

Reflection Electron-Energy-Loss Spectroscopy of $\text{Fe}_x\text{Si}_{1-x}$ Thin Films

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Abstract—The values of the product of the inelastic mean free path and the differential cross section for inelastic scattering of electrons have been determined from the reflection electron-energy-loss spectra of thin films of the $\text{Fe}_x\text{Si}_{1-x}$ system ($0 \leq x \leq 1$). A new approach to the quantitative analysis of components in such composite media is proposed.

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Compounds of the $\text{Fe}_x\text{Si}_{1-x}$ system ($0 \leq x \leq 1$) combining semiconductor and magnetic properties are promising materials for spintronic devices whose operation is based on the spin-dependent properties of charge carriers. In recent years, heterostructures comprising alternating layers of iron and silicon (Fe/Si) with thickness on the order of several nanometers have been extensively studied. Investigations of the exchange between ferromagnetic layers via a non-ferromagnetic spacer is of both applied and basic interest [1–3].

The preparation and investigation of Fe/Si heterostructures encounter considerable difficulties related to a significant interdiffusion of iron and silicon with the possible formation of various iron silicides at the interfaces between layers, which strongly modify the physical characteristics of these heterostructures. Owing to their extremely high surface sensitivity, various electron spectroscopy techniques including the Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), and electron-energy-loss spectroscopy (EELS) are best suited due for the study of elemental composition and the formation of chemical compounds in such thin films and multilayer structures. Investigations of thin iron and silicon films by various spectroscopy techniques reported previously (see, e.g., [4–6]) mostly employed a traditional approach to the interpretation of spectra, which is based on an analysis of the positions and intensities of peaks as functions of the ratio of iron and silicon concentrations and the experimental parameters. However, this approach is frequently low-effective because the electron spectra vary rather weakly depending on the chemical composition. For example, the energy positions of the peaks of bulk

plasmon losses in the EEL spectra of various iron silicides vary within only about 1 eV at a peak width of several electronvolts, which complicates the study of the formation of chemical compounds at the Fe/Si interfaces by the EELS method. At the same time, analysis of the available experimental data [7–9] shows that the maximum differential cross sections for the electron scattering in silicon and iron differ by a factor of more than 2. In addition, the shape of the dependence of the cross section on the electron energy losses and the position of maximum in this dependence for Fe and Si are also significantly different. These features provide a basis for the quantitative study of the elemental composition and chemical compounds in the given system using data on the differential cross section for inelastic scattering of electrons.

At present, there is a sufficiently large amount of experimental data on the cross sections for inelastic scattering of electrons in various materials and their relationships with the parameters of the EEL spectra. Tougaard and Chorkendorff [10] first demonstrated the possibility of calculating the differential cross section for inelastic scattering of electrons using the EEL spectra measured in the reflection mode. The original numerical algorithm was verified on the reflection EEL (REEL) spectra of aluminum. The cross section determined in this way was used for subtracting the background of inelastically scattered electrons from the photoelectron spectra of aluminum excited by the MgK_α and synchrotron radiation. In order to provide a deeper insight into the properties of the electron scattering cross section of various solids and determine the limits of applicability of this approach, systematic investigations of the cross section were performed for

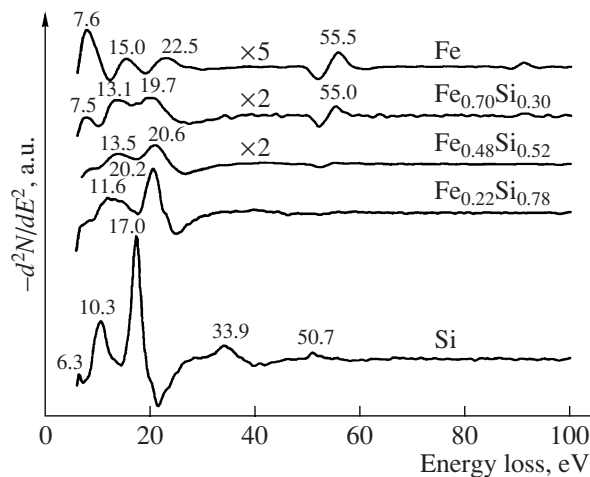


Fig. 1. The typical REEL spectra of various samples of the $\text{Fe}_x\text{Si}_{1-x}$ system (shifted along the vertical axis in the order of increasing Fe content), measured at a primary electron energy of $E_0 = 300$ eV. Figures at the peaks indicate their positions in electronvolts; multipliers ($\times 2$, $\times 5$) indicate the amplification coefficients used in recording the spectra at a gain exceeding unity.

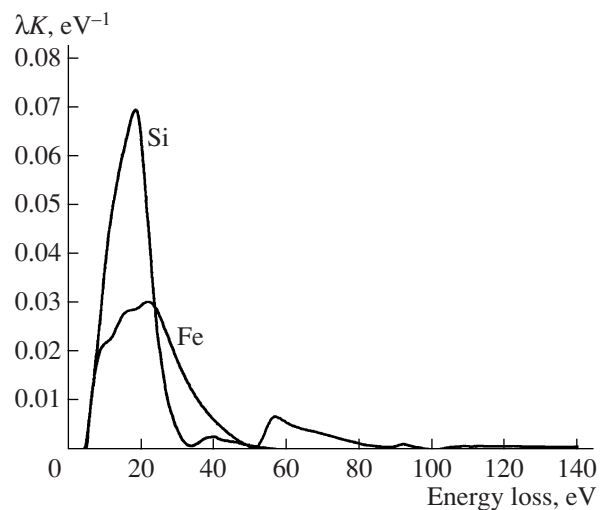


Fig. 2. The spectra of λK products calculated using the experimental REEL spectra of silicon and iron recorded at a primary electron energy of $E_0 = 300$ eV.

a broad class of materials. In particular, Tougaard and Kraer [9] reported on the cross sections of Si, Cu, Ag, Au, Ti, Fe, and Pd determined from an analysis of the experimental REEL spectra for the primary electron energies in the 300 eV–10 keV range. Based on these results, Tougaard has developed a commercial program package for the quantitative analysis of surfaces by electron spectroscopy (QUASESTM_XS_REELS) [11], which is intended for calculations of the product of the inelastic mean free path (IMFP) and the differential cross section for inelastic scattering of electrons using the data of REELS. With this software, it is possible to perform calculations in various regimes depending on the type of experimental spectra.

The present investigation is devoted to assessing the possibility of applying a new approach to the quantitative analysis of the $\text{Fe}_x\text{Si}_{1-x}$ system using experimental REELS data on the product of the IMFP and differential cross section for inelastic scattering of electrons.

The samples were prepared in the preparation chamber of a LAS-2000 ultrahigh-vacuum Auger spectrometer (Riber, France) by thermal deposition of silicon and iron onto *p*-type Si(111) substrates with a resistivity of $5 \times 10^{-10} \Omega \text{ cm}$ and dimensions $22 \times 5 \times 0.3$ mm. Prior to deposition, the substrates were cleaned from surface contaminations by annealing in vacuum at 600°C for 4 h, followed by flashing to 1200°C for 1–2 min at a residual gas pressure of $(1\text{--}2) \times 10^{-7}$ Pa. The source of Fe atoms was a resistively heated tantalum stripe with welded pieces of pure iron. The source of Si atoms was a resistively heated silicon plate with the same dimensions as the substrate. The rate of silicon deposition was about 0.07 monolayer per minute. The composition of deposited films was varied by control-

ling the rate of Fe deposition. In this way, five samples of the $\text{Fe}_x\text{Si}_{1-x}$ system were prepared, in which the atomic fraction of iron was $x = 0, 0.22, 0.48, 0.70$, and 1.

The atomic concentrations of elements were measured using the differential AES spectra and the known elemental sensitivity coefficients. The primary electron energy in these measurements was $E_0 = 3000$ eV. The concentrations of Fe and Si were determined for the Auger electron peaks at 92 eV (Si) and 703 eV (Fe), using the elemental sensitivity coefficients from [12]. The film growth on the substrate was performed at room temperature and a residual gas pressure of about 1×10^{-7} Pa.

The REEL spectra were recorded in the differential mode at a primary electron energy of $E_0 = 300$ and 1600 eV and a modulation voltage amplitude of 0.3 V. For the quantitative analysis, the spectra were numerically differentiated, and the positions of peaks were determined as corresponding to the maximum of d^2N/dE^2 , where $N(E)$ is the number of electrons with the energy E . Figure 1 shows the typical REEL spectra of samples measured at a primary electron energy of $E_0 = 300$ eV. In the spectrum of pure silicon, the main peak at 17.0 eV corresponds to the excitation of a bulk plasmon, while the peaks at 33.9 and 50.7 eV are due to losses for the excitation of double and triple bulk plasmons. The other peaks are, according to the published data [5], due to the excitation of a surface plasmon (10.3 eV) and the E_2 interband transition (6.3 eV) in silicon. The spectrum of pure iron also exhibits the characteristic peaks [6] due to the bulk plasmon (22.5 eV) and the surface plasmon (15.0 eV). The peak at 7.6 eV is usually attributed to transitions from the edge of the $\text{Fe}3d$ band to vacant states 1.5 eV above the Fermi

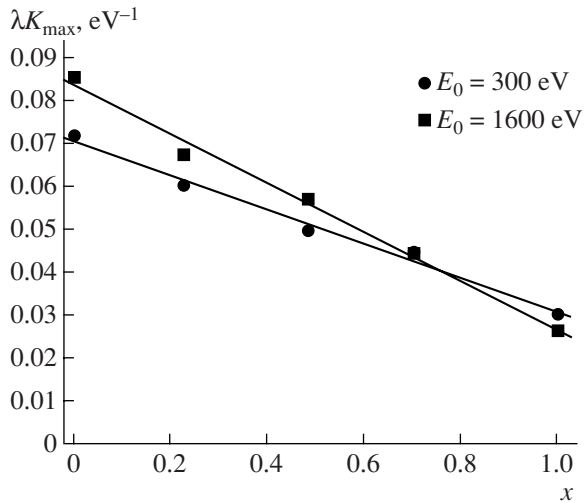


Fig. 3. Plots of the λK_{\max} values versus iron content in the $\text{Fe}_x\text{Si}_{1-x}$ system constructed using REEL spectra measured at different primary electron energies E_0 .

energy, while the peak at 55.5 eV corresponds to the M_{23} interband transition. The quantitative interpretation of the REEL spectra of intermediate compositions is complicated because of a complexity of the spectra of components and the large widths of the loss peaks. It is only possible to follow a monotonic decrease in intensity of the interband transition in iron with decreasing content of this element in the composite structure.

After subtraction of the instrumental function and numerical integration, the REEL spectra $j(E)$ were processed using the QUASESTM_XS_REELS program package for determining the product of the IMFP (λ) and the differential cross section for inelastic scattering of electrons $K(E_0, E_0 - E)$, where E_0 is the primary electron energy). The algorithm for calculating the product of $\lambda K(E_0, E_0 - E)$ is as follows [10, 13]:

$$\lambda K(E_0, E_0 - E) = \frac{1}{c} \left[j(E) - \int_E^{E_0} \lambda K(E_0, E' - E) j(E') dE' \right],$$

where c is the area under the elastic peak.

Figure 2 shows the results of processing of the experimental REEL spectra of pure silicon and iron recorded at a primary electron energy of $E_0 = 300$ eV. The results qualitatively and quantitatively agree with the available published data [7–9]. Analogous results were obtained for a primary electron energy of $E_0 = 1600$ eV and for other samples with various contents of silicon and iron. Figure 3 presents a plot of the maximum values of the $\lambda K(E_0, E_0 - E)$ product versus iron content in the system. As can be seen, these plots are linear for both primary electron energies. The results of our simulations of the λK value for silicon and iron

using a specially developed software [14] based on the model of Yubero and Tougaard [7] showed that this product for silicon increases from 0.080 eV⁻¹ at $E_0 = 300$ eV to 0.092 eV⁻¹ for $E_0 = 1600$ eV, while the product for iron exhibits the reverse change, decreasing from 0.027 eV⁻¹ at $E_0 = 300$ eV to 0.022 eV⁻¹ for $E_0 = 1600$ eV. These results agree with the experimentally observed changes in the slope of the plots in Fig. 3 depending on the primary electron energy.

Thus, it is established that the values of the product of the IMFP (λ) and the differential cross section (K) for inelastic scattering of electrons can serve a quantitative measure for determining the elemental composition of a composite medium from the calibration graph of λK_{\max} constructed for a series of reference samples.

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