LOOK ACADEMIC PUBLISHERS
OPEN ACCESS

INTERNATIONAL JOURNAL OF ENVIRONMENTAL & SCIENCE EDUCATION
2016, VOL. 11, NO. 17, 10423-10434

Ion Beam Diagnostics Planar Epitaxial Structures Nanophotonics, Nanoelectronics and Microsystems Technology

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ABSTRACT

The background of the study is that the planar film heterostructures are nowadays the most common objects nanophotonic and nanoelectronic technologies. The authors noted that, among such objects found so-called quantum wells, which are formed by a complex series of nanoscale layers are deposited with differing elemental composition, and simple single-layer coatings. Test heterostructure may be polycrystalline or amorphous sequentially deposited layers, but can be provided epitaxial objects. In addition to the interest in the composition, structure and thickness of the layers of heterostructure itself, it is important and knowing the width and the quality of the interlayer boundaries, as well as the presence-absence of their mutual diffuse infiltration. The results of the article is that of the operating experience of experts are usually quite clearly represent technological equipment, what type of heterostructures and the distribution of elemental composition therein can be expected. However, the intended routing is always in need of an objective independent confirmation. For planar nanoheterostructures most effective method of non-destructive testing of the objective distribution of elements over the depth of the target and the presence-absence is epitaxially ion beam diagnostics.

KEYWORDS
Heterostructure layer, nano, control and distribution

ARTICLE HISTORY
Received 31 May 2016
Revised 23 July 2016
Accepted 9 September 2016

Introduction

The interaction of ion beams medium energy (E = 1-2 MeV) with the material is accompanied by a complex of physical phenomena (Schmidt & Wetzig, 2013). Interaction of charged particles with the electron subsystem of condensed targets leads to their relatively uniform excitation and inhibition of this subsystem. Direct confrontation with the nuclei of the atoms that constitute the target under study, in contrast, leads to a sharp change in the energy and direction of propagation of the incident ions and, in some cases, to their extinction, accompanied by the emergence of another type of particle. The whole complex of these phenomena can be used to control non-destructive quantitative elemental composition and structural characteristics of the solid-state planar heterostructures in thickness from a few nanometers to a few micrometers (Bird

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& Williams, 1989). Ion-starting excitation of the electronic subsystem is accompanied by the release of the optical, ultraviolet and X-rays. Join the optical component of the output spectrum allows us to study some features of the band structure of the material (Pop et al., 1989). The X-ray component of the spectrum provides information on the elemental composition of the material (Kolyada, Zaichenko & Dmitrienko, 1978). It must be recognized that the X-ray fluorescence analysis (XRF) for the excitation of the ion (as in terms of other types of excitation) is highly sensitive, but a qualitative element diagnosis (Johanson, Campbell & Malquist, 1995; Je, 2017; Lambrechts & Saurabh, 2017). This is due, on the one hand, with the need to account for matrix effects (Revenko, 1994), and on the other - it should be understood that the depth of the output characteristic X-rays is determined by its energy (or wavelength). Therefore, each recorded by an X-ray line will correspond to the thickness of the surface layer defined by the absorption coefficient of the line.

Methodology

Interaction of ionic fluxes with the nuclei of the atoms that constitute the target under study is more specific. It can be accurately approximated by a theoretically based model Coulomb interaction of point charges. On the basis of this approximation method is to create a base ion beam diagnostics materials: Rutherford backscattering spectroscopy of ions (RBS) (Petrov & Abroyan, 1977). The main feature of this method is its absoluteness, ie method does not require the presence of templates. As a normalization factor in the POP method uses the knowledge of the total number of ions scattered by a target in the measurement session.

Results and Discussion

Example POP diagnostics elementary film structure is shown in Figure 1a. The results of the approximation made visually represented on the right (Fig. 1b). The inset shows the geometry of the measurements using a beam of helium ions E0=0.970 MeV.

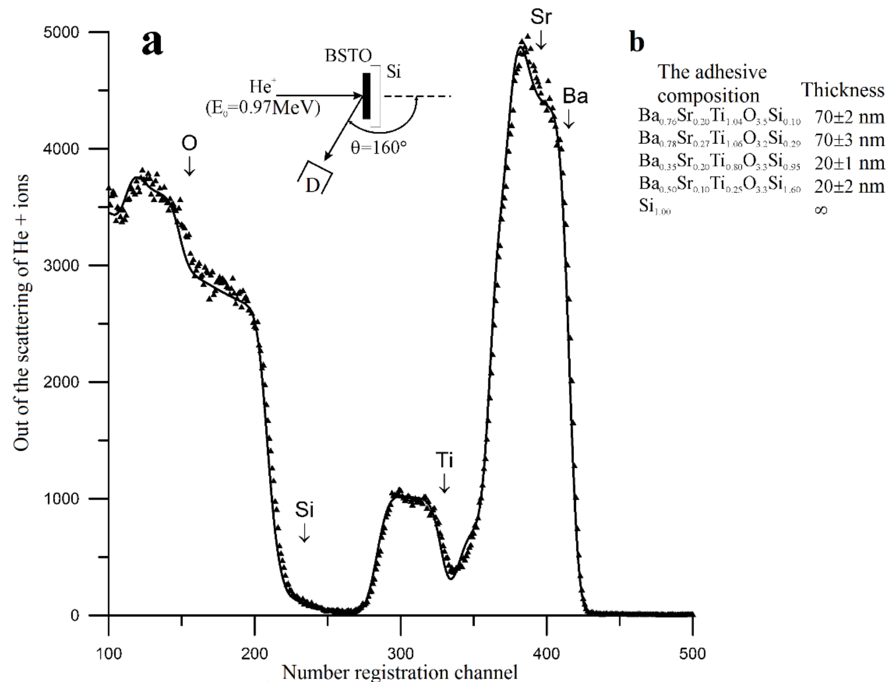

Figure 1. The theoretical and experimental spectra POP He^{+} (E_0=0.97 MeV) film barium strontium titanate on a silicon substrate. To the right a model of elemental concentration profile over the film thickness, the most adequate corresponding experiment. The arrows indicate the value of the energy of the scattered helium ions corresponding to atoms on the surface of the coating. Energy price channel 1.9 keV / channel. The inset shows the measurement geometry.

Presented spectrum consists of a broad line of intense, wide line with low intensity, the spectral level with a broad peak on the crest of this stage. Since the beginning it was known that the sample is a film of barium-strontium titanate oxide on a silicon substrate, a qualitative interpretation of the spectrum is not that difficult. Intensive spectrum line corresponds to scattering at the nuclei of heavy elements (Ba, Sr), low-intensity line reflects the presence in the film of titanium atoms, the step associated with scattering on nuclei of atoms of the substrate (Si), and the line is observed on the steps of the ridge, indicates the presence in the sample a significant amount of oxygen atoms. The arrows show the energy corresponding to scattering on the nuclei of atoms, which codes appear on the arrows under the condition of their location on the target surface.

Further, in the simplest model was chosen as a first approximation step Ba0.8Sr0.2Ti1.0O3/Si. The fit of the theoretical model to the experimentally obtained spectrum was carried out with the help of an interactive computer program RUMP (Doolittle, 1985). The calculations have shown that the simple model does not allow to approximate the range of POP obtained in the experiment. The increasing complexity of the model allowed us to obtain an approximation of satisfying the minimum discrepancy between the experimental and theoretical spectra (Egorov & Ohulkov, 1993; Lee, 2017). The theoretical model of the distribution of elements over the depth of the target investigated, corresponding to the best spectral fit is shown in Figure 1b.

Basically POP approximation may be subject to self-control. The fact that the registration of the experimental spectra ion backscattering can be carried out at different angles. As an example of self-control in Figure 2 shows the spectra of ions POP He+ (E0=1.5 MeV) to target Nb/Si. The spectra were recorded on an analytic complex Sokol-3 in parallel to the scattering angles θ1=167° and θ2=125°. Made fitting showed that the same model is perfectly approximates both the experimental spectrum. Of course, for a given target such approximation approach manifestly excessive, although its use is possible to obtain a more precise value for the thickness of the film of niobium t=135.2±0.7 nm, significantly better than the average depth resolution.

Rutherford measurements at normal incidence of the ion beam on the target surface have an average depth resolution of about 10 nm. This value is mainly determined by the energy resolution detectors recording the scattered ions. The resulting value of precision in determining the thickness in the case of niobium film is a formal value corresponding to the minimum residual considering two spectra POP. In fact, we must admit that the real accuracy of the slightly worse, at the level of 1.5-2 nm. At the same time, it should be noted that in the case of high surface studied target level of roughness 2.1 nm possible a significant improvement in the average of the experimental depth resolution of 2-3 nm by measurement at glancing angles of incidence of the ion beam on its surface (Komarov, Kumakhov & Tashlykov, 1987).

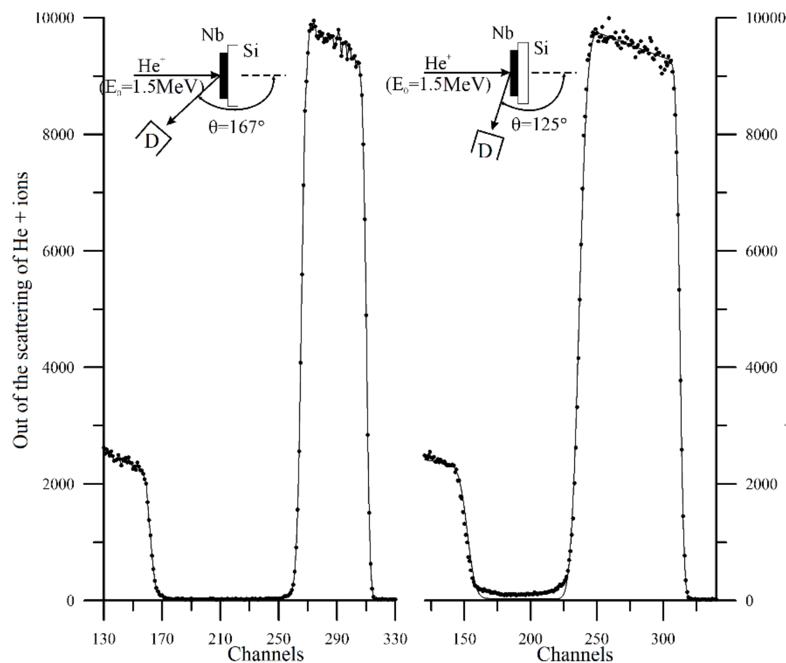

Figure 2. Experimental and theoretical spectra of ions POP $4He^{+}$ (E_0=1.5 MeV) for Nb / Si target, obtained at scattering angles of 167 and 125 degrees, the film thickness of the Nb 135.2 ±0.7 nm. Energy price channel 1.9 keV / channel.

Rutherford backscattering is sensitive to the density of the materials. Figure 3 shows the experimental spectra of ions POP H+ (E0=1.557 MeV) for the samples of the two modifications of carbon: diamond plates (ρ=3.51 g/cm3) and glassy (ρ=2.21 g/cm33). Changing the density is fixed by the value of the

scattering intensity output. At the same time, it should be noted that the nature of the scattering of hydrogen ions at various targets, particularly those containing light elements differs substantially from scattering helium ion energy range $E_0<2$ MeV. The fact that the helium ions in this energy range isotopes interact with the nuclei of all the elements in accordance with Coulomb's law. But the reaction of hydrogen ions is different from the Coulomb's law, and for different isotopes hold their individual differences. For example, Figure 4 shows the theoretically calculated and experimentally obtained scattering cross section of flow of hydrogen ions in the target nuclei, consisting of atoms of the isotope $^{12}C_6$ the energy of these ions (Rauhala, 1985). This factor must be borne in mind when approximation scattering spectra flow of hydrogen ions in the presence of the target atoms, which are characterized by no rezerfordo contribution to the scattering cross section. Having no Rutherford scattering effect, on the one hand, it creates additional problems, because it shows the energy dependence and, on the other - in some cases, allows to reduce significantly the detection limits of those elements for which this effect is characteristic. This fact is quite important, because the method is characterized by POP detection limits of impurities only at the level of 0.1% at.

Very useful, although quite laborious possibility ion beam analysis of materials seems the presence of the channeling effect of high-energy ion beams in the structure of perfect crystals (Feldman, Mayer & Picraux, 1982). Channeling ions can be carried out along the crystallographic planes and the crystal axes. For example, the axial channeling occurs when the propagation direction of the ion beam coincides with one of the crystallographic axes of the crystal. A small portion of the ion flow will experience scattering on nuclei of the crystal at the surface. The remainder of the fall in the interatomic low electron density and slow down in the depths of the crystal, hands without experiencing head-on collision with the nuclei of the target atoms. As a result, Rutherford backscattering spectra were recorded in conditions of channeling and lattice random orientation relative to the ion distribution will be radically different. The same difference is observed not only in the case study of single crystals, but also for testing really epitaxial structures. Figure 5 shows the RBS spectra of ions He^+ ($E_0=0.93$ MeV), obtained in conditions of channeling ions along the [100] and the random orientation of the epitaxial target $Ba_{0.8}Sr_{0.2}Ti_1O_3/MgO$. The spectrum shows the ion channeling symbatic decrease in the intensity of ions undergoing scattering as film on nuclei of atoms, so at the nuclei of atoms of magnesium oxide constituting the single-crystal substrate. In this case, the sinking of the spectrum POP proved significantly lower than expected for an ideal epitaxial structure. This result indicates a low structural perfection of the crystal substrate, and, as a consequence, lower structural perfection epitaxially grown perovskite films. Channeling ion is an effective method for studying the characteristics of the crystal structure of single crystals and epitaxial heterostructures, which allows not only to determine the position of the impurity atoms in the crystal lattice, but also to analyze the types of crystal defects (Gotz & Gartner, 1988).

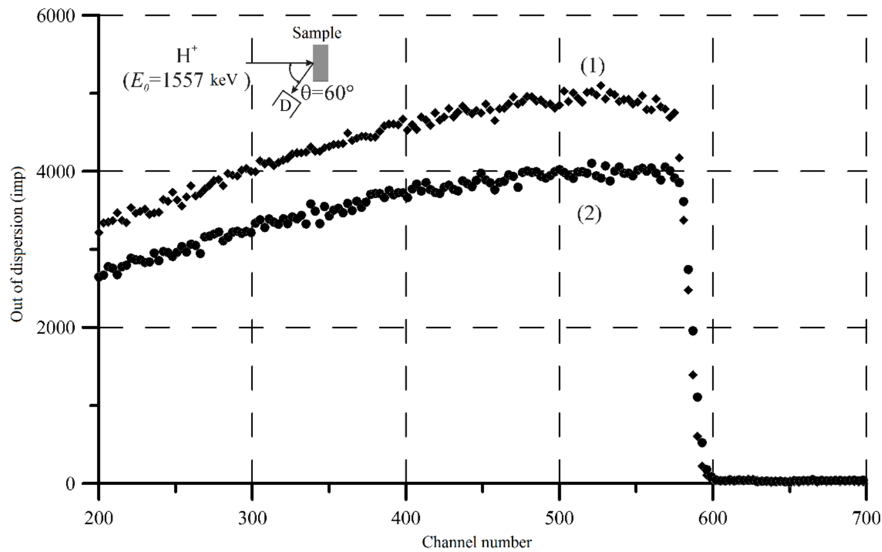

Figure 3. The experimental spectra POP H^{+} (E_0 = 1.55 MeV) registered for the diamond plate (1) and glassy carbon plate (2). Shows every third channel, the energy price channel 1.9 keV / channel.

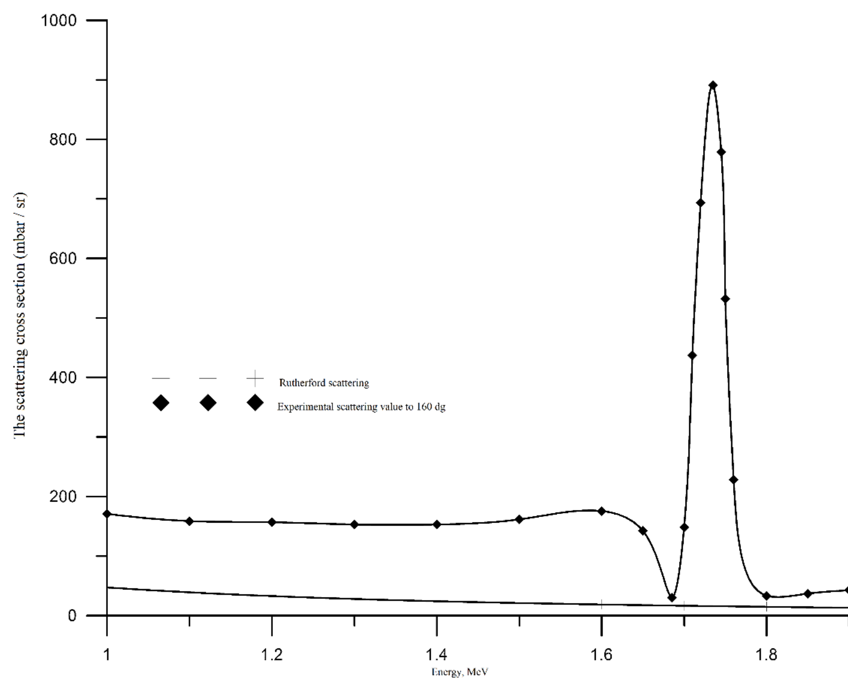

Figure 4. Experimental registered and calculated energy dependence of the scattering cross section of flow of hydrogen ions on a carbon target. Estimates correspond to scattering on a purely Coulomb potential (Rauhala, 1985).

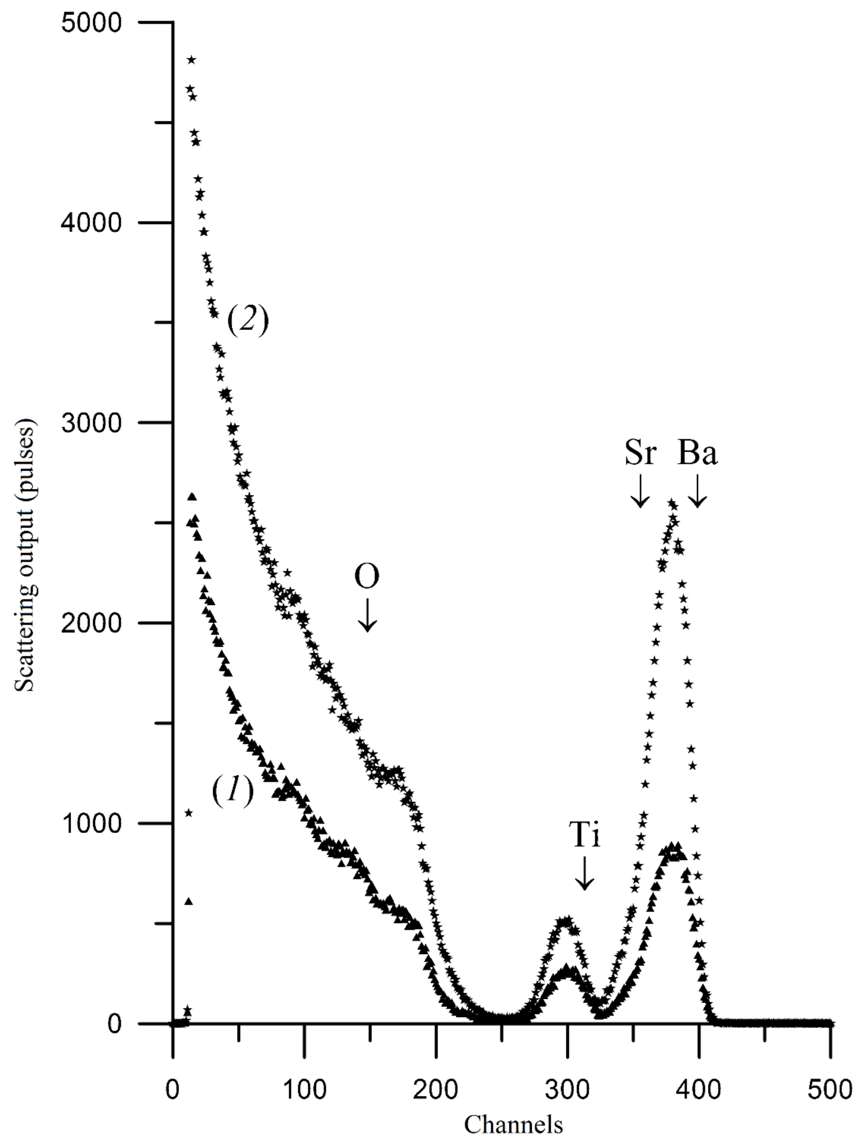

Figure 5. Spectra POP + ions (E_0 = 0.93 MeV) for the scattering angle 160°, obtained under the conditions of axial channeling ion flow along the crystallographic direction [100] (1) and a random target position (2) $Ba_{0.8}Sr_{0.2}Ti_{1.0}O_3$ / MgO. Arrows indicate the energy corresponding to the energy values of the scattering on the nuclei of atoms Ba, Sr, Ti and O, located on the target surface. Energy price channel 1.9 keV / channel.

The interaction of energetic ions with nuclei of the target atoms initial ion energy is redistributed between the scattered ion and atom, the nucleus of which was the diffuser. The lighter the scattering atom, the greater part of the energy it takes away from the incident ion. Experimental studies have shown that it is possible to analyze the energy spectrum of the scattered ions, and atoms to nuclei where they were scattered. The analysis of these spectra has led to a method of spectroscopy of recoil nuclei (Tirira, Serruys & Trocellier, 1996). recoil method is most effective for the non-destructive testing of the content of hydrogen atoms in the thickness of the target. The method is applicable for

diagnosis of superficial layers of the target 1-2 microns with a resolution of about 50 nm. The main problem of the method is to separate the hydrogen content of the surface on its concentration in the bulk of the target. Testing for the hydrogen content in various types of heterostructures is very important because even small concentrations can significantly affect the properties of semiconductor structures. Similar considerations baseless as in (Egorov, Egorov & Afanas'ev, 2012) it was found the presence of hydrogen at a concentration of about 4% at. in the single crystal structure SrTiO3.

Backscattering spectrometry and ion recoil method is based on the elastic interaction of streams of high-energy ion beams with the atomic nuclei of the target investigated. However, there are channels and inelastic interactions which are nuclear reaction. If ion manage to overcome the Coulomb barrier, he is captured by the nucleus of an atom, which leads to a change in the atomic number of the atom. This nucleus is in an excited state, and gets rid of excess energy emission of gamma rays or charged particles. Phenomena of this type are called nuclear reactions, and they are divided into two groups: the threshold and resonant. In our energy interval threshold reactions are characteristic only for the interaction of the nuclei with a flow of ions of deuterium and tritium, which is beyond the scope of this report. Resonant nuclear reactions typical for interaction of the beams of hydrogen ions (protons) with a number of nuclei of different isotopes. In the diagnosis of the distribution of isotopes or ion implantation and diffusion are analyzed distribution profiles of elements at a target depth of (Bird & Williams, 1989), and the depth resolution in this case will be determined by the energy of the nuclear resonance width. One of the most narrow resonances correspond to the nuclear reaction Al27(p;γ)Si28. Resonance occurs when the hydrogen ion beam energy E0=992 keV at width of 50 eV. The nuclear reaction is accompanied by the release of gamma rays with energies of 10.78 MeV, 7.93 MeV and 1.77 MeV. By registering the output of the gamma radiation lines depending on the initial energy of the proton beam, can be fixed distribution atoms Al, which is represented by a single isotope Nature Al27, the thickness of the target. An example of such measurements is shown in Figure 6 (Amsel, Nadai & D'artemare, 1971). It is a function of the output gamma line $E\gamma$=1.77 MeV for a given resonance of the hydrogen ion beam energy for an aluminum target with a surface oxide film thickness of 48 nm. Depth resolution of the target surface is 5 nm. However, this permission is deteriorating with the deepening of the ion beam at the target, as this increases the energy spread in the beam. If the target interface is characterized by a sharp rise in gamma-ray output, the internal interface is characterized by a significant broadening, although POP measurement data do not record such broadening. It should also be noted that the method of nuclear reactions is not absolute and is based on the normalization method of Rutherford backscattering, although it is characterized in comparison with POP lower detection limits. However, the most sensitive of all the ion beam methods for diagnosing cell material has X-ray fluorescence analysis with ion excitation (PIXE).

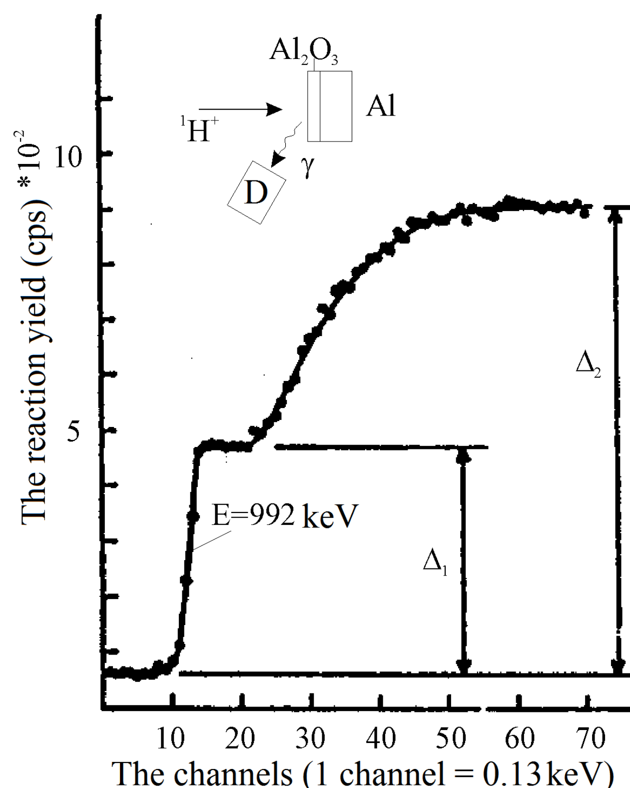

Figure 6. Experimental dependence of the recorded gamma rays with energies of 1.77 MeV resonance nuclear reaction ^{27}Al (p; γ) ^{28}Si resonance with the energy E_0 = 992 keV proton beam energy to the target Al_2O_3 / Al (Amsel, Nadai & D'artemare, 1971). The thickness of the oxide film was 48 nm. Energy price channel 0.13 keV

As noted above, the excitation of the characteristic X-ray fluorescence is due to the interaction of fast charged particles with the electronic subsystem investigated target, and require the ionization of atoms constituting the target of such fluorescence. Ionization is the result of drastic changes in the magnitude of the electromagnetic field in the vicinity of nuclear leave. Experiments with x-ray fluorescence excitation of electrons flows showed that the effective ionization of K and L of the electron shells of atoms sufficient electron beam energy of 10-20 keV (Reed, 1970). In order to achieve the same degree of ionization of the proton beam energy is necessary to compare the maximum transmitted atom for its electron ionization and ion that this ratio is in the range of two orders of magnitude (Komarov, Kumakhov & Tashlykov, 1987). Experiments confirm the estimates made, and it turns out that the effectiveness of low-energy excitations X-ray fluorescence much higher in comparison with the high-energy (Cahill, 1980). It is clear that the production of streams of electrons with an energy of 10-20 keV is much easier and less expensive in comparison with plants that generate beams of protons with energies of 1-2 MeV. However, the ion beam x-ray fluorescence excitation has a very important advantage in comparison with the electron excitation. The fact that the electronic excitation is characterized by the emergence of a significant part of the background, appearing due to the bremsstrahlung of electrons (Feldman & Mayer, 1989). Low background component and high excitation cross section of the low-energy x-ray fluorescence

to successfully diagnose multi-element targets, including those containing light elements. Figure 7 shows the X-ray fluorescence spectrum of a multiple target structure Na3Zr1.3Si1.9Al0.1P1.0O12C2, the resulting excitation of the target with a beam of protons with energies E0=1.18 MeV. In contrast to the standard measurement conditions, which recorded the integral output of X-ray fluorescence spectrum shown in Figure 7, the output reflects the characteristic X-ray radiation from the surface of the target layer thickness of 3 nm (Egorov, Egorov & Afanas'ev, 2015). In this case, the modification of the RFA at ion excitation was achieved through the use of a resonant waveguide structure to form the output of the excited ion beam x-ray fluorescence, limited the critical angle of total external reflection of the target material. Such an approach can be extremely useful for highly sensitive analysis of the elemental ultrafine surface layers of planar heterostructures containing light elements.

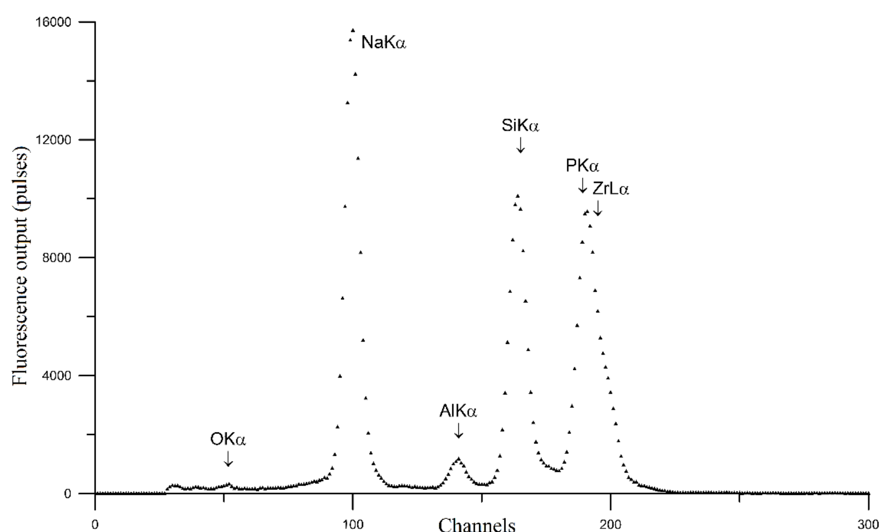

Figure 7. The range of x-ray fluorescence yield for $Na_3Zr_{1.3}Si_{1.9}P_{1.0}O_{12}C_2$ target, registered under the excitation beam of hydrogen ions (E_0 = 1.18 MeV) for its surface layer thickness of 3 nm. Formation of the fluorescence yield, corresponding to the surface layer, was carried out with the help of the waveguide resonator with a slit width of 0.15 m. Energy price channel 10.2 eV (Egorov, Egorov & Afanas'ev, 2015; Lee, Tseng & Carlo, 2017; Yang, Na & Heo, 2017).

Conclusion and Implications

Overall ion brame diagnosis of elemental composition and properties of various epitaxial heterostructures, focused on problem solving planar nanophotonics and nanoelectronics, it is a useful tool to quantitatively and non-destructively identify their features that are not available for testing by other methods.

Acknowledgement

The work was supported by the Ministry of Education of Russia, identifier ПНИЭР RFMEFI57715X0177.

Disclosure statement

No potential conflict of interest was reported by the authors.

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