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Effect of Low Energy Electron Exposure on Ion Scattering Spectroscopy

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Abstract: The effect of low energy electron (LEE) exposure on ISS, including on the sputter peak and scatter peak as well as on ion neutralization, has been investigated for different samples. Some new results are discussed.

Keywords: Ion scattering spectroscopy (ISS), charge neutralization, ion neutralization.

Low energy ion scattering spectroscopy (ISS) is a technique which can provide an elemental analysis of one monoatomic layer of a solid surface[1, 2]. The main advantage of ISS lies in its high sensitivity for the outermost atomic layer. ISS has been used to examine surface chemistry of a wide variety of solid materials, including insulators.

An important question in ISS analysis of insulators is the charging of sample surface. Charges formed under ion bombardment may influence peak position on a large scale. So far, charge neutralization has usually been performed with an extra low energy electron (LEE) beam. However, to our knowledge, no in-depth discussion about the effect of LEE exposure on ISS spectra has been put forward. In the present work, we have investigated the effect of LEE exposure on ISS.

Experimental

ISS analysis was performed on an ESCALab 220i-XL electron spectrometer. The base pressure in the analysis chamber was about 3×10^{-9} mbar. The spectra were measured with a primary beam of $^4He^+$ at an energy of 1000 eV and a current density of about 20 $\mu A/cm^2$. The scattering angle was fixed at 130^o. The filament current of the electron flood gun was fixed at 2.5 A and the electron energy was 6 eV for charge neutralization. ISS measurements were carried out after the primary ion gun and electron flood gun were in steady working conditions.

The specimens examined were smooth silver sheets (sample 1 and 2) and lead fluoride (sample 3). Sample 1 was in good electrical contact with the sample holder, and sample 2 was mounted on the sample holder with an insulating double sided adhesive tape. Resistivity measurement showed that it was insulating from the sample holder. Sample 3 was in analytical grade of PbF_2 reagent, which was pressed into a

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pellet. All samples were analyzed after a cleaning treatment with 3 keV Ar^+ ions etching for 1500 s. XPS analysis showed the presence of very weak C1s and O1s peaks from surface contamination.

Results and Discussion

Figure 1 shows a comparison of ISS spectra without (a) and with (b) LEE exposure of sample 1. The spectra have two regions. The low energy peak at about 20 eV is corresponding to the sputter peak and the higher energy peak at 887-888 eV is to the scatter peak. The energy of the scatter peak is in good agreement with the one predicted by the binary elastic collision model[1, 2].

It can be seen that the spectra a and b are similar, indicating that LEE exposure has no evident effect on the spectra, including the peak position and intensity. This is because electrically grounded conducting Ag does not develop a net positive charge during positive ion bombardment.

The results shown in **Figure 1** indicate that the low energy sputter peak can be observed not only on insulating sample, but also on surface contaminated conducting sample. Bertrand *et al*[3] have suggested that the low energy species are mainly ionized by low energy electrons. Our results showed that they were not ionized by the low energy electrons, but ionized by the primary He^+ ions.

Spectra a in **Figure 2** and **3** show the low energy sputter peak and the elastic scatter peak for electrically isolated Ag sheet without LEE exposure during ISS analysis, respectively. The sputter peak and scatter peak are located at 108 eV and 901 eV, respectively. By comparison, the sputter peak and scatter peak are located at 21 eV and 887 eV for the same sample with LEE exposure (see spectra b in **Figure 2** and **3**, respectively). Both of the sputter peak and scatter peak shift to much higher energy when the sample is analyzed without LEE exposure. This is because the conducting sample is not electrically grounded, it develops a net positive charge during He^+ ion bombardment. Once the sample is exposed to the LEE, the positive charge in sample surface is neutralized and the peaks resume the original position. However, the energy shift of the sputter peak (87 eV) is larger than that of the scatter peak (14 eV), and this phenomenon is also observed in sample 3. For a given charging of V volts, any singly charged ion will get an extra eV in kinetic energy. The observed difference is due to the fact that the incident ions are first decelerated. The final energy E_f^* of the ions for a charged surface is, therefore, given by $E_f^* = E_f + (1-k)eV$, where k is the kinematic factor[4]. The effect of charging and hence the shift of scatter peak is the largest for the lowest k (the lightest elements in sample).

LEE exposure also influences the peak intensity of sample 2. By comparing spectrum b with a in **Figure 2** and **3**, it is clear that the intensity of the sputter peak decreases while the intensity of the scatter peak increases when ISS analysis is carried out with LEE exposure. The decrease of the sputter peak intensity can be attributed to remove of parts of adsorbed species on sample surface due to electron bombardment, this leads to decrease of the sputter peak intensity and increase of the scatter peak intensity.

Serious charging effect also occurs during ISS analysis of PbF_2 pellet without LEE exposure, which made the sputter and scatter peaks move to higher energy side. The situation is similar to that of sample 2. However, we have observed that the scatter peaks are quite broad and have shoulders (see **Figure 4**). We have mainly attributed this to inhomogeneous charging on the sample surface. Since sample 3 is pressed pellet,

Figure 1 Comparison of ISS spec-cally grounded Ag sheet without (a) and with (b) LEE exposure

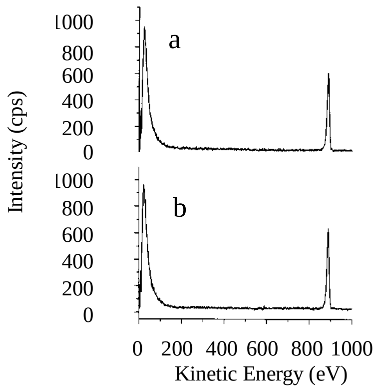


Figure 2 Comparison of sputter peak in ISS spectra of electrically isolated Ag sheet without (a) and with (b) LEE exposure

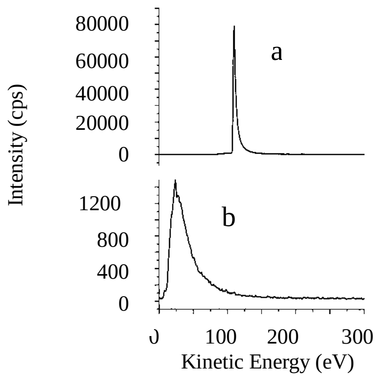


Figure 3 Comparison of scatter peak in ISS spectra of electrically isolated Ag sheet without (a) and with (b) LEE exposure

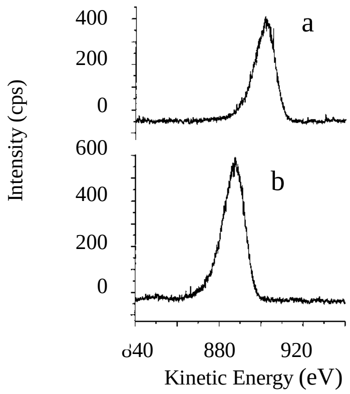
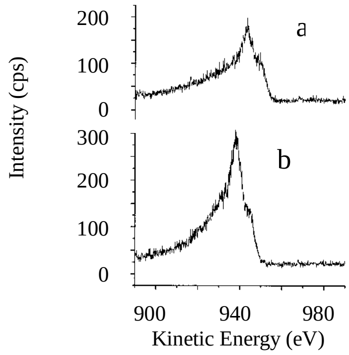


Figure 4 Comparison of scatter peak in ISS spectra of PbF_2 pellet without (a) and with (b) LEE exposure



the surface is not so smooth. When an insulating sample is impacted by the primary ions either with or without LEE exposure, surface roughness will lead to inhomogeneous charging. This gives peak broadening and with shoulders.

The effect of electron energy and filament current of LEE gun upon the sputter and scatter peak intensities was examined. Our results on sample 2 showed that as the electron energy or filament current increased, the intensity of the sputter peak decreased and the intensity of the scatter peak increased. This is understandable because exposure to LEE with higher energy or current can remove more adsorbed species on sample surface.

In addition to the charge neutralization, another important question concerns the ion neutralization. Parts of primary inert ions penetrating the surface are neutralized. Ion neutralization can have an important influence on the peak height therefore on quantitative interpretation of ISS spectra. Our experimental results showed in **Figure 1, 3** and **4** reveal that the scatter peak intensities increase when samples are analyzed with LEE exposure, which implies that electrons can not neutralize He^+ ions. In fact effective neutralization of He^+ is only possible in a 3-body interaction. This occurs only under extreme electron densities.

Conclusion

The energy shifts due to surface charging disappear and the intensity of the sputter peak decreases while the intensity of the scatter peak increases when ISS analysis for electrically isolated conductor or insulator is carried out with LEE exposure. Besides, the energy shift of the sputter peak between the analysis without and with LEE exposure is much larger than that of the scatter peak. As the electron energy and filament current of LEE gun increase, the intensity of the sputter peak decreases while the intensity of the scatter peak increases. Finally, it has been found that LEE exposure does not affect ion neutralization.

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