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Determination of the thicknesses and depths of subsurface nanostructures using a scanning electron microscope

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Received September 12, 2022
Revised October 12, 2022
Accepted October 12, 2022

The calculated ratios of the signal of backscattered electrons for multilayer nanostructures are derived depending on the energy of probing electrons and composition of multicomponent samples. From experimentally measured signals and calculated ratios, not only thicknesses but also, for the first time, depths of occurrence of local microheterogeneities of three−dimensional nanostructures were determined. The studies were carried out by a non−destructive method of detecting backscattered electrons in a scanning electron microscope.

Keywords: multilayer nanostructures, scanning electron microscopy.

DOI: 10.21883/TPL.2022.12.54939.19361

More and more three−dimensional (3D) submicronic nanostructures are at present produced by using up−to−date nanotechnologies. Among those there are, for instance, multilayer microelectronic devices including integrated chips, optoelectronic and X−ray elements, etc. In these fields of fundamental and applied researches there exists an actual task of determining the 3D architecture of microobjects, e. g., thicknesses of multilayer nanocoatings, depths of occurrence of fragments of various inhomogeneities in the matrix. At present, a common method for determining those parameters is optical confocal microscopy; however, it is applicable only to transparent optical media and does not provide the required submicron resolution. As for transmission electron microscopy, it is essentially destructive because needs preparation or fabrication of thin slices. An alternative technique is scanning electron microscopy that enables probing solid structures with the spatial resolution of units of nanometers both in the lateral and longitudinal (in−depth) directions [1]. Thicknesses of ultrathin films on bulk substrates are measured in a scanning electron microscope (SEM) with detecting fluxes of backscattered electrons (BSE) [2] or their energy spectra [3–5]. However, all these methods are applicable only to single−layer coatings, i. e. they are restricted to the „film-on-substrate“combination. This paper proposes a method for determining not only film coating thicknesses but also depths of the subsurface elements occurrence in multilayer structures. The experiments were performed by SEM in the BSE mode at the incident electron energy of 2 to 30 keV. An important property of BSEs is their relatively large escape depth amounting up to hundreds of nanometers. Primary electrons with energy $E_B = E_1$ penetrate into the sample to depth R_0. A part of electrons undergo reflection at depth x_1; moving along the forward and backward trajectories, BSEs lose some energy. Designate the energy loss coefficient as α:

$$\alpha = \langle E\rangle / E_B,$$

where $\langle E\rangle$ is the mean BSE energy. Primary electrons with energy $E_B = E_{B2} > E_{B1}$ undergo reflection at a larger depth x_2. If the flux integrated over all the depths and angles is measured within solid angle Ω, then signal I from the semiconductor detector is generally defined as follows [1,6]:

$$I = I_B \eta \alpha E_B E_i^{-1}\,\Omega \mathrm{F} = K\eta\alpha F, \tag{1}$$

where η is the coefficient of BSEs from the sample, $K = \mathrm{const}$, E_i is the energy of carrier−pair creation in the detector material, F is the detector response function, $F = (1 - \eta_{\mathrm{Si}}\alpha_{\mathrm{Si}})\big(1 - \frac{E_{th}}{\langle E\rangle}\big)$. Here $\eta_{\mathrm{Si}}\alpha_{\mathrm{Si}}$ is the flux of electrons reflected by the Si−detector, $E_{th} = 1$ keV is the electron loss in the „dead“ face electrode of the Si−detector. In the experiments, parameter $I_B E_B$ was kept constant, i. e., when E_B was varied from 2 to 30 keV, probe current I_B was set to a value ranging, e. g., from 0.1 to 3 nA.

First the SEM gray screen scale will be calibrated in arbitrary units from 0 to 1.0. This gradation allows signal differences of 2% to be distinguished. Detector signal I_{0s} can be controlled in the above−specified range by setting two parameters (level L and contrast C) on two bulk test samples:

$$I_{0s} = L + C(\eta_0\alpha_0)F. \tag{2}$$

Factor $\eta_0\alpha_0$ will be calculated for each test material using the following formulae [6–8]:

$$\eta_0(Z) = \exp\big[-(6.24Z^{-0.5})\big], \quad \alpha_0 = 0.47(1 + 1.4\eta_0), \tag{3}$$

where Z is the atomic number.

In our experiments, the test multilayer structure (Fig. 1) consisted of a bulk Si substrate coated with three layers of gold d in thickness (in the case of gold, $\eta_0 = 0.5$ and $\langle E\rangle/E_B = 0.8$). The structure top was partly covered with an aluminum film with thickness t. To find unknown L and C in (2), two equations for reference Si and Au samples at two selected energies E_{B1} and E_{B2} were used. Using

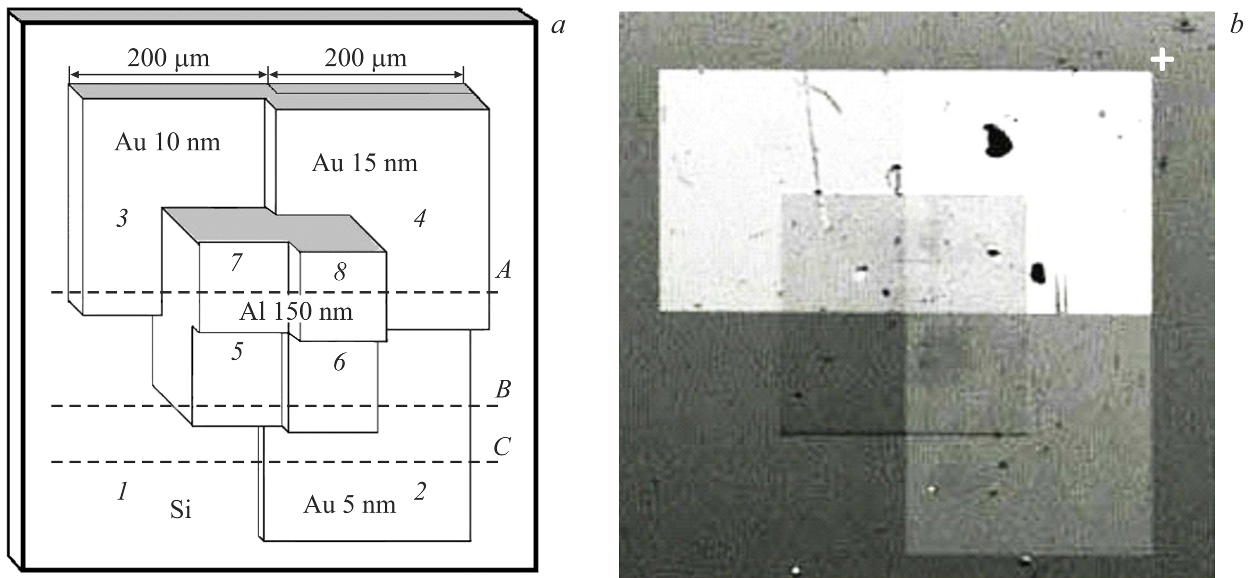

Figure 1. *a* — schematic diagram of the calibrated three–layer test sample (digits *1–8* designate the structure sections), *b* — is the test sample BSE image at $E_B = 15$ keV.

formula (2), we can find the L and C values from the equations for I_{01} and I_{02}. For the samples each being a combination of a film d in thickness on a substrate (in this case, an Au film on a Si–substrate), formula (2) takes the following form:

$$I_{sf} = 0.3 + 1.5(\eta_{sf}\alpha_{sf})F,$$

where η_{sf} and α_{sf} are the coefficients of reflection and mean BSE energy for the given probing point. Values of η_{sf} will be determined using formula [9,10]:

$$\eta_{sf} = \left[\eta_{0s} + (\eta_{0f} - \eta_{0s})(\eta_f/\eta_{0f})\right], \tag{4}$$

where η_{0s} is the BSE coefficient for the bulk substrate material, η_{0f} is that for the bulk film material, η_f is that for the free coating film d in thickness. The last value will be obtained from the empiric relation [7,8]:

$$\frac{\eta_f}{\eta_{0f}} = 1 - \exp\left(-A\left(\frac{d}{R_0 - d}\right)^p\right),$$

$$R_0 = 74E_B^{1.55}\rho^{-1}, \tag{5}$$

where R_0 is the primary electron range in the film material with specific density ρ (taken from [11]). For the test structure under consideration, we obtained $R_0(\text{Au}) = 3.83E_B^{1.55}$, $R_2(\text{Al}) = 27E_B^{1.55}$ (R_0 is expressed in nm, E_B is given in keV). Parameter A in (5) defines the mean reduced depth of the BSE reflection x_c [8]:

$$A^{-1} = x_c/R_0 = 0.52\exp\left[-0.022(Z+2)\right]. \tag{6}$$

The exponent power p in (5) determined in [4,7] is $p = 1.12\eta_0^{-0.333}$. Let us also take into account that, when BSE are detected at the mean escape angle $\theta = 45°$, their total path (in both the forward and backward direction) is

$$s = 0.5x_c\left(1 + \frac{1}{\cos\theta}\right) = 1.207x_c.$$

Therefore, it is necessary to make the following correction to factor A (formulae (5) and (6)): $\frac{x_c}{R_0} = 0.106$; $A = (1/0.106)^{1.41} = 23.7$ for Au and $A = 4.97$ for Al. Finally, obtain the formula for calculations:

$$\eta_f/\eta_{0f} = 1 - \exp\left[23.7\left(d/(R_0 - d)\right)^{1.41}\right]. \tag{7}$$

For the considered test sample, values of I_s will be taken from the Au–Si sections designated in Fig. 1 by digits *2–4*. Fig. 2 presents examples of records obtained in scanning along lines A and C at two energies E_B. Based on experimental values of I_s taken from sections *2–4*, the required values of d will be obtained via the above formulae. Fig. 3 presents the dependences of signals I_2, I_3 and I_4 on E_B. The following Au film thicknesses were obtained: $d_1 = 5.00 \pm 0.25$ nm, $d_2 = 10.0 \pm 0.5$ nm, $d_3 = 15.0 \pm 1.0$ nm. The sample structure in section *5* is as follows: the Si–substrate is coated by an Al layer with thickness $t = 150$ nm. This example demonstrates the methodś ability to distinguish different–type structures whose atomic numbers differ from each other only by unity. The procedure for determining film thicknesses in three–layer structures is more complicated. In the case of a test structure under consideration, the role of the substrate was played by the „Au film on Si-substrate“combination which, in its turn, was partly covered by an aluminum film t in thickness (Fig. 1). Here it is necessary to

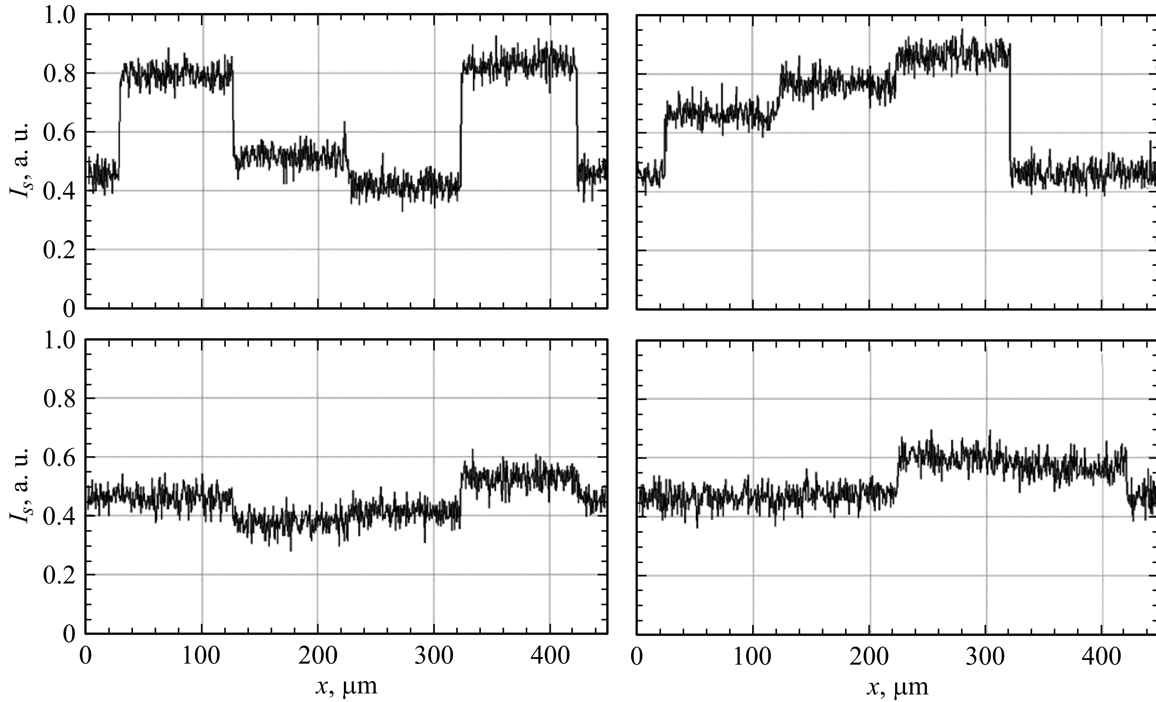

Figure 2. Records of the test structure scanning along lines A (left) and C (right) (see Fig. 1) measured at energies $E_B = 7$ (top) and 20 keV (bottom).

notice two important factors taking place during interaction between the incident electron beam and described sandwich structure. First, a part of primary electrons is absorbed by the aluminum coating film; due to this, the composite Au–Si substrate is bombarded not by the I_B flux but only by its part $I_B\eta_t$, where η_t is the electron absorption coefficient. Second, this flux has lower energy. Hence, the complex substrate is irradiated by a flux with mean energy $\langle E_t\rangle = \beta_t E_B$. As per [8], those coefficients for the aluminum film are

$$\eta_t = \exp\left[-4.6\left(t/R_2\right)^2\right], \quad \beta_t = 0.95\exp\left[-\frac{t}{R_2}\right], \qquad (8)$$

where R_2 is the electron free path in Al. A decrease in energy E_B by β_t times results in that the primary electron range in the „Au film on Si-substrate" system is not R_0 as it was earlier determined for the sample sections free of aluminum but has a lower value $R_1 = 3.83\langle E_t\rangle^{1.55}$. As a result, relations (5)–(7) get transformed with replacing R_0 with R_1 and introducing factor η_t defined by (8). Thus, equations for calculating signals I_s for the structure containing the double layer on the substrate include two unknown parameters: d and t. Hence, it is necessary to use two equations for I_s calculated for the given structure point at two energies E_B, i.e. $I_1(E_{B1})$ and $I_2(E_{B2})$. Then the required film thickness parameters d and t will be found from two equations. Calculated and experimental values of signals $I_s(E_B)$ taken from sections *6–8* are represented in Fig. 3 by curves I_6, I_7, I_8. The results confirm the validity of the model proposed for calculating thicknesses d and t

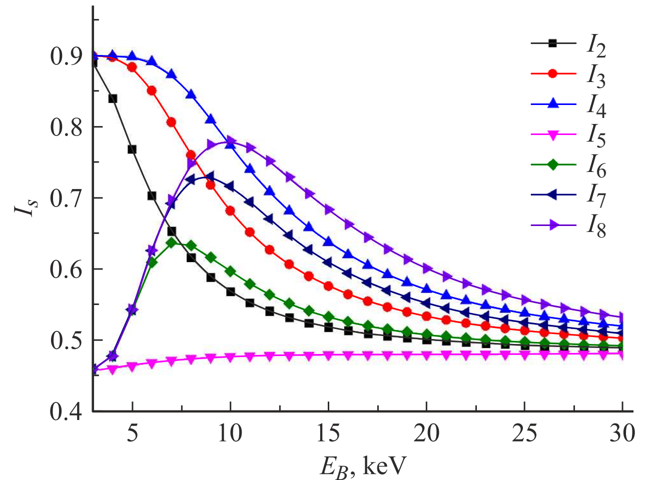

Figure 3. Calculated (lines) and experimental (points) dependences of signals I_s on the primary electron energy E_B for the relevant structure sections shown in Fig. 1. Values of I_s are normalized by the detector response function F_{sf}.

of the double thin–film layer on the substrate. Deviations between the calculations and measurements do not exceed 10%. Curves for the „film-on-substrate" system presented in Fig. 3 demonstrate an inversely proportional dependence of signal I_s on energy E_B and directly proportional dependence on thickness d (see curves I_2, I_3, I_4). However, those dependences for the double layer on the substrate are non–monotonic and have maxima (see curves I_6, I_7, I_8).

Moreover, there was revealed a new effect consisting in enhancement of the signal from the buried under−surface layer with respect to the signal from the uncovered layer on the substrate. This unexpected phenomenon needs additional independent investigation.

Thus, an algorithm is proposed for the first time for determining not only thicknesses of nanometer elements of multilayer structures but also the depth of their occurrence beneath the surface. The spatial resolution in the lateral sample-scanning plane in SEM is governed in the first approximation by the probe diameter and equals units or tens of nanometers. The longitudinal resolution „in depth" of the structure reaches tenths and units of nanometers. The presented method may be extrapolated to any multilayer nanostructures.

Conflict of interests

The authors declare that they have no conflict of interests.

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