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# Inelastic Scattering Cross Section of Si Determined from Angular Dependent Reflection Electron Energy Loss Spectra

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Inelastic scattering cross sections of Si in the form of $\lambda K(\Delta\mathrm{E})$, the production of the inelastic mean free path and the inelastic scattering cross section, were obtained from the angular dependent reflection electron energy loss spectroscopy (REELS) spectra. The REELS spectra with the energy and angular dependence were measured using an inclined sample holder. The spectra were taken for the primary beam energy from 500 to 4500 eV at a fixed incidence angle and various emission angles. For each primary energy, a series of ten $\lambda K(\Delta\mathrm{E})$ spectra were obtained. The experimental $\lambda K(\Delta\mathrm{E})$ spectra obtained from the angular dependent REELS spectra were compared in absolute unit. It clearly showed the variation in the relative contribution of bulk and surface losses in these series of the $\lambda K(\Delta\mathrm{E})$ spectra after subtracting the multiple scattering. It allowed separating the surface and bulk loss contribution based on the changing of the emission angles.

## 1. Introduction

Quantitative analysis and the interpretation of the surface electron spectroscopy require the understanding of the inelastic scattering properties of low-energy electrons in solids. Therefore a good knowledge of the electron inelastic scattering cross sections is necessary.[1-3] Reflection electron energy loss spectroscopy (REELS) is a useful technique in surface and interface analysis because the backscattered electrons provide information on the electron-solid inelastic interaction and can motivate the analysis of nano structures for the use of medium primary energy.[4,5] In REELS, the incident electrons can be inelastically scattered through interaction with either outer- or inner-shell atomic electrons. So REELS is a complicate combination of separate physical phenomena including bulk and surface plasmon excitation, interband transition, multiple scattering and momentum transfer.[6] The inelastic scattering cross section describes the energy loss of electrons traveling in solids. It has relation to the primary electron energy, the trajectory of the electron traveling and the dielectric properties of the medium. Since the probing depth in REELS is very small and in nanometer scale, the excitation in the surface region is also important. The inelastic scattering cross section contains both contributions of surface and bulk losses. Extracting bulk excitation by quantitative analysis of REELS spectra can also give us direct information on the electronic properties of the dielectrics.[7] Inelastic scattering cross section of electrons is an important bulk-material-parameter for surface electron spectroscopy analysis. It determines quantities such as the inelastic mean free path (IMFP).[8] Besides, we can obtain the energy loss function, which is directly connected to optical constants $\hat{\varepsilon}$, from the bulk inelastic scattering cross section.[9] A good knowledge of dielectric properties is also a requirement for better understanding of electron spectroscopy.

The aim of this study is to obtain the variation of the bulk and surface loss contributions with the dependence on the primary electron energies and emission angles of REELS spectra for Si. We obtained the experimental inelastic scattering cross section from series of REELS spectra directly. These series of inelastic scattering cross section spectra clearly showed the variation of relative contributions of bulk and surface losses dependent on the angles and primary beam energies. Separation of bulk and surface loss contributions in the experimental inelastic scattering cross section can give us the above-mentioned valuable information on the electronic structure and the knowledge of surface excitation described by the surface excitation parameters (SEP).[10]

## 2. Experiment

The REELS spectra were measured using Ulvac-Phi model 5500 electron spectrometer equipped with a concentric hemispherical analyzer. Primary electron beam energies were from 500 to 4500 eV. The spectra were measured in the constant analyzer energy mode with pass energy of 23.5 eV. The minimum energy loss interval was 0.1 eV. The signal acquisition time was optimized to give an adequate signal-to-noise ratio for the inelastic part of the spectrum and was set at 100 ms time/step. The spectra were measured in a wide energy range (0-100 eV). REELS experiments at various emission angles were carried out using an inclined sample holder. Figure 1 shows the diagram for the geometry of the REELS experiments. The Si sample was mounted on a sample holder which was inclined by $30^0$. This setup can provide a wide range of emission angles to the surface normal ($15^0$-$75^0$) when rotating the sample holder. The electron-gun axis is in the vertical direction. This gives a constant incidence angle of $30^0$ with respect to the surface normal. The angle between the axis of the electron-gun and the analyzer is $45^0$. We obtained a series of spectra at different emission angles for fixed primary beam energy. The Si sample was sputtered for 5 minutes using 2 keV $Ar^+$ sputtering for removing the surface contamination. Due to the wide observation area projected on the sample at higher emission angle, the sputtering area should be large to cover the whole sample. The cleanness of the sample was checked by Auger electron spectroscopy. The base pressure in the analysis chamber was approximately < $2x10^{-9}$ Torr during the spectra measurement.

## 3. Results and discussion

It should take care of some spectrometer parameters before measuring the REELS spectra. It need ensure the linearity response of the analyzer. There should be no saturation of an electron detector when measuring the elastic peak. One should adjust the beam current to get the spectrum with a good signal-to-noise ratio and a sharp elastic peak.[11] In addition, the selection of the acceptance angle of the analyzer need also be considered when there is necessity to measure the REELS spectra for high primary beam energy. The acceptance angle 2, 5 and 7 degree of the analyzer are selectable in the machine control software. However, the acceptance angle of 7 degree was excluded by the following machine limitation. Figure 2 shows the comparison of wide energy loss electron spectra for primary electron energies of 5000 eV at the acceptance angle 2, 5 and 7 degree of the analyzer. These spectra were measured in fixed analyzer transmission mode and scanned in a wide energy range (500-4500 eV). We can notice there is strange intensity in the energy range of 2200-3200eV and sudden drop off of the spectra at the acceptance angle of 7 degree. This trouble means the inadequate input lens condition over the upper limit of power supply of analyzer for measurement at high primary beam energy of 5000 eV. Higher acceptance angle needs higher voltage applied on the electrode of the input lens. It gives the normal spectrum at the acceptance angle of 2 and 5 degree. It is better in our case to use the acceptance angle 5 degree of the analyzer considering the option of analysis area.

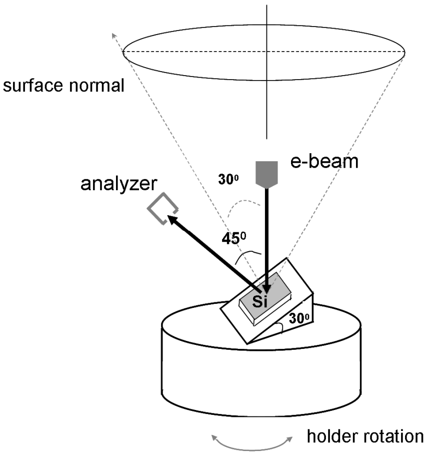

**Fig. 1** Diagram of geometry for the reflection electron energy loss spectroscopy experiments.

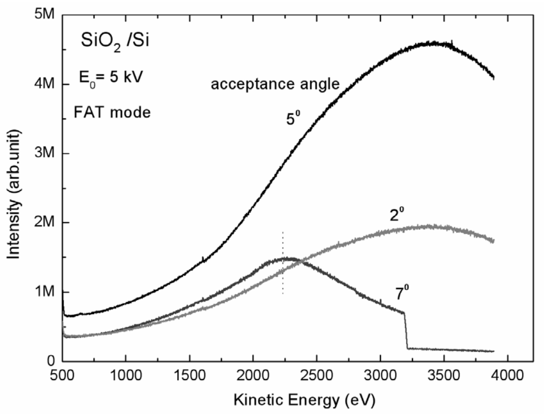

**Fig. 2** The comparison of wide energy loss spectra for primary beam energy of 5000 eV at the acceptance angle 2, 5 and 7 degree of the analyzer.

Figure 3 shows the comparison of the REELS spectra at various emission angles for primary electrons of energy 500, 1000, 2000, 3000, 4000 and 4500 eV. All the spectra were normalized at the elastic peak. Collective excitations and electronic transition are considered to characterize the REELS spectra. The bulk plasmon peak at 16.8 eV and the surface plasmon at about 11 eV for Si were clearly recognized and in agreement with the others designation.[12] For each series of spectra at a fixed primary electron beam energy, the surface plasmon increased in intensity relative to the bulk plasmon in particular for emission angles higher than $56^0$. These series of spectra showed the gradual variation of bulk and surface plasmon peaks intensity with the dependence of the primary electron energies and emission angles. In addition, a broad loss feature at about 34 eV is one of the multiple bulk plasmon ($n\hbar\omega$) peaks. It clearly showed the multiple bulk plasmon in Si in the wide energy loss range. Here we just show the spectra in the energy loss range of 0- 40 eV for clear observation of single surface and bulk plasmon peaks.

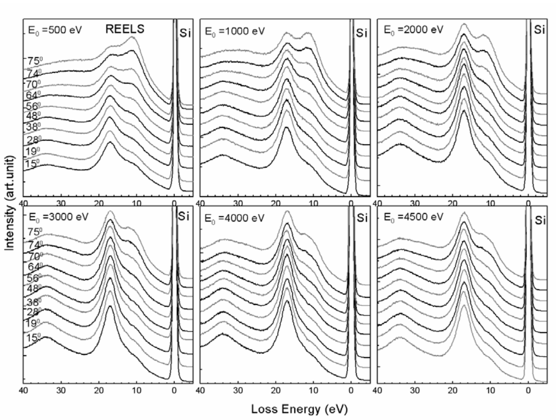

**Fig. 3** Comparison of reflection electron energy loss spectra of Si at various emission angles for primary electrons of energy 500, 1000, 2000, 3000, 4000 and 4500 eV [14].

A series of the experimental $\lambda K(\Delta \mathrm{E})$ spectra calculated from the corresponding REELS spectra at various emission angles was shown in figure 4. The $\lambda K(\Delta \mathrm{E})$ spectrum was obtained using QUASES-XS-REELS software developed by Tougaard.[11,13] The $\lambda K(\Delta \mathrm{E})$ means the product of the inelastic mean free path and inelastic scattering cross section. The inelastic scattering cross section $K(\mathrm{E}, \Delta \mathrm{E})$ is the probability that an electron of energy $E$ shall lose energy $\Delta \mathrm{E}$ per unit energy loss and per unit path length traveled in the solid. Since multiple scattering features were removed in the $\lambda K(\Delta \mathrm{E})$ curve, the bulk and surface plasmon are now clearly identified. The $\lambda K(\Delta \mathrm{E})$ spectra are in absolute values and make it possible to compare the $\lambda K(\Delta \mathrm{E})$ spectra on an absolute scale. There was an apparent surface loss peak at about 11 eV for spectra at 500 eV and for higher emission angles due to the decreased penetration of the primary electrons. At higher primary energy and low emission angle, the contribution of bulk loss was significant and a little surface loss feature gradually became visible with increasing emission angle. These $\lambda K(\Delta \mathrm{E})$ spectra clearly showed the variation of bulk and surface loss contributions with the changing of primary electron energies and emission angles. The negative part of the spectra appeared at $\sim \hbar\omega_s + \hbar\omega_p$ . Here the parameter $\hbar\omega_s$ is the surface plasmon energy and $\hbar\omega_p$ is the bulk plasmon energy. This is due to the differences of estimated multiple scattering events in the Tougaard's algorithm and the experimental situation. Therefore, the relative intensities in the various multiple surface and bulk plasmon excitation peaks will be slightly different from the real experimental situation.[2,3,13] This effect is only seen for solids with a sharp intense plasmon structure (like in Al and Si). However it is less obvious for other materials with wide loss structure [3], such as some oxides, $SiO_2$, $HfO_2$. For spectra at primary energies higher than 2000 eV, only a small negative parts of $\lambda K(\Delta \mathrm{E})$ appeared at high emission angle.

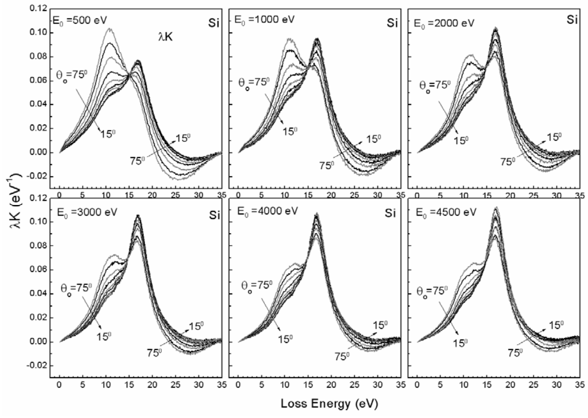

**Fig.4** Series of the experimental $\lambda K(\Delta \mathrm{E})$ spectra obtained from the corresponding REELS spectra in Fig. 3 at various emission angles. $\theta_o$ is the electron emission angle with respect to the surface normal [14].

Even the surface loss contribution at higher primary energy is not prominent; its contribution should affect the results of energy loss function evaluated from the inelastic scattering cross section. We can obtain the exact energy loss function and further the optical constant using the Kramers-Kronig analysis by extraction of the bulk inelastic scattering cross section from inelastic scattering cross section. We will use the factor analysis method to do quantitative analysis for these series of $\lambda K(\Delta E)$ spectra.

## 4. Conclusions

We measured the REELS spectra of Si with the variation of primary energies and emission angles by rotating an inclined sample holder. From these series of REELS spectra, we determined the experimental $\lambda K(\Delta E)$ spectra using Tougaard's algorithm. The variation of the relative contribution of bulk and surface plasmon losses were clearly observed in these series of $\lambda K(\Delta E)$ spectra dependent on the emission angles and primary electron energies. In a further study, we will factor analysis method to separate the surface and bulk loss contribution in the inelastic scattering cross section in term of the $\lambda K(\Delta E)$ variation dependence on the emission angles. Then, we can estimate some important parameters, such as optical constants and the surface excitation parameter.

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