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**INTENSITY OF VALENCE AUGER TRANSITIONS ( $L_{23}VV$ )  
OF Al AND Si IN METAL, OXIDE AND NITRIDE**

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Abstract

$L_{23}VV$  Auger transition has been studied in Si,  $SiO_2$ , Al, AlN,  $Al_2O_3$  by electron spectroscopy excited either by electron beam or X Rays. A strong difference is observed in intensity between pure solid and oxide or nitride under electron bombardment. Auger intensity is very sensitive to changes in the back-scattering coefficient or inelastic mean free path. However transient local trapping of electrons seems to be responsible for the large change observed.

Key Words: Valence Auger transition, electron trapping, transition rate, X-ray photoemission.

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Introduction

Valence Auger transitions involve outermost electrons and, therefore, are sensitive to the solid state environment. The Auger lineshape is related to the density of states in the valence band and then is quite different in the metal as compared to the oxide. The line intensity can be calculated using atomic Auger matrix elements obtained from Hartree Fock Slater calculations. Agreement with measurements is only fairly poor. When one comes to the case of oxides, the agreement is much worse. However, comparison between theoretical and experimental results is hampered by solid effects which occur after Auger electron emission and decrease the intensity detected outside the solid.

This paper deals with such effects. Intensities of  $L_{23}VV$  line of Al and Si were studied on Al, AlN,  $Al_2O_3$ , Si and  $SiO_2$  samples. An important decrease of the line is observed upon oxidation and nitridation. Comparison between electron beam or X-ray excitation emphasizes the role of solid effects. They could correspond to the creation of local electric fields triggered by the trapping of electrons into the lattice defects. These results must be taken into account for quantitative surface analysis by Auger electron spectroscopy.

Experimental

Method

Auger electron spectra were recorded using different electron spectrometers: cylindrical mirror analyser in JEOL Jamp 10 and PHI scanning Auger microscope, hemispherical analyser in V.G. Escalab Mark II. The base pressure was around  $10^{-10}$  Torr in the microscopes and  $5 \cdot 10^{-11}$  Torr in Escalab. Detection was made by counting method. Data could be stored and processed using either an Inter technique

IN96 computer (Jamp 10 and Escalab) or a PDP-DEC (PHI).LVV Auger line of aluminum and silicon were recorded either in constant analyser energy mode (hemispherical analyser) or in constant energy resolution mode (cylindrical mirror analyser). When using electron beam excitation, the primary accelerating voltage could be varied between 100 eV and 10 keV. Incidence and detection angles were different in Jamp 10, PHI and Escalab. For X-ray excitation the Al K $\alpha$  (1486 eV) and Zr L $\alpha$  (2042 eV) were employed.

Electron energy loss spectroscopy was also performed in Escalab in order to check the cleanliness of the samples and to obtain information about the presence of defects.

#### Samples

Surfaces of pure silicon and aluminum were prepared by heating and ion etching. They were considered clean when the surface plasmon peak in the energy loss spectrum was as intense as the bulk one. At this stage, no carbon or oxygen Auger peak could be detected.

Aluminum nitride was produced by nitrogen implantation in the preparation chamber of the Escalab. 10 keV nitrogen ions triggered a 200 Å AlN layer. XPS spectrum showed that a pure aluminum nitride thin film was obtained as only N and Al photoelectron lines could be detected.

Aluminum and silicon oxide were obtained by exposing the clean samples to oxygen pressures greater than 5000 L (1 L =  $10^{-6}$  Torr.s). This treatment leads to an oxide layer thickness smaller than 20 Å (Fontaine et al., 1982). In order to compare results obtained on thin oxide layer with those obtained on bulk oxide, pure amorphous SiO $_2$  and crystalline Al $_2$ O $_3$  were studied. Their surfaces were also cleaned by heating and ion etching.

#### Results

##### Electron Beam Excitation

Figure 1 shows the L $_{23}$ VV Auger peak recorded on Si, SiO $_2$ , Al, Al $_2$ O $_3$  with a cylindrical mirror analyser. As the analyser resolution is constant, the curves represent the E.N(E) distribution. Accelerating voltage was 5 keV for Al and 10 keV for Si. The oxide energy position is not shifted with respect to the theoretical value and previous experimental results [Citrin et al., 1976; Madden and Goodman, 1985].

Figure 2 represents the L $_{23}$ VV Auger peak obtained on Al, Al $_2$ O $_3$  and AlN with the hemispherical analyser at 250 eV and 3 keV primary energy. In this case the N(E) curve is displayed owing to the

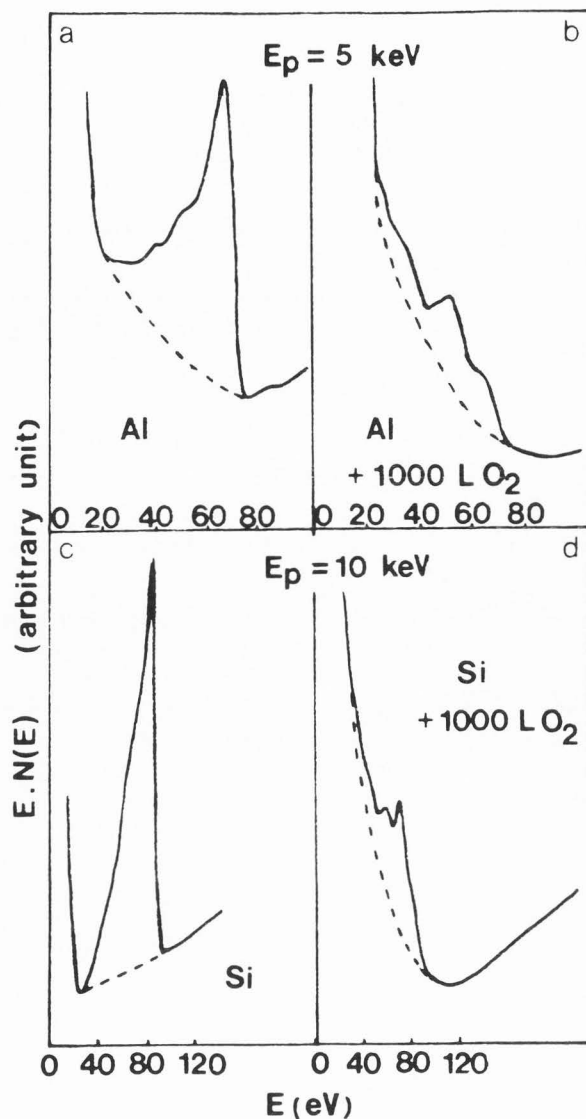


Fig. 1. Shape of the L $_{23}$ VV Auger transition recorded with a CMA, in the E.N(E) mode on (a) Al; (b) Al $_2$ O $_3$ ; (c) Si; (d) SiO $_2$ .  $I_p \approx 10^{-8}$  A.

constant analyser energy. The experimental conditions (excitation and detection) were identical for Al, Al $_2$ O $_3$  and AlN. In order to normalize our results, we used the ratio of the peak over background. As previously pointed out by Janssen et al. (1977), and Langeron et al. (1985), this quantity does not vary with experimental conditions, such as beam intensity, detection angle, electron multiplier yield, etc... The Auger peak being superimposed on a steep sloping background, this later was fitted either by extrapolating the

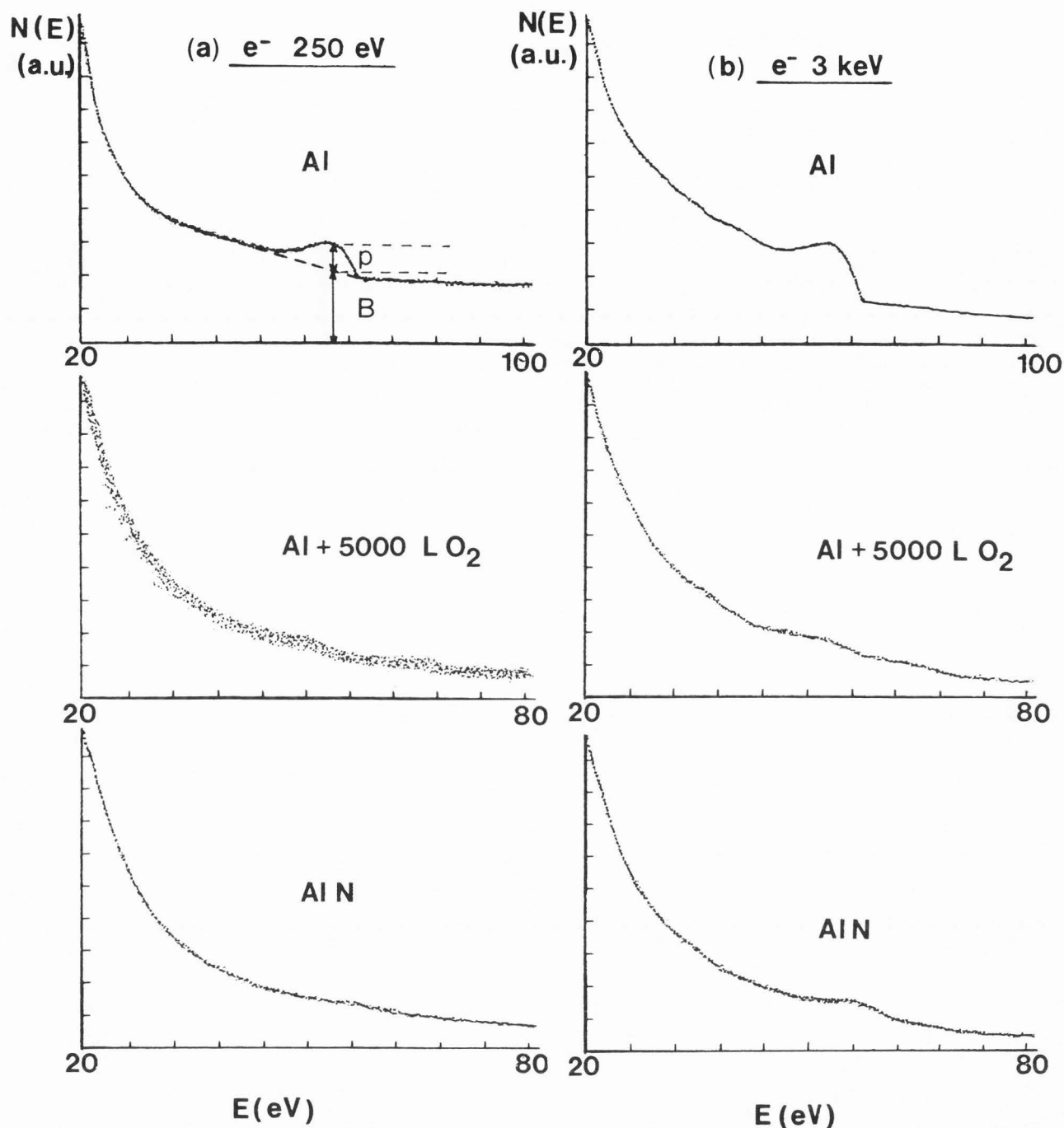


Fig. 2. Shape of the  $L_{23}VV$  Auger line recorded on Al, Al+5000L O<sub>2</sub> and AlN with a hemispherical analyser, in the N(E) mode under  $e^-$  bombardment: (a)  $E_p = 250$  eV; (b)  $E_p = 3$  keV.  $I_p \approx 10^{-9}$  A. Also displayed is the peak over background measurement.

background under the peak (fig. 1), or by a straight line drawn between the high and low energy side of the peak (fig. 2). The peak height was taken between the maximum and the background at the maximum energy. The results are plotted in Table 1 for figure 1, and in Table 2 for figure

2. They show an important decrease in the Auger intensity between pure solid (Al or Si) and the corresponding oxide (Al<sub>2</sub>O<sub>3</sub> or SiO<sub>2</sub>) or nitride (AlN). On figure 2, the Auger peak appears at 64 eV for Al, which is consistent with the results of Citrin et al. (1976) and

Al	Al+1000 LO <sub>2</sub>	Si	Si+1000 LO <sub>2</sub>
0.9	0.24	0.85	0.20

Table 1. Intensity of Auger L<sub>23</sub>VV line in Al, Al+1000L O<sub>2</sub>, Si and Si+1000L O<sub>2</sub> measured as the ratio of the peak over background on figure 1.

Source	Al		Al + 5000L O <sub>2</sub>		AlN	
	$\frac{P}{B}$	E <sub>a</sub>	$\frac{P}{B}$	E <sub>a</sub>	$\frac{P}{B}$	E <sub>a</sub>
e <sup>-</sup> 250 eV	0.4	64	<0.05	-	<0.05	-
e <sup>-</sup> 3 keV	0.8	64	0.15	52	0.24	56
X AlK $\alpha$	1	64	1.5	50	1.6	57
X ZrL $\alpha$	0.7	64	<0.05	-	<0.2	57

Table 2. Intensity of Auger L<sub>23</sub>VV lines in pure Al, oxide (Al+5000L O<sub>2</sub>) and nitride AlN (measured as the ratio of the peak of background  $\frac{P}{B}$ ), under different excitations: electron bombardment (E<sub>p</sub> = 250 eV and 3 keV) and X-ray irradiation (Al K $\alpha$  and Zr L $\alpha$  lines). E<sub>a</sub> is the energy of the Auger peak (eV), referred to the vacuum level (the work function of the analyzer is  $\phi = 4.3$  eV).

Madden and Goodman (1985). In the case of Al<sub>2</sub>O<sub>3</sub>, a slight difference is observed, compared with previous results. They indicate a 2 eV negative charge at 3 keV, whereas the peak recorded at 250 eV is too weak to be located accurately.

#### X-ray Excitation

With X-ray excitation the normalization problem is very stringent. Indeed measurement of the primary beam is not straightforward. This leads us to take the ratio peak over background to compare the results. The most striking behavior which appears in Table 2, is the strong difference between Al K $\alpha$  and Zr L $\alpha$  excitation. While the values are roughly the same under Al K $\alpha$  X-ray excitation for Al, AlN and Al<sub>2</sub>O<sub>3</sub>, an important decrease ( $\sim 10$ ) is observed under Zr L $\alpha$  X-ray excitation when comparing Al and Al<sub>2</sub>O<sub>3</sub> (figure 3). It must be noted that the ratio  $\frac{p}{B}$  is equivalent under Al K $\alpha$  or Zr L $\alpha$  X-ray excitation for clean Al. No shift of the Auger peaks was observed under Al K $\alpha$  X-ray excitation. In the case of Zr L $\alpha$  source, the signal was too

weak to locate exactly the position of the maximum. The same behavior was observed on SiO<sub>2</sub>.

#### Discussion

Recently Turner and Ramaker (1983) invoked a final state shake off mechanism to explain intensity variation in S<sup>-</sup>, SO<sub>4</sub><sup>-</sup> and Si, SiO<sub>2</sub>. However our experimental results show a quite different behavior under electron or X-ray excitation, which cannot be accounted for by this model. Several other factors can be adduced to explain the striking differences observed on the Al L<sub>23</sub>VV intensity depending on its atomic environment: variation of Auger intensity parameters like backscattering coefficient R (under electron excitation) inelastic mean free path  $\lambda$ , or atomic density n; variation of the Auger yield in compounds like Al<sub>2</sub>O<sub>3</sub>, AlN, SiO<sub>2</sub>; modification of the wave vector of the Auger electron due to local field created by the primary beam.

#### Auger Intensity Parameters

In electron excited Auger spectroscopy, the line intensity is proportional to several factors, which can change when going from pure solid to the oxide:

- The backscattering coefficient, R, takes into account the inner shell ionization by the backscattered electrons. Backscattering factors have been extensively calculated by Shimizu and Ichimura (1981), using Monte Carlo calculations. According to their data,  $R \approx 1.6$  for Al<sub>2</sub>O<sub>3</sub> and  $R \approx 1.7$  for Al, with a 3 keV electron beam and an incidence angle of 30°.

- On another hand, the Al atoms density (in atoms per cm<sup>3</sup>) varies by a factor of 6 between Al and Al<sub>2</sub>O<sub>3</sub>.

- The inelastic mean free path  $\lambda$  has been measured and calculated in Al metal and oxide (Tung et al., 1977). It is around two times larger in the oxide (7 Å) than in the pure metal (3.5 Å).

Then R, n and  $\lambda$  will decrease the Auger intensity by a factor 3 when going from the pure metal Al to the oxide Al<sub>2</sub>O<sub>3</sub>, which is less than the observed ratio under an electron beam. Furthermore, under X-ray excitation, the intensity behaves quite differently.

#### Interatomic Auger Transitions

Experimental data and band calculations emphasize that, in silicon and aluminum oxide, the valence electrons are localized on the oxygen atoms [Batra, 1982]. The degree of localization depends strongly on the ionicity of bonding.

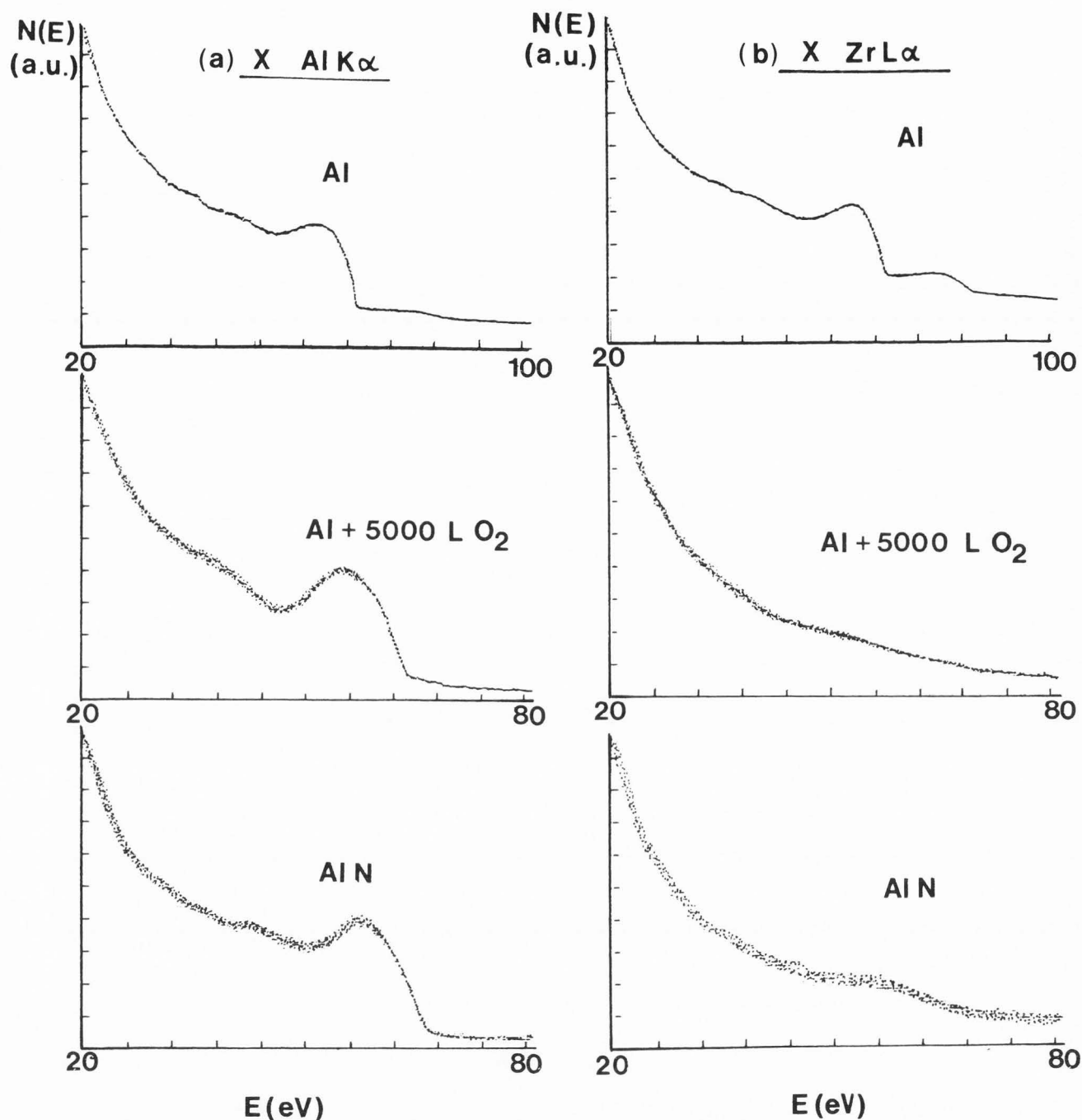


Fig. 3. Shape of the  $L_{23}VV$  Auger line recorded on Al, Al+5000L  $O_2$  and AlN with a hemispherical analyser, in the  $N(E)$  mode under X-ray irradiation: (a) with Al  $K\alpha$  line:  $\hbar\omega = 1486.6$  eV; (b) Zr  $L\alpha$  line:  $\hbar\omega = 2042.4$  eV.

Cluster calculations showed that non-bonding  $O(2p)$  and  $O-Al$  hybridized levels were in closer proximity than for  $Si-O$  and pointed out that the bonding in alumina is more ionic than in quartz. The holes created in  $L_{23}$  levels can only de-excite through an interatomic Auger process. As the transition rate is governed

by the overlapping of hole and electron wave functions, a difference can be predicted. Transition rates for interatomic Auger processes have been calculated by Matthew and Komninos (1975), in the case of  $MgO$ , and by Yafet and Watson (1977), for  $NaCl$ . In the case of the de-exciting on the  $2p$  hole the authors calculated

that in Mg, metal and interatomic rate were in close values:  $1.10^{-5}$  [ $Mg_{2p} \rightarrow Mg_{3s} O_{2s}$ ] versus  $1.6 10^{-5}$  for Mg  $L_{23}VV$ . Al-O interatomic transition has been extensively studied by Citrin et al. 1976, but no attempt has been made to calculate its intensity. There exists no data for AlN. The same problem arises with Si and  $SiO_2$ . Anyhow metal and oxide behaviors seem not to be due to an interatomic Auger process [Bonnelle, private communication] owing to the great intensity difference which is observed. Furthermore in this frame the changes observed under different sources of excitation could not be accounted for.

#### Charge Build-up under Irradiation

In other publications [Vigouroux et al., 1984; Vigouroux et al., 1985], we studied charge build-up in  $SiO_2$  and  $Al_2O_3$  under irradiation. This corresponds to the trapping of holes or electrons into levels localized in the band gap arising from impurities or structural defects preexisting or produced in the materials under irradiation. Particularly, we have shown that, in  $SiO_2$ , a close Frenkel pair could be created under electronic excitation and stabilized either by reionization or by trapping of negative charge, leading to an  $E'$  center precursor state, which is a somewhat strained bond between two adjacent silicon atoms at the site of an oxygen vacancy. This defect can act as an electron trapping center. The charging process changes the local potential and field distribution which in turn can affect the number of emitted electrons as well as their energy and angular distribution. Upon heating at temperatures greater than  $300^\circ C$ , the point defects are annealed, leading to a detrapping of the charges.

On the opposite,  $Al_2O_3$  is much harder against ionizing radiations [Vigouroux et al., 1984]; therefore electron trapping can only occur on intrinsic defects.

In order to check the charge origin of the intensity variation,  $SiO_2$  and  $Al_2O_3$  were heated at  $700^\circ C$  and observed in Auger electron spectroscopy. No noticeable difference from previous room temperature experiments could be detected. For  $SiO_2$ , upon temperature, a balance exists between defects creation under the electron beam and annealing. The latter process requires a time constant  $10^{-12}$  s, which is much greater than the time associated with the Auger transition ( $\sim 10^{-14}$  s). Therefore the lifetime of the charge localized close to the oxygen atom could be high enough, so that an interaction still takes place.

In  $Al_2O_3$ , electrons are trapped on intrinsic defects, which are very stable against temperature increase. Heating

can then only produce detrapping of electrons localized very close to the bottom of the conduction band.

In both cases, charges can be temporarily trapped even at a high temperature and modify the Auger emission.

Under Al  $K\alpha$  X-ray bombardment, the density of electronic excitation is lower. Then the electrons can only be trapped on preexisting defects. Holes, created in the valence band, can be trapped in levels localized in the gap, near the top of the valence band. Holes and electrons are not trapped into the same sites. These differences in the nature of the trapped charge and of the trapping center could account for the observed

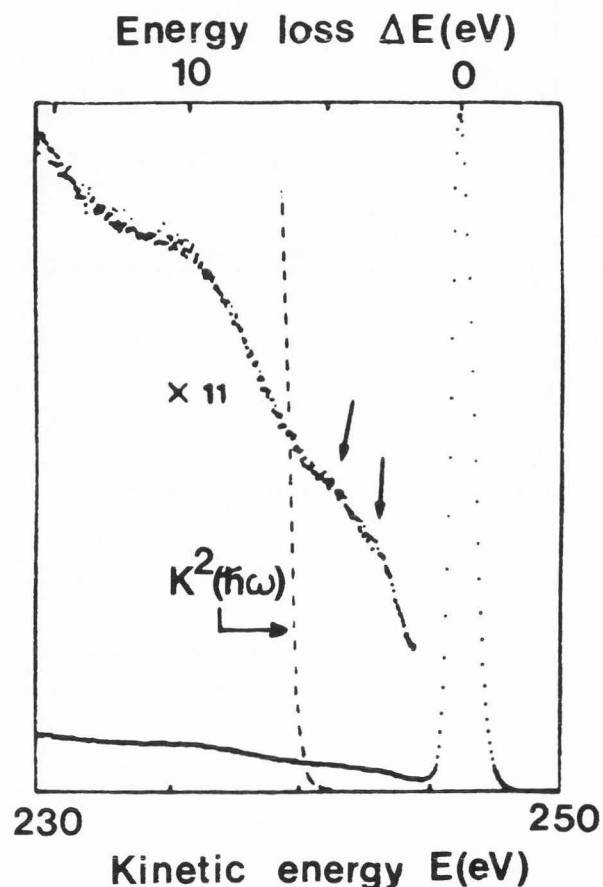


Fig. 4. Electron energy spectrum obtained on AlN, with electron primary energy  $E_p = 250$  eV.

Dashed line = absorption coefficient measurement  $K^2(\hbar\omega)$  by Perry and Rutz (1978), showing the 6 eV wide band gap. Two loss peaks (3 eV-5 eV) appear in the band gap (corresponding to localized levels due to structural defects). The 10 eV loss peak arises from an interband transition.

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variation in both  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  under electron and Al  $K\alpha$  X-ray excitation.

Under Zr  $L\alpha$  X-ray excitation, energetic secondary electrons are produced by the interaction of the intense Bremsstrahlung and the Al window. They act as a parasitic electron beam, leading to a situation similar to the electron bombardment.

The intensity variation of the Auger  $L_{23}VV$  line between Al and AlN could be explained in this framework, if one takes into account the large band gap of this solid ( $\sim 6.2$  eV) [Lieske and Hezel, 1981]. Radiation effects under electron beam have previously been observed by Kovacich et al. (1984). Electron energy loss spectroscopy we performed clearly shows structures in the band gap (figure 4). The observed structures can be attributed to structural defects produced by the nitrogen ion implantation, leading to localized levels in the band gap, on which electrons can be trapped, in a similar way as for  $\text{Al}_2\text{O}_3$ . These results will be extensively discussed in a forthcoming paper.

### Conclusion

Experimental results obtained on Si,  $\text{SiO}_2$ , Al,  $\text{Al}_2\text{O}_3$  and AlN exhibit large differences in the intensity of the  $L_{23}VV$  Auger peak, according to the excitation source: electron beam, Al  $K\alpha$  or Zr  $L\alpha$  X-rays. In the case of oxides, defects related to oxygen could be created under irradiation. They could trap electrons which will modify locally and temporarily the electric potential and then the emission of electrons. With regards to AlN results, an analogous explanation can be made as defects are created under electron irradiation in this large band gap solid.

These results must be taken into account before studying interatomic Auger rates with line intensities. Much attention must also be paid to them in the frame of quantitative surface analysis by Auger electron spectroscopy. In this case, the exact conditions under which the Auger intensity can be measured and related to a given concentration are yet to be determined.

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### Discussion with Reviewers

R. Shimizu: During the measurement, particularly oxide and nitride, have you observed any change in the surface concentration due to electron bombardment? If so, how large was it?

Authors: During these experiments, low current beams were used ( $\sim 10^{-9}$  A).



Under these conditions, no concentration change could be detected in the Auger spectra.

R. Shimizu: With respect to charge build-up under irradiation haven't you measured S(E) spectrum as you did before [Le Gressus et al. SEM/1984], which would be more direct evidence on this argument?

Authors: The shift of the S(E) spectrum is characteristic of the build-up of a macroscopic charge. In this study we did not observe any shift under X-ray radiation, but that does not imply the lack of localized trapped charges: indeed a lot of secondary electrons are produced by the X-ray source, which can reach the sample and compensate the positive charge induced by the X-rays.

In the case of electron bombardment, a 2 eV negative charge is observed, which could be observed in the secondary electron spectra.

R. Shimizu: The structures indicated with arrows in fig. 4 seems to be single peak due to surface plasmon provided that another peak is bulk plasmon. Is'nt it?

Authors: The energy of bulk plasmon in AlN is  $\hbar\omega = 20$  V; therefore it does not appear on the loss spectrum of Fig. 4. The structures indicated with arrows in this figure (at 3 eV and 5 eV) are attributed to structural defects, inducing localized levels in the band gap. (The band gap region, determined by optical methods [Perry and Rutz, 1978] is represented on the spectrum). Another peak, located at 10 eV from elastic peak, corresponds to an interband transition.

J.C. Rivière: What are the energies of the losses arrived at in Figure 4, and to what precise transitions are they attributable? Have any other workers measured losses from AlN, and are your results in agreement? Where does "nitrogen ion implantation" come into the argument?

Authors: The loss spectrum of Fig. 4 extends from loss energy 0 (elastic peak) to loss energy 15 eV. Therefore the energies of the losses are: 3 eV, 5 eV and 10 eV. As said in the text, these results will be discussed in a forthcoming paper. The 10 eV loss peak corresponds to an interband transition, between an occupied state in the valence band and an empty state in the conduction band. The 5 eV and 3 eV loss peaks are located in the band gap (see fig. 4). Therefore they are related to the presence of structural defects, which induce localized levels in the band gap: the losses observed correspond then to transitions between an occupied state of the valence band and a localized level in the band gap. As explained in the paper the nitrogen ion implantation is used to prepare the AlN

film; indeed it is known that, when the saturation is reached, the obtained AlN film is stoichiometric [Lieske and Hezel, 1981] and crystalline [Denanot et al. 1985].

A previous study on AlN has been realized by Lieske and Hezel (1981), using electron energy loss spectroscopy in the second derivative mode and with high current densities leading to radiation damage. They observed structures similar to that obtained in our experiments in the N(E) mode.

J.C. Matthew: It is well known that oxides tend to have very high secondary emission coefficients under some conditions; important modifications of the secondary emission could affect the background intensity near the Auger peak and then change the ratio peak over background. The results presented here may reflect that kind of anomaly. Could you give a brief discussion about this point?

Authors: We have measured the absolute intensity of secondary emission on Al as a function of oxidation: the secondary intensity, measured as the area of the N(E) curve between 0 eV and 100 eV increases by a factor 1.5 between pure aluminum Al and Al+5000L O<sub>2</sub>. The intensity of background in the vicinity of the Auger peak increases by a factor 1.4. Nevertheless this variation could not account for the larger observed difference in the Auger intensities.

D.E. Ramaker: If one assumes that the core ionization cross section does not change upon oxidation, and that the fluorescence yield is negligible relative to the Auger decay for both the metal and the oxide, then it seems that the Auger process is the primary mechanism available for decay of the core hole. What difference then, does it make to the total Auger intensity if the decay is intraatomic or interatomic in nature, since each core hole decay produces one Auger electron?

Authors: It is not obvious that the  $L_{23}VV$  Auger yield is the same in the metal and the oxide, as far as the chemical environment of the Al atom is different. This problem was already the subject of previous publications [Matthew and Komninos, 1975; Yafet and Watson, 1977].

D.E. Ramaker: Why should the defect centers in the oxides, created by irradiation, selectively trap Auger electrons, versus secondary or background electrons, since they coexist at the same energy?

Authors: Not only Auger electrons can be trapped on defect centers, but any electron, if its energy allows recombination. The charged defects can then change the

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local potential and affect the energy and angular distribution of emitted electrons. Moreover, as most of the defects are oxygen related, it seems reasonable to infer that radiation effects due to electron excitation may affect the intensity of Auger electrons resulting from an interatomic process.

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