$_{LMM}$ transitions of sputtered species in the vacuum. Their proposed identifications can be found in our former papers [8,9] and elsewhere [10]. The atomic-like peak from Ti can also be observed at 26 eV in Fig. 3. We ascribe it to the Ti$_{MNN}$ transition according to its kinetic energy. There is a very broad peak between 40-200 eV in

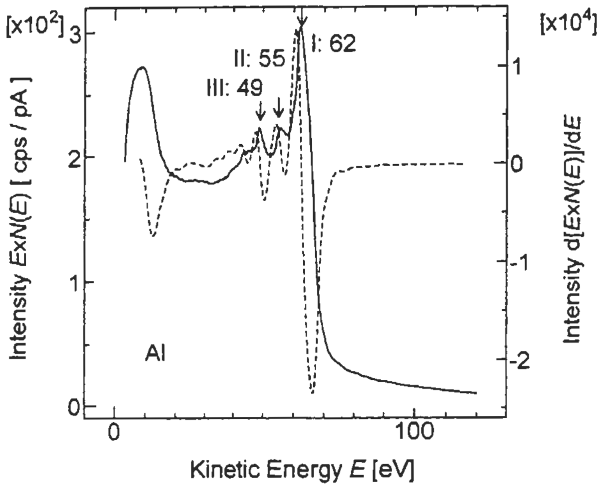

Figure 1. Ga FIB IAE spectra from Al. The atomic-like Al$_{LMM}$ peaks are indicated with Roman numbers.

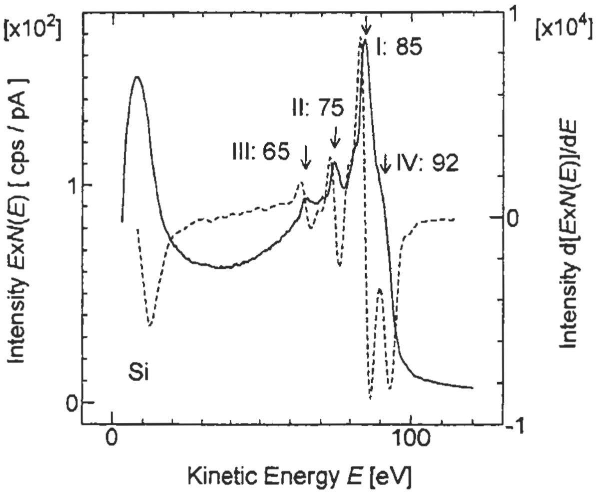

Figure 2. Ga FIB IAE spectra from Si. The atomic-like Si$_{LMM}$ peaks are indicated with Roman numbers.

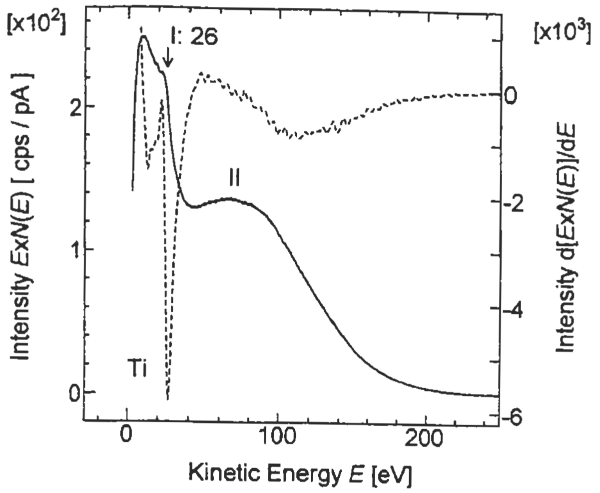

Figure 3. Ga FIB IAE spectra from Ti. The atomic-like Ti$_{MNN}$ peaks are indicated with Roman numbers.

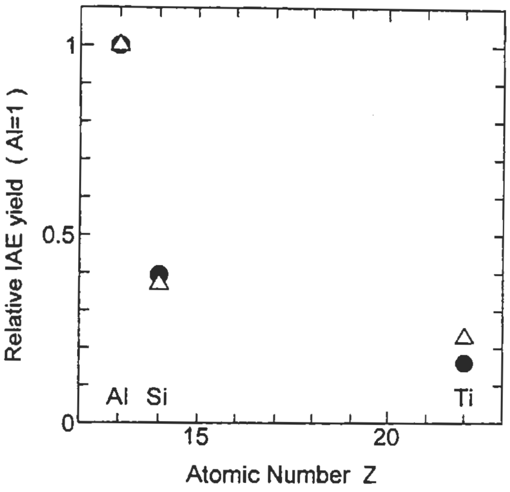

Figure 4. Relative intensity of Ga FIB IAE for Al, Si and Ti.
●: intensity from *N(E)* spectra.
△: p-p intensity from *dN(E)/dE* spectra.

Fig. 3. Since only Ti and Ga existed on the surface and there are no possible Auger transitions energetically for Ti in this energy range, we suppose it come from Auger emission of Ga species and/or quasi-molecules formed during the Ga FIB bombardment. Exact assignment requires further investigation.

Figure 4 shows the relative IAE intensities versus atomic number Z (LMM for Al and Si, MNN for Ti.). The $N(E)$ and $dN(E)/dE$ intensities are used in Fig. 4 and normalized to those of Al. Open triangles show the peak-to-peak (p-p) intensities on the $dN(E)/dE$ spectra of the prominent atomic-like peaks (peak I's in Figs. 1-3). The solid circle represents the sum of the atomic-like and bulk-like intensities on the $N(E)$ spectrum obtained by subtracting true secondary electron intensity using a method by Sickafus [11]. From Figs. 1-3, it can be found that the widths of atomic-like peaks are nearly the same and are much narrower than the corresponding EAE peaks (Fig. 5). The broad bulk-like component is considered to have the similar line shape to the corresponding EAE peak [12]. Therefore, we can assume that the p-p intensities in Fig. 4 approximately represent the relative IAE yield of peak I's in Figs. 1-3. Since the spectra were obtained in the same conditions, Fig. 4 also shows the dependence of inner-shell excitation probability (P) on Z. The $dN(E)/dE$ data show that P decreases with increasing Z monotonously. It can be explained by theoretical calculation of Coulomb ionization based on binary encounter approximation [13]. The calculation also shows that P for M-shell electrons is higher than that for L-shell electrons for the same element. Barat *et al.* said the level-matching effects work in the inner-shell electron excitation through asymmetric atomic collisions, *i.e.*, inner-shell electron promotion occurs when an energy level of an atom matches that of another[14]. However, among the three elements in our experiment, the binding energy of L-shell electrons of Si matches that of M-shell electrons of Ga most. This shows that mechanisms other than level-matching effects (*e.g.*, Coulomb interaction) also affect P in case of Ga$^+$ bombardment. The dependence of $N(E)$ intensity (solid circle) on Z for Al, Si and Ti coincides well to that obtained from *dN(E)/dE* spectra. Since the existence of peak II in Fig. 3, the intensity of the true secondary electron was over subtracted in extracting IAE intensity of Ti (peak I in Fig. 3.). The real magnitude should be a bit larger than the depicted one in Fig. 4.

In the next, the sensitivity of atomic-like Al$_{LMM}$ emission per Ga ion was also evaluated from the experimental results. Considering the mechanism of atomic-like IAE emission, we express its detected intensity as:

$$I_{i,IAE} = X_i n S P_{at} \omega_A \times B \times T(E_A) D(E_A) I_p . \quad (1)$$

Meanwhile, the detected intensity of EAE is generally expressed as:

$$I_{i,EAE} = X_i n \sigma_i (E_p, E_c) \omega'_A \times R_i (E_p, E_c, \theta) \\ \times \lambda_i (E'_A) B \times T(E'_A) D(E'_A) I'_p , \quad (2)$$

where X_i and n are atomic concentration of element i and atomic density of the sample, respectively, S the sputter yield of the sample, P_{at} the number of inner-shell excited atoms (ions) sputtered into the vacuum per incident ion, *i.e.*, the sensitivity of atomic-like $\mathrm{Al_{LMM}}$ emission per Ga ion. ω_A and ω_A' the probabilities for Auger transitions, B the parameter related to the roughness of sample surface, T and D the transmittance of CMA and efficiency of the detector, respectively, σ_i the cross section for inner-shell electron excitation, R_i the backscattering correction factor, λ_i the inelastic mean free path of the electron in the solid, I_p and I_p', the intensities of Ga FIB and EB, respectively. As the $\mathrm{Al_{LVV}}$ and $\mathrm{Al_{LMM}}$ electrons have similar kinetic energies, it is reasonable to consider the instrument factors T and D in Eq. (1) and (2) as the same. For pure Al, we get the following equation from Eq. (1) and (2):

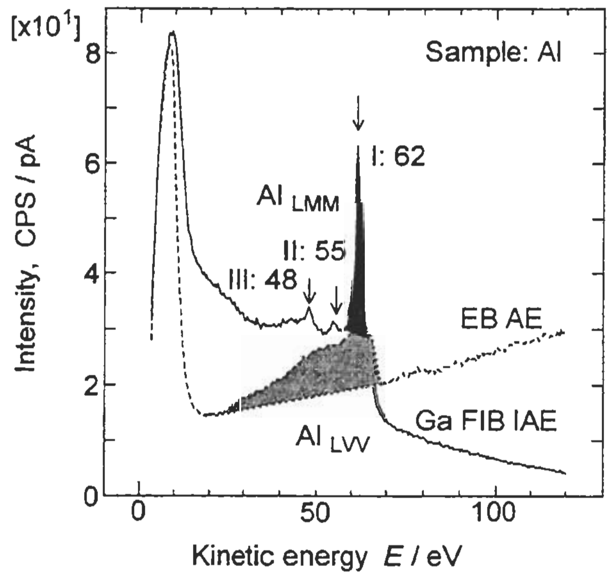

Figure 5. Calculation of L-shell electron excitation probability of Al under 20 keV Ga FIB bombardment.

$$P_{at} = \frac{\sigma_i \times R_i \times \lambda_i}{S \times \dfrac{I_{i,EAE}}{I_{i,IAE}}}. \qquad (3)$$

In consideration of the setting of our apparatus, we measured the IAE and EAE spectra at normal incidence, calculated the intensities of $\mathrm{Al_{LVV}}$ peak and the prominent $\mathrm{Al_{LMM}}$ peak (peak I) in Fig. 5, and evaluated the P_{at} for peak I with Eq. (3). The result was 4×10^{-3} atoms/$\mathrm{Ga^+}$. Precise result can be obtained if exact curve fitting is performed.

4. Conclusions

Ga FIB IAE emission from Al, Si and Ti was studied. Intense atomic-like Auger emission was observed at 62 eV, 85 eV and 26 eV, respectively. It was shown that the relative IAE intensity or yield decreases with atomic number. The sensitivity of atomic-like $\mathrm{Al_{LMM}}$ emission per Ga ion corresponding to peak I was evaluated to be 4×10^{-3} for Al from the experiment results.

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