

**ELASTIC NUCLEAR RESONANCE BACKSCATTERING SPECTROMETRY
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The possibilities of using of the elastic nuclear resonance backscattering of ions for the investigation of materials are analyzed. Broad resonances having the cross-section varying smoothly in large energy range are considered. Under this condition the simplicity of information extraction inherent in the Rutherford backscattering technique is retained. Concurrently, the detection sensitivity for low-mass impurities is improved. The elastic nuclear resonance scattering reaction is always accompanied by the Coulomb and potential scatterings. The above components of the elastic scattering are coherent and therefore interfere. This distinction of the reaction of this type is also revealed in applying resonant scattering for materials analysis. Depending on level parameters of the compound nucleus the interference may be constructive (the cross-section is boosted) or destructive (the cross-section is attenuated). To improve the sensitivity the resonant scattering by impurity nuclei is used in the first case while in the second case the resonant scattering by substrate nuclei is applied (to decrease the background). The physical principles are confirmed by the examples of the impurities investigation of nitrogen in beryllium and oxygen in silicon.

1. Introduction

The nuclear analytical method of the Rutherford backscattering [1] has acquired large application because of its unique analytical power. At present its technique improvement continues mainly through the enhancement of the energy resolution of the detecting system. The depth resolution of the method depends on the detecting system resolution directly however the detection sensitivity does not concurrently rise. To increase the sensitivity of the backscattering method the nuclear resonant scattering using both narrow (≤ 20 keV) and broad resonances came into use. Further we focus on the broad isolated or overlapping resonances whose scattering cross sections vary smoothly over the wide energetic range. Such resonances are not very numerous. Selected data on the low-energy ions elastic scattering [2] may be a good reference in the resonance choice in each specific case.

In the fundamental of Rutherford backscattering spectrometry lies the law of electrostatic interaction between charged bodies (Coulomb's law). Rutherford's formula for the differential cross section demonstrates the simple charge (Z^2) and energy (E^{-2}) dependences. It considerably simplifies the analysis procedure. But with the increase of energy a moment comes for each ion-nucleus pair when the kinetic energy (c. m.) exceeds the Coulomb barrier and nuclear forces begin to have effect on the scattering. A theoretical model of the classical scattering in the combination of Coulomb and disturbed nuclear fields was suggested. The model allows calculating the ion energy when the elastic backscattering cross-sections begin to deviate from their Rutherford values [3, 4].

The main limitation of the Rutherford backscattering spectrometry consists in the

difficulties that occur in the investigation of a low atomic mass impurity within of a higher atomic mass substrate because the more weak impurity signal is superimposed on the strong substrate signal. The nuclear reaction technique or the elastic recoil nuclei detection method enables to overcome these difficulties. But in both cases the analysis is much more complicated and requires more time. The nuclear resonant scattering method can be used to improve the detection sensitivity for low-mass impurities as well. But in this case relative simplicity of the information extraction inherent in the Rutherford backscattering method retains.

Research on the issue began in the late 1980s [5 - 7] and in 1990s [8 - 10]. As long as there is no general theory describing the nuclear reaction cross-section and the levels scheme the experimental information must be used in the analysis. It should be noted that the more accurate data on the reaction cross-sections and the resonance parameters were found to be necessary for practical purposes than for nuclear physics itself. That is why repeated measurements were carried out (for protons [11 - 15] and alpha-particles [12, 16 - 20], for instance).

In this paper the physical principles of the broad resonances use in the solution of nuclear microanalysis problems are considered. The principles are confirmed by practical examples of the analysis.

**2. Use of broad resonances in elastic scattering
by impurity nuclei****2.1. Physical principles**

The nuclear reaction of the elastic scattering is specific among the other reactions being always accompanied by Coulomb and nuclear potential

scatterings. The above components of the elastic scattering are coherent and interfere. Because of interference the resonance form depends both on quantum characteristics of a compound nucleus level and on a scattering angle. Interference may be either constructive (the scattering cross-section increases) or destructive (the cross-section decreases). In this connection the physical principles of the broad resonances use fall into two groups. We relate the resonant scattering on impurity nuclei to first group and the scattering on the substrate nuclei to the second one.

The Rutherford backscattering spectrometry is known to be based on four fundamental physical concepts [1]. They are: the kinematic factor, scattering cross-section, stopping cross-section and energy straggling. When the nuclear scattering is used it is only the cross-section scattering concept that has the different than the Rutherford backscattering meaning. The other ones take no modification. Let us consider what new features the presence of the nuclear interaction brings into the backscattering spectra interpretation problem, specifically, into the question of the connection between the height of the energy spectrum and the number of scattering centers per unit area within the sample, where the backscattering occurs. The spectrum height $H(E_i)$ for the scattering at a certain depth of a sample can be presented as [21]

$$H(E_i) = \sigma(E)\Omega Q \frac{\delta E}{[\varepsilon(E)]\cos\theta_1} \frac{\varepsilon(KE)}{\varepsilon(E_1)}, \quad (1)$$

where E_i is the energy of the detected particle, E is the energy of the particle immediately before the scattering, $\sigma(E)$ is the cross-section at the energy E and averaged over the solid angle Ω spanned by the detector, Q is the total number of the particles incident on a target, δE is the energy width of the analyzer channel, $[\varepsilon(E)]$ is stopping cross-section factor for backscattering at the energy E , $\varepsilon(KE)$ and $\varepsilon(E_i)$ are stopping cross-sections at the energy KE and E_i , K is kinematic factor. Naturally, when using the nuclear scattering the requirements to the E determination are substantially increased because in this case the cross-section dependence from energy is stronger than for Rutherford scattering and, principally, it does not conform to certain regularity. It should be noted that the spectrum height depends on energy not only in terms of the scattering cross-section $\sigma(E)$. It is inversely proportional to factor $[\varepsilon(E)]$ and consequently to the stopping cross-section $\varepsilon(E)$ that depends on the energy as well. This confirms the necessity for the more exact determination of the energy of the particles before scattering. As in Rutherford backscattering

spectrometry different methods for finding E are used [1].

It is clear that taking into account the nuclear scattering in the case of broad resonances leaves the equations of the theory of Rutherford backscattering spectrometry unchanged. We will not cite these numerous equations. Let us have only the formula necessary for further calculation of the impurity content N_i per square centimeter on the substrate surface

$$N_i = \frac{A_i}{H_M} \frac{\sigma_M(E)}{\sigma_i(E)} \frac{\delta E}{[\varepsilon]_M^M}, \quad (2)$$

where the indexes M and i refer to the substrate and impurity respectively, A_i is the area (in counts) under an impurity peak of the backscattering spectrum, H_M is the spectrum height for scattering from the surface of the substrate, $\sigma_M(E)$ is the scattering cross-section at the energy E by a substrate nucleus, $\sigma_i(E)$ is the scattering cross-section at the energy E by an impurity nucleus, δE is the energy width of one channel of the detecting system, $[\varepsilon]_M^M$ is the stopping cross-section factor with the subscript to denote the scattering nucleus and the superscript to denote the stopping medium.

2.2. Investigation of nitrogen and other impurities in the top surface layers of beryllium

The idea of using of broad resonances at elastic scattering by impurities nuclei has arisen in connection with the practical necessity to study samples of beryllium and its alloys [5]. Nowadays beryllium has rather wide application in various branches of science and technology. Since impurities presence has strong effect on the properties of beryllium products there is reasonable interest in their investigation.

To have the general notion about a sample under study at first the backscattering spectra (in standard and tilted geometries) are acquired. In experiments we used the α -particles beam in the energy interval 2.0 - 3.4 MeV. Fig. 1 shows one of the spectra received at such conditions: α -particles beam of incident energy $E_0 = 2.2$ MeV, scattering angle $\theta_{\text{lab.}} = 164^\circ$ (standard geometry). Positions of impurities were identified through of calculations.

The study of nitrogen became the focal point of the experiment. Both nitrogen and oxygen are present in nearly all solids that come in contact with air. The nitrogen influence may be different depending on its reaction with matter and its penetration into depth. Therefore, analysis of the nitrogen content and distribution requires of adequate methods [22]. Among a variety of nuclear

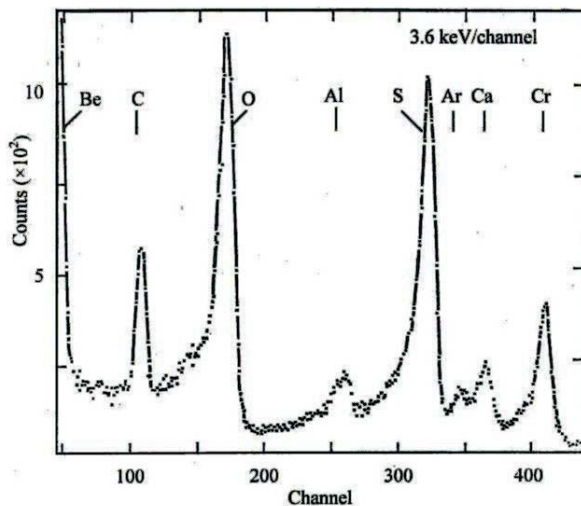


Fig. 1. Backscattering spectrum of alpha-particles from the beryllium sample with impurities ($E_0 = 2.20$ MeV, $\theta_{\text{lab}} = 164^\circ$, standard geometry).

reactions in the ^{14}N and ^{15}N nitrogen stable isotopes one can specify some that are actually used for analysis: $^{14}\text{N}(p,p)^{14}\text{N}$ [23], $^{14}\text{N}(d,p)^{15}\text{N}$ [23], $^{14}\text{N}(d,\alpha)^{12}\text{C}$ [24-26], $^{14}\text{N}(\alpha,p)^{16}\text{O}$ [27], $^{14}\text{N}(\alpha,\alpha)^{14}\text{N}$ [27, 28], $^{15}\text{N}(p,\alpha)^{12}\text{C}$, $^{15}\text{N}(p,\alpha\gamma)^{12}\text{C}$ [29], $^{15}\text{N}(d,\alpha)^{13}\text{C}$ [30].

The use of the ions elastic scattering reaction in nitrogen in case of the beryllium substrate is advantageous because nitrogen is heavier than beryllium. Still scattering by other impurities heavier than nitrogen forms the background. Firstly, it is the scattering by carbon and oxygen that are the closest to nitrogen by the charge. The results of the oxygen analysis showed that its content in the top layers is within the limits $8.8 \cdot 10^{15} - 5.1 \cdot 10^{16}$ atoms/cm² and at the depth of ~ 500 Å its volume density does not exceed 10^{18} atoms/cm³. That is why even the absence of the peak caused by scattering of α -particles by nitrogen (see Fig. 1) does not discard its presence in general. Meriting attention is that the spectrum showed in Fig. 1 has been taken for 2.2 MeV α -particles at which the scattering can be considered Rutherford scattering proper.

To improve the detection sensitivity for nitrogen we decided to use α -particles resonant scattering in the region of broad resonances. The 3.1 - 3.5 MeV α -particles energy range seems to be especially suitable for that [31]. The cross-section here is determined by nuclear resonant and potential scatterings. Broad resonances lie beyond the indicated interval. Their resonance energies and widths are: $E_r = 3.08$ keV ($\Gamma = 100$ keV), $E_r = 3.67$ keV ($\Gamma = 45$ keV), $E_r = 3.72$ keV ($\Gamma = 3.72$ keV) and so on. Full angular momentums of the corresponding levels of the ^{18}F compound nucleus are such that the elastic scattering excitation

function in the 3.1 - 3.5 MeV interval at large angles forms the plateau. The scattering cross-section attains 90 mbn and almost four times exceeds the Coulomb scattering cross-section.

Fig. 2 shows the spectra for α -particles backscattered from a beryllium sample at the energies of 3.17, 3.044 and 2.2 MeV. At 3.17 MeV between the carbon and oxygen peaks the nitrogen peak is clearly visible. At $E_\alpha = 3.044$ MeV the nitrogen peak is absent. This is caused by the fall of cross-section in the region of the already mentioned resonance at $E_\alpha = 3.08$ MeV. At $E_\alpha = 2.2$ MeV the nitrogen peak is poorly displayed and it partially overlaps with the oxygen one.

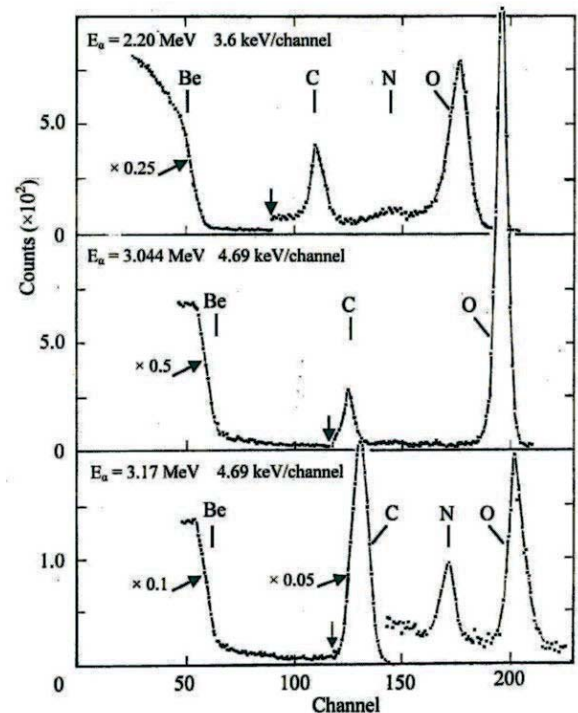


Fig. 2. Low-energy parts of backscattering spectra of alpha-particles scattered from the beryllium sample at the incident energies $E_0 = 2.20, 3.044$ and 3.17 MeV.

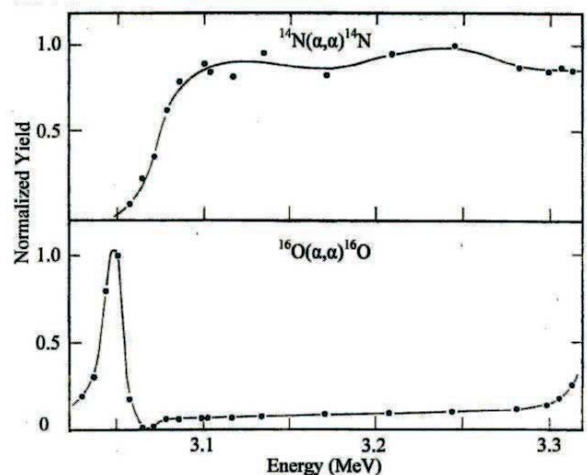


Fig. 3. Excitation functions of the alpha-particles elastic scattering yield from the surface impurities of nitrogen and oxygen into beryllium.

Fig. 3 shows the yield excitation function of the α -particles elastic scattering by nitrogen for the sample under investigation. Fig. 3 confirms the justification of the 3.1 - 3.5 MeV energy interval. The location of the bend at $E_\alpha = 3.08$ MeV in the yield curve also confirms that nitrogen is located in the surface region of beryllium.

Another circumstance remains to be rather a factor. Carbon, nitrogen and oxygen happen in fact always as the surface impurities. Depth profiles of their distributions may be such that their signals somewhat overlap each other. It is favorable for the nitrogen analysis in the case of the α -particles elastic scattering use that for oxygen the energy interval proposed above is not resonant one and the cross-section value is only slightly different from Rutherford one. The energy dependence of the relative yield of the α -particles elastic scattering by oxygen, that is in the surface region of sample under investigation, is showed in Fig. 3 (lower curve). The behavior of the yield curve and the position of the known resonance at $E_\alpha = 3.045$ MeV agree with the literature data on the cross-section. Scattering by carbon in the 3.0 - 3.5 energy range is defined by the very broad (~ 400 keV) resonance at $E_\alpha = 3.222$ MeV [33]. It is the resonance that causes the great difference of the carbon spectra heights at $E_\alpha = 3.044$ MeV and $E_\alpha = 3.17$ MeV (see Fig. 2).

Lastly, let us use the spectrum at $E_\alpha = 3.17$ MeV and the formula (2) of the theory for the nitrogen content calculation. Data on the α -particles scattering cross-section for beryllium and nitrogen (and the theoretical analysis of the data) are in the literature [31, 34, 35]. The multilevel approximation of the dispersion theory allows calculating the scattering cross-sections for a selected angle too. It should be noted, the ${}^9\text{Be}(\alpha, \alpha){}^9\text{Be}$ reaction has no resonances in the wide energy interval which exceeds the interval indicated above and the cross-section differs from Rutherford value a little. The stopping cross-section factor in the surface energy approximation and at standard geometry is evaluated from the equation

$$[\varepsilon]_{\text{Be}}^{\text{Be}} = K_{\text{Be}} \varepsilon(E_0) + \frac{1}{\cos \theta} \varepsilon(K_{\text{Be}} E_0), \quad (3)$$

where K_{Be} is the kinematic factor for beryllium, θ is the scattering angle (laboratory system of coordinates), $\varepsilon(E)$ is the stopping cross-section of beryllium at the energy E_0 and $K_{\text{Be}} E_0$. The results of the analysis showed that the content of nitrogen under investigation is $(9.8 \pm 1.7) \cdot 10^{15}$ atoms/cm².

3. Use of broad resonances in elastic scattering by substrate nuclei

3.1. Physical principles

So far analyzing the problem how to improve the detection sensitivity for low-mass impurities we

basically concentrated on how to increase the quantity of the useful signal. But the detection criterion is determined by the ration of the spectra heights from the impurity and the substrate. Thus decreasing the background signal the sensitivity of the impurity detection can be improved as well. Applying the Rutherford backscattering techniques with single-crystal substrates the channeling effect can be used to reduce the yield from the substrate [1]. The disadvantage of this way is that it can be used for single-crystal samples only. Moreover, because of the necessity to take the channeling effect into account, the experiment and the data treatment are complicated.

With the same aim, to improve the detection sensitivity of the backscattering technique for low-mass impurities, we offered to use the broad resonances in elastic scattering by the substrate nuclei [6, 7]. These are the resonances the excitation function in the region of which shows great destructive interference. Resonances with such energy dependence of the cross-section occur at scattering from light and medium nuclei [2]. Thus the task is practically reduced to the necessity to select the type and to calculate the energy of the accelerated particles so that particles, scattered by the light impurity nuclei and particles, scattered by the substrate nuclei in the region of the cross-section resonant minimum, acquire the same exit energy when they abandon the target.

To obtain the working formula we make use of Fig. 4 that gives schematically the backscattering process and spectrum. Symbols used in the description of backscattering events are: E_0 is the energy of the incident particles, x is the depth of the impurity location, E_m is the energy at which the elastic scattering excitation function from substrate nuclei passes the minimum for a given angle of scattering, E_l is the energy of the particles that emerge from the target and whose scattering by substrate nuclei was at the energy E_m , E is the energy at which there should be the scattering by impurity nuclei in order the emerging energy of particles would be E_l , x^l is the depth at which the particles energy is decreased to E_m . The listed quantity are connected by the equation

$$\begin{aligned} E_l &= K_l E - \frac{dE}{dx} \Big|_{K_l E_m} \frac{x}{|\cos \theta|} = \\ &= K_M E_m - \frac{dE}{dx} \Big|_{K_M E_m} \frac{x^l}{|\cos \theta|}, \end{aligned} \quad (4)$$

where

$$E = E_0 - \frac{dE}{dx} \Big|_{E_m} \cdot x, \quad E_m = E_0 - \frac{dE}{dx} \Big|_{E_m} \cdot x^l. \quad (5)$$

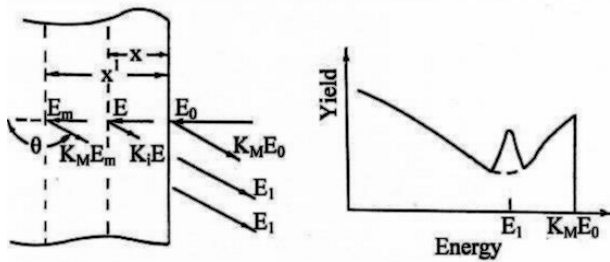


Fig. 4. Geometry and the ions backscattering spectrum in the case when the scattering excitation function for substrate nuclei shows large destructive interference (schematically).

The energy loss per unit length (dE/dx) for the substrate medium is evaluated for the inward and outward paths at the energy indicated near vertical line, K_i and K_m are the kinematic factors for scattering from atoms of the impurity and the substrate.

Simple transformation gives the formula for calculation of the necessary energy of the accelerated particles

$$E_0 = \frac{\left(K_M + \frac{dE}{dx} \Big|_{K_M E_m} / \frac{dE}{dx} \Big|_{E_m} |\cos \theta| \right) + [S]_I^M x}{K_i + \frac{dE}{dx} \Big|_{K_M E_m} / \frac{dE}{dx} \Big|_{E_m} |\cos \theta|}, \quad (6)$$

where the energy loss factor

$$[S]_I^M = K_I \frac{dE}{dx} \Big|_{E_m} + \frac{1}{|\cos \theta|} \frac{dE}{dx} \Big|_{K_I E_m} \quad (7)$$

is calculated for the case when stopping occurs in the substrate (M) and scattering takes place by impurity atoms (I).

Suggested method does not exclude the use of channeling effect for the sensitive improvement in the case of single-crystalline target. Evaluations show that in this case which is more general one can achieve the sensitivity improvement by an order of magnitude.

3.2. Investigation of oxygen implanted into silicon samples

In practice broad resonances in elastic scattering by substrate nuclei were used for detecting of oxygen implanted into silicon. The implantation energy of oxygen ions was 100 keV. The backscattering analysis was carried out with protons as projectiles.

It is known that in the elastic scattering of protons by ^{28}Si very broad resonance is displayed in the energy range 2.5 - 3.1 MeV [36]. Resonant scattering is caused by the ^{29}P compound nucleus level. The width of the resonance is approximately 400 keV and the total angular

momentum of the level is $\frac{1}{2}$. At these conditions and for great angles of scattering in the scattering cross-section a deep minimum is observed. The protons energy E_m that corresponds to the minimum of the excitation function equals 2.80 MeV for the scattering angle of 164° (in the laboratory coordinate system).

Fig. 5 shows a backscattering spectrum for one of the silicon samples implanted with oxygen. The spectrum has been taken for the $E_0 = 2,93$ MeV proton energy. This energy is slightly smaller than the calculation according to (6) gives. In Fig. 5 the spectrum for $\langle 111 \rangle$ -aligned orientation of 2.3 MeV protons incident on the Si single-crystal is also shown. In the 425 channel region there is a clear peak caused by scattering by oxygen. The insertion in Fig. 5 shows this oxygen peak isolated by experimental subtraction of the scattering spectrum from the silicon sample without implanted oxygen. It is clear, the background may be subtracted in other way. Just to facilitate the background subtraction the oxygen peak was shifted on the slope of the scattering excitation function for silicon by the choice of the incident proton energy.

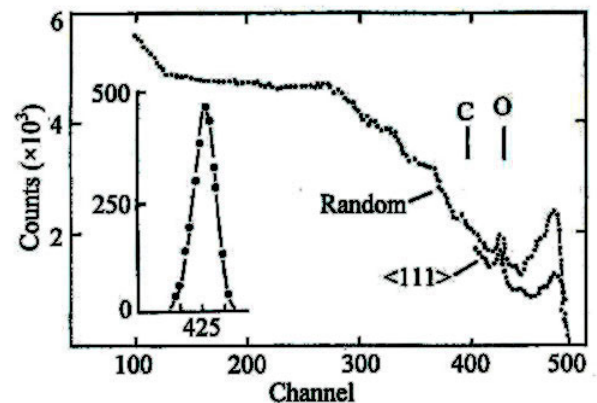


Fig. 5. Random and $\langle 111 \rangle$ -aligned spectra of protons scattered from the silicon sample implanted with oxygen ($E_0 = 2.93$ MeV, $\theta_{\text{Lab}} = 164^\circ$).

To determine the dose of oxygen implanted in silicon we shall again use the formula (2). The data on the proton scattering cross-section for silicon and oxygen can be picked from the [36, 37]. The cross-section can be also calculated within the scope of the dispersion theory because the levels parameters of the compound nuclei are known [38, 39]. The computation for oxygen gives the dose of $(2.8 \pm 0.3) \cdot 10^{17}$ atoms/cm 2 .

In the rest of this section we give another example of the possible use of the cross-section resonance decrease. The example is very interesting in practical aspect. In elastic scattering of alpha-particles by carbon there is broad ($\Gamma_{\text{c.m.}} = 400$ keV) resonance at 3.32 MeV [33]. For some angles of backscattering the cross-section is approximately zero. It is known that carbon is especially suitable

for coating of internal walls of the thermonuclear plasma installations since it has small atomic number and large thermal stability. But carbon has same disadvantages: (i) considerable containment of the hydrogen isotopes and (ii) considerable erosion as a result of ion bombardment under high temperature. Beryllium and boron have yet lower atomic numbers but their thermophysical properties are worse. Therefore in practice combined coating is applied. There is evident necessity to monitor the beryllium or boron deposition on graphite and to know its redistribution, in particular, the formation of beryllium carbide and oxide. This is one of the tasks successfully solved by different nuclear microanalysis methods. The published work [40] was dedicated to the comparison of the known alternative possibilities of the solution of this task.

4. Conclusion

The ions elastic scattering being one of the types of nuclear reactions, it is necessary to consider the

analytical method based on this reaction type as a variety of the nuclear reaction method. But the elastic scattering nuclear reaction has some unique peculiarities. In the first place they are the identity of the input and output channel of the reaction and the equality to zero of the reaction energy. Besides, the nuclear elastic scattering is always accompanied by the Coulomb coherent scattering and interferes with it. Such features are manifested when the elastic resonant scattering is used with analytical purpose. These are reasons to separate this type of the reactions. We considered the broad resonances in the elastic scattering. Some characteristic properties of the narrow resonances use caused by the same interference effects will be considered additionally. Obviously, the possibilities of the elastic resonant scattering for the material analysis are limited. But in special cases it may be successfully applied either independently or as an addition to the other analytical methods.

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ПРУЖНА ЯДЕРНА РЕЗОНАНСНА СПЕКТРОМЕТРІЯ ЗВОРОТНОГО РОЗСІЯННЯ (ШИРОКІ РЕЗОНАНСИ)

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Аналізуються можливості використання пружного, ядерного, резонансного, зворотного розсіяння іонів при дослідженні матеріалів. Розглядаються широкі резонанси, для яких у значній енергетичній області переріз розсіяння змінюється плавно. За такої умови зберігається порівняна простота отримання інформації, властива методу резерфордівського розсіяння і, разом з тим, підвищується чутливість до виявлення легких домішок. Ядерне резонансне розсіяння завжди супроводжується потенціальним і кулонівським розсіяннями. Перелічені складові є когерентними, а тому інтерферують. Така особливість даного типу ядерної реакції проявляється й при використанні резонансного розсіяння в роботах по аналізу матеріалів. Залежно від параметрів рівня складеного ядра інтерференція може бути як конструктивною (переріз зростає), так і деструктивною (переріз зменшується). Для підвищення чутливості в першому випадку використовують розсіяння на ядрах домішки, а в другому – на ядрах основи, для зменшення фону. Фізичні принципи підтверджуються взятими з практики прикладами дослідження домішок азоту в берилії та кисню в кремнії.

УПРУГАЯ ЯДЕРНАЯ РЕЗОНАНСНАЯ СПЕКТРОМЕТРИЯ ОБРАТНОГО РАССЕЯНИЯ (ШИРОКИЕ РЕЗОНАНСЫ)

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Анализируются возможности использования упругого, ядерного, резонансного, обратного рассеяния ионов при исследовании материалов. Рассматриваются широкие резонансы, для которых сечение рассеяния

изменяется плавно в большом энергетическом интервале. При таком условии сохраняется сравнительная простота получения информации, присущая методу Резерфордского рассеяния и, вместе с тем, повышается чувствительность к обнаружению легких примесей. Ядерное резонансное рассеяние всегда сопровождается потенциальным и кулоновским рассеяниями. Перечисленные составляющие когерентны, а поэтому интерферируют. Такая особенность ядерной реакции данного типа проявляется и при использовании резонансного рассеяния в работах по анализу материалов. В зависимости от параметров уровня составного ядра интерференция может быть или конструктивной (сечение возрастает), или деструктивной (сечение уменьшается). Для повышения чувствительности в первом случае используется резонансное рассеяние на ядрах примеси, а во втором - на ядрах основы, для уменьшения фона. Физические принципы подтверждаются взятыми с практики примерами исследования примесей азота в бериллии и кислорода в кремнии.

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