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MODIFIED BACKSCATTERING METHOD OF THE NANOMETER SELF-SUPPORTING FILMS AND SURFACE LAYERS THICKNESS MEASUREMENTS

Monitor-interrupter of accelerated ions beam is used in the backscattering standard scheme. The procedure of the thickness measurement requires getting of two spectra now. First spectrum is obtained for investigated target and second one for the target, which imitates a thick substrate. The monitor-interrupter system ensures the same expositions for both spectra. If the substrate is from the same chemical element as the investigated target, the formula for the thickness calculation is simplified. The method is applicable for both self-supporting films and surface layers.

Keywords: backscattering, monitor-interrupter, self-supporting film, surface layer, imitating substrate, thickness.

Introduction

It has been accepted that the nanometer scale of material dimensions used in the nanotechnology includes the region from atomic dimensions to about 100 nm (although for one of three measurements). Only thickness will be considered in this consideration. Self-supporting films and surface layers had become as object of our study. Of the many known methods of the thickness measurements we focus on non-destructive nuclear-analytical method using elastic backscattering of light ions of the MeV energies.

Theory of backscattering spectrometry [1, 2] in application to the thickness measurements of thin films and surface layers is based on the so-called surface energy approximation. In this approximation the energy dependence of differential cross section is neglected. The approximation works very well if there is only the Rutherford scattering. But for the light ions the non-Rutherford scattering for projectile-target combinations may occur. In this case it is necessary to use experimental cross-sections, which are not complete and contain errors. Therefore the first purpose of such investigation is to by-pass this defect and to not include the cross-section parameter in the explicit formula of the thickness calculation.

But the finite formula includes another two sensitive quantities. They are: total number of incident particles on the target and ~~a~~ solid angle of detection. They require the special precise measurement. Therefore, the absolute calibration of the system is necessary and the thickness measurements are complicated. For special cases there are approximate approaches, which simplify the measurement procedure. That is the case, when thickness of the thin surface layer of a heavy chemical element on a light substrate is measured. It has been showed, that for such combination the product of quantities of incident particles on a target and solid angle of detection can be expressed through the spectrum scattering height from the substrate surface layer. So that other purpose of our investigation is to widen this approach for the case of the thickness measurement of thin self-supporting films and for the nuclear scattering. Finally, the paper presents examples of the practical measurements.

Principle of the method

Scheme of the backscattering experiment is showed in Fig. 1.

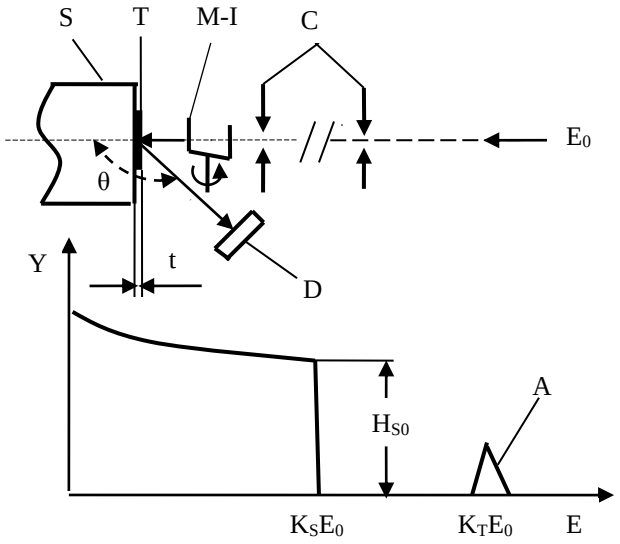

Fig. 1. Scheme of the backscattering experiment and Rutherford backscattering spectrum.

Difference of this scheme from the standard one is introduction of a supplementary element. This is the monitor-interrupter (M-I) of the formed beam, which is placed between collimator (C) and investigated target (T). The beam is interrupted periodically with high frequency. The typical spectrum of the Rutherford scattered ions is also

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schematically showed. Such spectrum corresponds to a thin surface layer of heavy chemical element with mass T on a surface of light mass substrate S. The area A (in units of counts number) of the surface layer signal contains information about the quantity of target atoms, therefore about the target thickness. The pick location on the energy scale is labeled by K_TE_0, where K_T is the kinematic factor for the angle scattering θ and E_0 is the initial ions energy.

The area A is dependent from some quantities:

$$A = \sigma_T(E_0)\Omega Q(Nt)_T, \quad (1)$$

where $\sigma_T(E_0)$ is the scattering cross section (valued in the surface energy approximation) for the E_0 energy; Ω is the solid angle of detection, Q is the total number of particles incident on the target, N is atomic density in of the target, t is the target thickness. The $(Nt)_T$ product is ~~the~~ amount of the target atoms per unit area. Let us analyze the Eq. (1). The $\sigma_T(E_0)$ value is given by the Rutherford formula. But it is necessary to take into account the scattering cross section from experiment if the nuclear scattering occurs. The Ω and Q values ~~must~~ have to be measured separately. This presupposes an absolutely calibrated system. There is another way to determine $(Nt)_T$. The use of the height H_{S0} for scattering from the surface of the substrate S is characteristic in this case. The subscript 0 refers to the surface energy approximation. It was shown [1, 2] that it can be expressed as

$$H_{S0} = \sigma_S(E_0)\Omega Q\delta E/\left[\varepsilon_0\right]_S. \quad (2)$$

Here $\sigma_S(E_0)$ is the cross section for scattering from the substrate nuclei and δE is the energy width of one channel in the spectrum. The stopping cross section factor $\left[\varepsilon_0\right]_S$ for backscattered particles, evaluated for the region near the surface and for a normally incident beam, is calculated as

$$[\varepsilon_0]_S = K_S\varepsilon(E_0) + \frac{1}{|\cos\theta|}\varepsilon\left(K_SE_0\right). \quad (3)$$

The quantities $\varepsilon(E_0)$ and $\varepsilon(K_SE_0)$ are stopping cross section evaluated at energies E_0 and K_SE_0, respectively. K_S is the backscattering kinematic factor for the substrate nuclei. The ΩQ product is determined from Eg. (2). Substituting the ΩQ value into Eg. (1) the expression for $(Nt)_T$ is obtained in the form

$$(Nt)_T = \frac{A}{H_{S0}}\frac{\sigma_S}{\sigma_T}\frac{\delta E}{\left[\varepsilon_0\right]_S}. \quad (4)$$

Let us consider what can provide additional block of the monitor-interrupter (M-I) in the scheme (see Fig. 1). The thickness measurement procedure requires the receiving of two spectra now. The first spectrum is obtained for target under investigation. It is important that this target may be self-supporting. The second one is obtained for a thick target which imitates the substrate. This target is placed with help of a manipulator on the spot of the investigated target. The geometry of the both experiments is the same. The substrate target may be from the lighter or heavier chemical element than the target under investigation. There is one requirement only. The total number Q of particles incident on the targets must be the same for both spectra. The M-I block fulfils the control function. A part of the beam falls on the rotating insulated plates of the block. The chard is taken off from rotatable platform of the M-I block. It is substantially, the secondary electrons emitted from the plates of the interrupter is the same at setting of both spectra. The spectra setting are stopped automatically after the command from the current digitizer. The ΩQ product is equal for the both experiments. Thus the Eqs. (2) and (4) can be used, where H_{S0} is the spectrum height for the scattering from a top surface layer of the thick target now. The arbitrary choice of the thick target has the advantages not only for the thickness measurement of thin self-supporting foils. Heaving the information about the energy dependence of the scattering cross section for the selected scattering angle one can choose the energy of accelerated particles in such a way that the cross sections vary smoothly even for the non-Rutherford scattering as for nuclei of a thick substrate and for nuclei of a film or a surface layer. This diminishes the error of cross section choice from the experimental date. Though the error is still retained.

The greatest advantage of the measurement procedure with ~~the~~ getting of the two spectra is possibility of displaying when an imitating substrate is selected from the same chemical element as an investigated target. The Eq. (4) is simplified at such choice

$$(Nt)_T = \frac{A_T}{H_{S0}}\frac{\delta E}{\left[\varepsilon_0\right]_S}. \quad (5)$$

Therefore the influence of a systematic error of the cross section choice disappears. Moreover, the scattering on the surface of the imitating substrate occurs at the energy E_0 exactly. There is no need to neglect the energy loss of particle passing across the investigated target. It should be also indicated that the spectrum for the imitating substrate may be ~~got~~ obtained only once and use for series of the measurements. For instance it can be measurement

of the sample thickness uniformity or the thickness measurement of the same type samples.

It should be noted that Eq. (5) is actually analogous to that used at the study of disorders in the crystal lattice [1]. In this case the ions channeling effect in crystals is used and the backscattering spectra are possible for both the random and aligned orientation of a damaged crystal.

Testing of the method

Measurements have been carried out to test the analytical method first of all. At the same time, we would like to obtain the necessary information about the thickness of carbon self-supporting foils as the stripper targets in the Tandem accelerator [3] and to measure the thickness of a titanium surface layer of the titanium-silicon structure. The experiment was performed using the 3.50 MeV proton beam from the EPG-10K Tandem accelerator (Kyiv, Ukraine) and the KANION installation [4]. The KANION installation was assigned for applications of channeling effects of energetic ions interacting with single-crystals. We have made use of this technique with some changes to fulfill the routine backscattering experiment more precisely. Changes relate to the procedures of the beam formation and its monitoring, the target orientation relatively beam, selection of the irradiation place on the target surface and selection of the necessary scattering angle.

Two spectra are shown in Fig. 2. They correspond to scatterings of protons incident on: a) a thin carbon self-supporting target and b) a thick target of carbon. Targets are from the same carbon sample, and the conditions of measurement were identical. The scattering angle in this experiment was constant and equal to 165º (in laboratory coordinate system). The scattering of protons from carbon in the energy interval from about 2.0 to 4.7 MeV is free from resonances and the cross section is equal to ~ 0,1 b/sr [4]. The calculation of the carbon self-supporting film thickness with use of the Eq. (5) gives value of $(7.4 \pm 0.6)\cdot 10^{17}$ atoms/cm^2 (statistical error is indicated). This is equivalent to ~ (9.7 ± 0.8) μg/cm^2 or ≈ 42 nm. Such thickness is too large for transmission of protons through the accelerating tube. According to [6] the thickness of ~ 5 μg/cm^2 is optimal even at the terminal voltage of 5 MV.

Since in practice the uniformity of the foil thickness is the important parameter, the supplementary measurement has been performed too. The result of such measurement is illustrated in Fig. 3. Average value of thickness is equal to $(9.6 \pm \pm 0.8)$ μg/cm or $(7.3 \pm 0.6) \cdot 10^{17}$ atoms/cm^2. The non-uniformity of thickness on the investigated area of the target does not exceed the error of experiment.

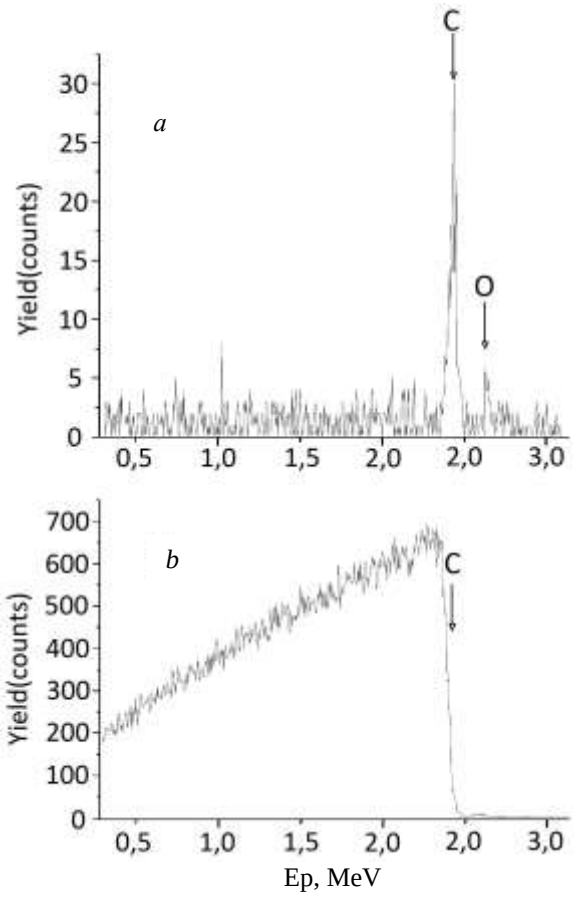

Fig. 2. Backscattering spectra for 3.5 MeV protons, and angle θ_{lab} = 165º:
a - carbon stripper target; *b* - thick carbon target.

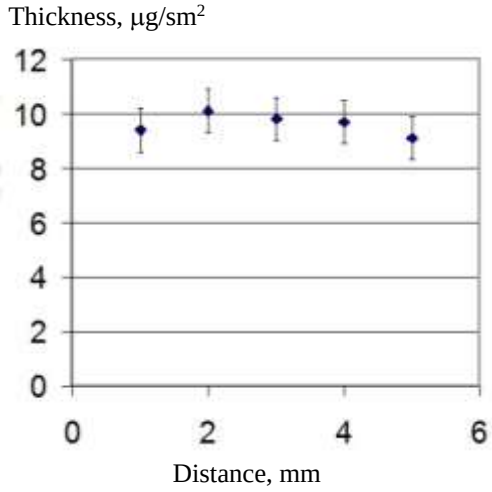

Fig. 3. Dependence of the stripper target thickness from the place of its surface.

For testing of method we have also measured the thickness of titanium surface layer on the silicon substrate. Two spectra shown in Fig. 4 correspond to scatterings of protons from: a) the thin surface layer of titanium on the silicon substrate and b) the thick target of titanium. The known narrow resonances at energies of 3.337 and 3.100 MeV and the broad destructive resonance at 2.880 MeV [7] are seen

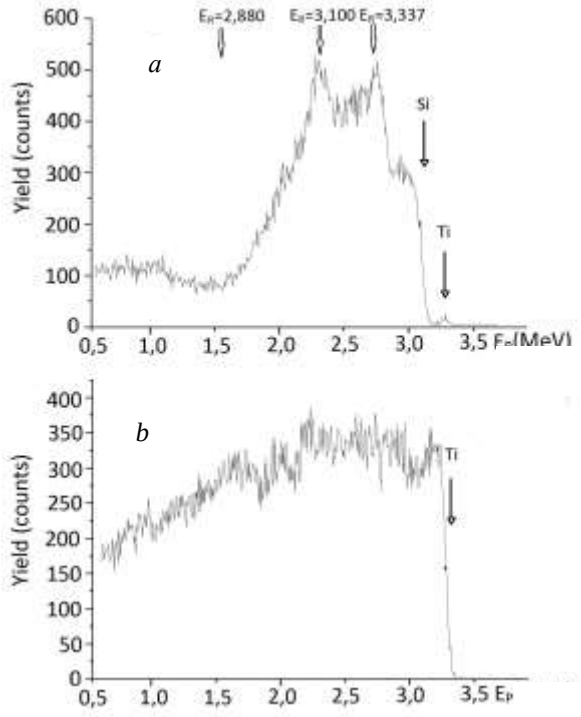

Fig. 4. Backscattering spectra for 3.5 MeV protons and angle $\theta_{lab} = 165^{o}$:
a - titanium-silicon structure target;
b - thick titanium target.

at the conditions the elastic scattering of protons on silicon shows (see Fig. 4, *a*). The vertical arrow indicates the scattering from surface atoms of silicon. The signal of the elastic scattering of protons on titanium is visible in the region of 3.28 MeV. Falling on the middle of the scattering spectrum from the titanium thick target corresponds to 3.28 MeV too. Calculation of the titanium surface layer thickness using the Eq. (5) gives the value of (17.5 ± 1.9) μg/cm^2 or $(2.2 \pm 0.24) \cdot 10^{17}$ atoms/cm^2.

Conclusion

The principle of the modified backscattering method of the thickness measurement of nanometer self-supporting films and surface layers is suggested. Applicability of the method has been confirmed experimentally. The obtained experimental result concerning the carbon stripper target thickness for EPG-10K tandem accelerator is too large in comparison with optimal one. It was suggested to introduce corrections in the technology manufacturing of such targets.

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ВИДОЗМІНЕНИЙ МЕТОД ЗВОРОТНОГО РОЗСІЯННЯ ВИМІРЮВАННЯ НАНОМЕТРИЧНИХ ТОВЩИН САМОПІДТРИМУЮЧИХ ПЛІВОК ТА ПОВЕРХНЕВИХ ПОКРИТТІВ

До типової схеми зворотного розсіяння додається монітор-переривник пучка прискорених іонів. Процедура вимірювання товщини тепер потребує набору двох спектрів. Один спектр отримується для досліджуваної мішені, а другий – для мішені, що імітує товсту підкладку. Однакові експозиції при наборі обох спектрів забезпечуються монітором-переривником. Якщо підібрати підкладку з того ж самого хімічного елемента, що й досліджувана мішень, то кінцева формула для розрахунку товщини значно спрощується. Сфера застосування методу поширюється як на самопідтримуючі плівки, так і на поверхневі шари.

Ключові слова: зворотне розсіяння, монітор-переривник, самопідтримуюча плівка, поверхневий шар, імітуюча підкладка.

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ВИДОИЗМЕНЕННЫЙ МЕТОД ОБРАТНОГО РАССЕЯНИЯ ИЗМЕРЕНИЯ НАНОМЕТРИЧЕСКИХ ТОЛЩИН САМОПОДДЕРЖИВАЮЩИХ ПЛЕНОК И ПОВЕРХНОСТНЫХ ПОКРЫТИЙ

К типичной схеме обратного рассеяния добавлен монитор-прерыватель пучка ускоренных ионов. Процедура измерения толщины теперь требует набора двух спектров. Один спектр регистрируется для исследуемой мишени, а второй – для мишени, которая имитирует толстую подкладку. Одинаковые экспозиции при наборе обоих спектров обеспечиваются монитором-прерывателем. Если подобрать подкладку из того же самого химического элемента, что и исследуемая мишень, то конечная формула для расчета толщины значительно упрощается. Область применения метода распространяется как на самоподдерживающие пленки, так и на поверхностные слои.

Ключевые слова: обратное рассеяние, монитор-прерыватель, самоподдерживающая пленка, поверхностный слой, имитирующая подкладка.

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