NEW ION ELASTIC SCATTERING BEAM LINE OF ANALYTICAL COMPLEX AT IAP NAS UKRAINE

A.A. Drozdenko, S.M. Duvanov, S.N. Mordyk, V.E. Storizhko
Institute of Applied Physics of the National Academy of Sciences of Ukraine, Sumy, Ukraine
E-mail: ipfmail@ipfcentr.sumy.ua

The paper presents a description of new Ion Backscattering Beam Line. Small-size electrostatic accelerator "Sokol" with a beam energy range of 0.2…2.0 MeV is used as an ion source. A detail of the Beam Line is using of magnetic spectrometer which allows to carry out precise (several nm) quantitative layer-by-layer non-destructive element analysis of solid composition and structure using Ion Elastic Scattering technique. Design of Backscattering Chamber allows realizing a full set of Ion Beam Analysis techniques. Proton and helium ion backscattering spectra are presented from the thin film samples. Simulated spectra fit well the experimental ones together. The last shows the analytical efficiency of Backscattering Beam Line.

PACS: 29.17.+w, 29.30.Ep, 29.30.Aj

1. INTRODUCTION

Elastic Ion Scattering with the energy of several MeV is widely used as an analytical technique to study the element composition and surface and near-surface layers structure of materials in the depth range up to 10 μm [1,2]. The most-used technique is Rutherford Backscattering Spectroscopy (RBS). A depth resolution of this technique depends on the accelerated ion beam mono-chromaticity (~100 eV for the electrostatic accelerators), ion energy loss straggling during transport trough the matter, detector energy resolution, ion mass and geometry of the experiment. Relative poor energy resolution of modern semiconductor detectors (~10… 12 keV) limits essentially the depth resolution in depth profiling experiments. At the same time, the requirements to improve depth resolution in studies of the low-dimensional solid-state structures in the different fields of technology and science are permanently increased. The principle solution of this problem is application of magnetic and electrostatic spectrometers in ion backscattering to detect the spectra.

This paper presents a description of new Beam Line of Analytical Accelerator Complex built at the Institute of Applied Physics of National Academy of Sciences of Ukraine (IAP NAS Ukraine) for the researches with using of Ion Elastic Scattering technique. Unlike existing ones, this Beam Line combines possibilities of the traditional technique with utilization of semiconductor detectors and new possibility for the precise measurwith ements using magnetic spectrometer.

2. BRIEF REVIEW OF ANALITICAL DEVICES WITH MAGNETIC SPECTROMETERS

Below a brief review of recent works on existing facilities with magnetic or electrostatic spectrometers is presented. Paper [3] describes new microbeam equipments which contains 15 MeV tandem ion accelerator and 90° magnetic spectrometer of vertical type. The magnet has a relative energetic dispersion of $\Delta E/(E\times x)=2.1\times10^{-4}$ mm^{-1}. A standard linear CCD-chip was used as the position-sensitive ion detector. A solid angle of the spectrometer is less than 0.5 msr. A resolution of 3.8×10^{-5} (or 3.4 keV) was obtained in transmittance experiment using 700 nm focused beam of 90 MeV $^{32}S^{8+}$ ions.

A magnetic spectrometer with the ultrahigh resolution for RBS- and ERD- analysis is described in paper [4]. It uses MeV ions (up to 6.5 MeV) from tandem accelerator of Utrecht University, The Netherlands. Magnetic spectrometer Beam Line consists of two quadrupole lenses, a Wien filter and a magnetic dipole. With this combination of elements one unique charge-to-mass ratio of ions with energies within a chosen range can be selected for detection with two-dimensional position-sensitive detector placed behind the dipole. 13% of the energy window range is imaged with a resolution of $\Delta E/E=3\times10^{-4}$. At the same time, an angular range of 5° is imaged with a resolution of 0.15°. A solid angle of the spectrometer is approximately 0.5 msr. Spectrometer has ultra high vacuum scattering chamber with three-axis goniometer and "on-line" sample preparation chamber. This paper describes a study of the epitaxial grown δ-layers of Ge in c-Si using 1.3 MeV $^{4}He^{+}$ ions. An angle between the beam and the sample was 64.0°, a scattering angle was 70.0°. In this case, the obtained depth resolution in the near surface layers was 0.3 nm.

Paper [5] describes an electrostatic spectrometer for high resolution measurements using MeV ion registration (with the energy up to 6 MeV). This spectrometer consists of a system of the electrostatic lenses, electrostatic analyser and linear position-sensitive detector. The analyser is 100° cylindrical condenser with 700 mm radius and 19.8 mm aperture width. 2 MeV double-charged ions were analysed with ±60 KV voltage on analyser plates. Energetic resolution of analyser is better than 3×10^{-4}. Using microchannel plate instead of silicon surface-barrier detector linear detector was allowed to obtain depth resolution of 0.1 nm in gold target sample with 1.5 MeV Ne analysing ion beam.

This review shows that the using of magnetic and electrostatic spectrometers essentially improves analytical possibility of Ion Backscattering technique.

3. ELECTROSTATIC ACCELERATOR AND ION-OPTICAL SYSTEM OF ION ELASTIC SCATTERING BEAM LINE

A small-size electrostatic 2 MeV accelerator "Sokol" is used as a source of hydrogen and helium ions [2]. The accelerator design was significantly modified to obtain high-quality ion beams. In particular, the ion source with a system of the permanent magnets designed for the increasing of plasma density as well as beam prefocusing [6]. Instead of analysing magnet which is also used as distributing one in "Sokol" accelerator, a 90° magnetic analyser MA-90-500 with 500 mm radius and distributing magnet MS-45-7 for seven Beam Lines was developed at IAP NAS of Ukraine and manufactured at Efremov Scientific-Research Electrophysical Institute (St. Petersburg, Russia). Different focusing regimes are provided by a quadruplet of the electrostatic quadrupole lenses placed between these analysers. The quadruplet was also manufactured at Efremov Scientific-Research Electrophysical Institute. Stabilised power supplies for these analysers and lenses were created at IAP NAS of Ukraine.

4. ANALYTICAL ELASTIC BACKSCATTERING BEAM LINE

The described Beam Line is intended for the study of element composition and structure of surface layers of solids, crystalline materials and other materials of different origin. The Beam Line is used when solving problems of microelectronics, materials science, biology, medicine, etc. with utilisation of light ion Rutherford Backscattering (RBS) technique.

Backscattering Beam Line (BBL) consists of two main parts: magnetic spectrometer with accessories and beam line with Backscattering Chamber. Backscattering Chamber consists of backscattering measurement equipment to perform the measurements in standard, channelling and transmittance modes.

Functional scheme of BBL is shown on Fig.1. Fig.1,a demonstrates schematic top view of the accelerator (1), beam line in front of (2) and behind (3) 90° analysing magnet (4) in beam direction, quadruplet of the electrostatic quadrupole lenses (5) and also side view of BBL (Fig.1,b). Beam Line is mounted behind distributing magnet (6) of Analytical Accelerator Complex. Its optical axis in horizontal plane forms 30° angle with main optical axis of the Complex. BBL including beam line (7) and Backscattering Chamber (8) is placed behind distributing magnet in beam direction. Vacuum Beam Line equipment contains: silphon units (9,10); vacuum gate valves used for vacuum cutoff of vacuum tubes of BBL from vacuum chamber of distributing magnet (11) and of Backscattering Chamber from its tube (12) and tube of magnetic spectrometer (13); two aperture diaphragms (14); beam monitoring unit (15) equipped with Faraday cup and luminescence screen with branch pipe for optical TV monitor.

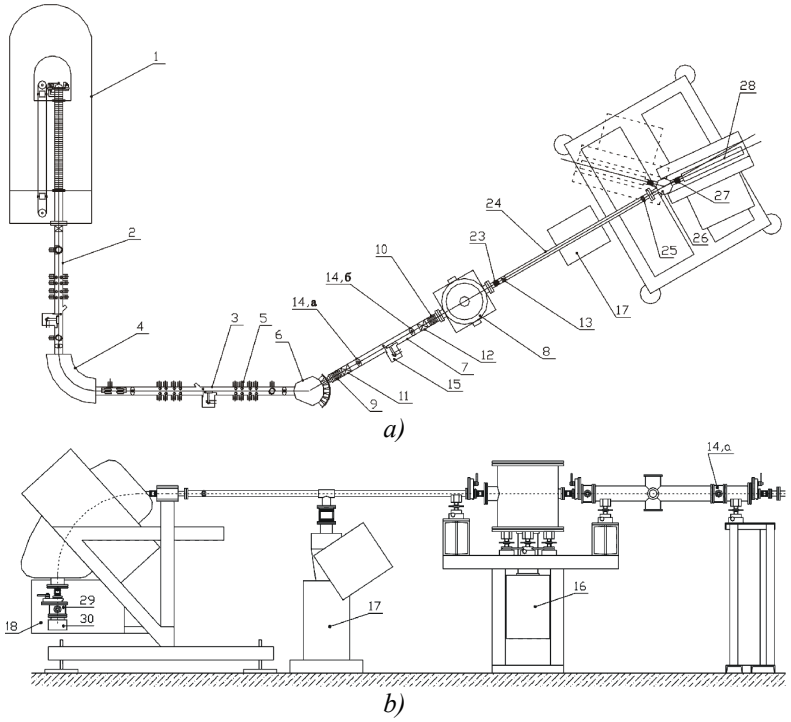


Fig.1. A functional scheme of the Backscattering Beam Line with accelerator and magnets (a, top view) and schematic draw of the line behind the distributing magnet (b, side view). Dashed line shows ion beam trajectory. Accelerator is MeV ion source. On Fig.1,b, ion direction is right-to-left

BBL vacuum tube (7) and Backscattering Chamber (8) are placed on supports which allows to set beam line optical and chamber axis on 1350 mm height from the floor. Supports provide chamber vertical movement in range of ±50 mm, and ±100 mm in horizontal plane.

Pumping system consists of three vacuum posts (16, 17, and 18) that work both separately and together and provide vacuum conditions better than 10^{-4} Pa in chambers and beam lines.

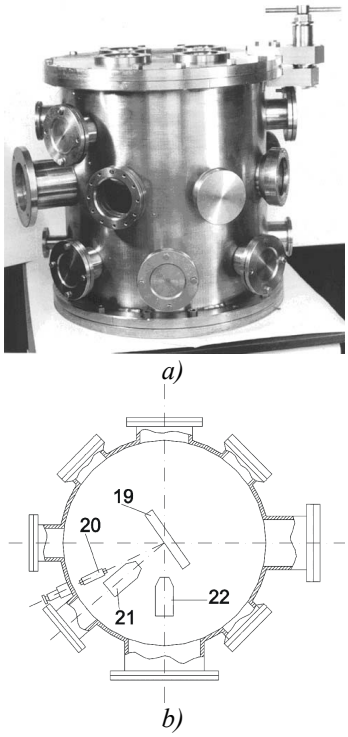
Photography of BBL with magnetic spectrometer is shown on Fig.2.

Fig.2. Backscattering Beam Line photography

5. BACKSCATTERING CHAMBER

Backscattering Chamber (fig.3) is a first of two functional elements of BBL and is placed behind the distributing magnet. RBS is a base technique of the analysis realised in the chamber. ERD, PIXE and NRA techniques are complementary to RBS one. Design of the chamber allows to perform scattering experiments in standard geometry (angle between beam and target sample is in range of φ=60…90º, and backscattering angle θ is close to 180º and is usually equal to 170º), in glancing angle geometry (φ≈15º, θ≈30º) as well as in channeling and transmittance regimes. Backscattering chamber is described in paper [7] in more details.

Photography and scheme of the chamber (top view) are shown on Fig.3. Chamber (8) (see Fig.1,a) of Backscattering Beam Line consists of: a frame of the chamber with branch pipes (Fig.3,b), lift off cover, cover lift off device, and sample holder (9).



a)

b)

Fig.3. Backscattering Chamber photo (a) and its scheme (b, top view) with beam–detectors plane cross section,, sample holder, and secondary radiation detectors. On Fig.1, this chamber has label 8. Description of the labels is presented in the text

Charged particles are registered with semiconductor detector (SCD) (20) placed in the chamber branch pipe at 170° angle to ion beam direction. X-ray detector (21) and gamma-ray detector (22) are used for registration of secondary radiation induced by primary analysing charged particles beam. A pipe for X-ray detector is 80 mm in diameter and is located at 135° angle to ion beam direction. Gamma-ray detector pipe is 158 mm in diameter and is placed at 90° to ion beam direction. Gamma-ray detection is also possible by using scintillation detector with outer diameter of 160 mm and based on NaI(Tl) crystal (150 mm in diameter and 100 mm in height), which is placed outside the vacuum chamber.

Sample holder is electrically isolated from other installation parts: backscattering chamber, vacuum tubes, Faraday cup, vacuum pumps, and support. Ion beam current is measured with current integrator from sample holder via vacuum connector.

Chamber vacuum pumping system has two stages. First, vacuum in the chamber is created using 7.5 l/sec baking pump LEYBOLD-HERAEUS D16A having an oil trap. 450 l/sec turbo-molecular pump LEYBOLD-HERAEUS TURBOVAC 450 creates working vacuum conditions in the chamber. It is installed over the branch pipe placed on the bottom cover of the chamber. Two-stage pumping system provides a vacuum better than 10^{-4} Pa during 30 min in the scattering chamber and beam line volume.

6. 90° MAGNETIC SPECTROMETER WITH UNIFORM FIELD AND DOUBLE FOCUSING

Magnetic spectrometer with equipment is the second functional part of Backscattering Beam Line (see Fig.1). Spectrometer is placed behind the Backscattering Chamber. Parts of magnetic spectrometer are connected according to the following scheme: sylphon unit (23), vacuum gate valve (13), beam line of magnetic spectrometer with joint unit for pump system (24), pump system (17), diaphragm (25), magnetic spectrometer scattering chamber (26), sylphon unit (27), magnetic spectrometer vacuum chamber (28), magnetic spectrometer vacuum chamber pump (19), output aperture slit unit (29), charged particle detector unit (30). Based technique implemented in chamber is RBS with glancing angle, transmittance, and standard geometry. Magnetic spectrometer as a charged particles detector and glancing angle geometry allow to achieve a high depth resolution (several nm) [1, 3, 4]. This resolution is five times grater than that in standard backscattering spectrometry with semiconductor surface-barrier detectors of charged particles. Analytical possibilities of BBL could be essentially extended when magnetic spectrometer is used in coincidence mode with the other detectors of secondary emission products induced by primary analysing beam.

Magnetic spectrometer with uniform field and double focusing is described in [10] in more details. This spectrometer has the following parameters: a radius of the particles trajectory in the magnetic field –

320 mm; rotation angle – 90°; input cut angle – 46°, output cut angle – 4°51′; gap height – 16 mm, gap width – 106 mm; distances from source (target) to magnet entrance and from detector to magnet exit are 400 and 700 mm, respectively; magnet weight – 1500 kg.

Magnet is powered from stabilised power supply. Magnet current is stabilised with 0.05% accuracy in 4… 40 A range which is corresponding to field alteration in range of $2\cdot10^3$…$15\cdot10^3$ Oersted (0.2…1.5 Tesla). Respect detected protons energy is in the range of 0.2… 8 MeV. Magnet is remotely controlled.

Spectrometer vacuum chamber is connected to Scattering Chamber trough silphon unit. Detector is place on the end of the vacuum chamber of the magnet at 686 mm distance from output cut. 1 mm aperture is installed in front of the detector unit. Standard semiconductor surface-barrier detector is operated in the counting regime.

Magnet with scattering chamber is mounted on the platform which allows to rotate the magnet at 0…150° to the left and 0…20° to the right relatively to incident beam.

Pumping systems (18,16,17) creates a vacuum in magnetic spectrometer scattering chamber (26) and in magnet vacuum chamber (28).

Experimentally measured resolution of the spectrometer is (0.15±0.03)% using α-active calibrating source with 1×8 mm^2 and 1 mm detector aperture and is in a good agreement with the calculated value. Measured solid angle value is found out to be $(3.56\pm2.23)\times10^{-3}$ sr and is also close to the calculated value.

7. EXPERIMENT

Fig.4 shows one of the first backscattering spectra measured using RBS Beam Line of IAP NAN of Ukraine equipment.

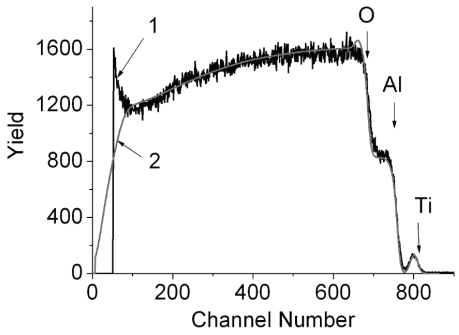


Fig.4. Proton backscattering spectrum with initial energy of 1.05 MeV measured from polycrystalline alpha-alumina sample implanted with Ti ions at 10^{17} cm^{-2} fluence (1 – experimental spectra, 2 – simulated one

Testing sample is polycrystalline α-Al_2O_3 implanted with Ti ions using a vacuum-arc source in the pulsed mode. This sample has good conductivity of modified surface layer and precisely definite matrix composition. The sample was also analysed with proton and helium ion beams in two different Accelerator Laboratories located in Germany and Russia.

Initial energy of analysing proton beam is 1.05 MeV, angle between beam and sample – 90°, backscattering angle – 170°, beam current – 10 nA.

Standard spectrometry equipment with semiconductor surface-barrier detector manufactured by "SILENA" was used for the spectrum registration. Resulting data were processed using SIMNRA [11] and DVBS [12] codes. Depth profile of Ti-implant was extracted from the spectrum.

Fig.4 shows good agreement between experimental spectrum and simulated one. A broadening of the high energy edges of partial spectra for Al and O is well simulated when spectrometer energy resolution is equal to about 22 keV.

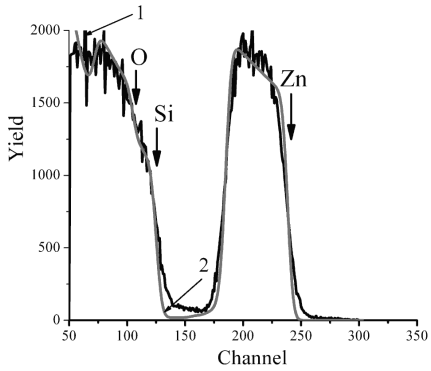


Fig.5. 0.8 MeV $^4He^+$ RBS spectrum from ZnO/Si sample: 1 –experiment; 2 – simulation

Using of backscattered helium ions instead of protons allows measure the element depth profiles in the samples more sensitively and precisely. It is clear from the RBS spectra (see Fig.5) of helium ions measured from thin films of ZnO deposited on silicon substrate applied reactive magnetron sputtering machine. Depth profiles of Zn and O in the film was extracted from the spectra. Film thickness of about 220 nm was estimated. Zn concentration in thin film was found out about 20 at. % less then that in stoichiometric composition. At the same time, oxygen concentration corresponds to ZnO stoichiometry. It is assumed that thin film contains another light element additionally to oxygen. This technique has limited sensitivity to detection of low-Z impurities in heavy matrix so it is necessary to apply an additional resonance proton scattering or nuclear reactions analysis to clear up the situation with the additional element in composition of thin film. In spite of some limitations, RBS of light ions is very informative and important when it used as an analytic one in thin film deposition technology. It is also very suitable in the particular case of thin ceramic film synthesis technologies developed at IAP NAS of Ukraine.

CONCLUSION

New Backscattering Beam Line as a part of Analytical Accelerator Complex was developed at the Institute of Applied Physics of National Academy of Sciences of Ukraine. Beam Line allows to perform applied and fundamental experiments in the different fields of science and technology (microelectronics, geology, materials science, biology, medicine etc.) It is possible to realise full set of complementary techniques for non-destructive nuclear-physical (or ion-beam) elemental and structural analysis of material namely:

Rutherford backscattering Spectroscopy (RBS), Resonance and Non-Rutherford Ion Backscattering Spectrometry (BS), Elastic Recoil Detection Analysis (ERDA) of protons, Particle Induced X-ray Emission registration (PIXE), Nuclear Reactions Analysis (NRA), Ion Induced Luminescence Spectroscopy (IL) and many other ones in the Backscattering Chamber. The design of the Beam Line provides a possibility to perform the experiments in standard, channelling, and transmittance regimes. Backscattering Chamber and Magnetic Spectrometer combination allows to make precise measurements (several nm) of elements concentration depth distributions in samples as well as development new additional analytical techniques of non-destructive layer-by-layer elemental and structural microanalysis of surface layers of solids. An agreement between the experimental and simulated spectra demonstrates an analytical efficiency of new Backscattering Beam Line.

REFERENCES

1. V.E. Storizhko. Nuclear microanalysis techniques // *Proc. of the II All-Union Conference «Microanalysis on Ion Beams», October 11-13, 1988, Khar'kov.* Sumy: «Redaktsionno-izdatel'skiy otdel oblupravleniya po pechati». 1991, p.3-53 (in Russian).
2. A.D. Vergunov, Yu.Z. Levchenko, M.T. Novikov, V.M. Pistrjak, V.E. Storizhko, S.A. Chekanov // *Voprosy atomnoy nauki i tekhniki. Serija «Obcaja I jadernaya fizika» 24*. 1983, № 3, p.13-15 (in Russian).
3. G. Datzmann, G.Dollinger, Ch.Goeden, A.Hauptner, H.-J.Körner, P.Reichart, O.Schmelmer // *Nucl. Instr. and Meth.* 2001, v.B181, p.20-26.
4. D.O. Boerma, W.M. Arnoldbik, W. Wolfswinkel. Use of an Ultra-High Resolution Magnetic Spectrograph for Material Research // *Mater. Sci. Forum 248-249*. 1997, p.451-458.
5. H.D.Carstanjen. Ion Beam Analysis with Monolayer Depth Resolution // *Mater. Sci. Forum 248-249*. 1997, p.459-466.
6. V.I. Vozny, V.I. Miroshnichenko, S.N. Mordik, V.E. Storizhko, B. Suklio-Cleff. A possibility of the improvement of plasma density of RF ion sources // *Zbirnyk naukovykh prats' Instytutu jadernykh doslidzhen' 9*. 2003, №1, p.75-81 (in Ukrainian).
7. A.N. Dedik, S.M. Duvanovв, O.I. Ekhichev // *Proc. of the III All-Union Conference «Microanalysis on Ion Beams», September 17-19, 1990, Sumy*. 1991, c.135-143 (in Russian).
8. J.S. Williams // *Nucl. Instr. Meth.* 1978, v.149, p.2017.
9. C.R. Gossett // *Nucl. Instr. Meth. in Phys. Res.* 1986, v.B15, p.481-485.
10. A.S. Dejneko, A.I. Popov, P.V. Sorokin, A.Ya. Taranov // *Izvestya Akademii nauk SSSR. Serija fizicheskaja.* 1960, v.XXIV, №7, p.924-928 (in Russian).
11. M. Mayer. *SIMNRA, a Simulation Program for the Analysis of NRA, RBS and ERDA.* Proceedings of the 15th International Conference on the Application of Accelerators in Research and Industry, J.L. Duggan and I.L. Morgan (eds.). American Institute of Physics. Conference Proceedings 475, 1999, p.541.
12. V. Bogac and D.M. Shirokov // *Nucl. Instr. and Meth.* 1994, v.B84, p.497.

НОВЫЙ КАНАЛ УПРУГОГО РАССЕЯНИЯ ИОНОВ АНАЛИТИЧЕСКОГО КОМПЛЕКСА ИНСТИТУТА ПРИКЛАДНОЙ ФИЗИКИ НАН УКРАИНЫ

А.А. Дрозденко, С.М. Дуванов, С.Н. Мордик, В.Е. Сторижко

Описан новый канал обратного рассеяния ионов. Источником ионов служит малогабаритный электростатический ускоритель «Сокол» с энергией пучка диапазоне 0,2…2,0 МэВ. Особенностью канала является использование магнитного спектрометра, позволяющего проводить количественный прецизионный (несколько нм) послойный неразрушающий элементный анализ состава и структуры твёрдых тел методом упругого рассеяния ионов. Канал состоит из: камеры обратного рассеяния общего назначения и камеры магнитного спектрометра с ионопроводом. Конструкция камер обратного рассеяния позволяет реализовать полный набор ионно-пучковых методов элементного анализа. Представлены спектры обратного рассеяния протонов и ионов гелия от тонкопленочных образцов. Хорошее согласие экспериментального и расчетного спектров от имплантированного образца корунда свидетельствует о работоспособности канала обратного рассеяния как аналитического инструмента.

НОВИЙ КАНАЛ ПРУЖНОГО РОЗСІЮВАННЯ ІОНІВ АНАЛІТИЧНОГО КОМПЛЕКСУ ІНСТИТУТУ ПРИКЛАДНОЇ ФІЗИКИ НАН УКРАЇНИ

О.О. Дрозденко, С.М. Дуванов, С.М. Мордик, В.Ю. Сторіжко

Описано новий канал зворотного розсіювання іонів. Джерелом іонів служить малогабаритний електростатичний прискорювач «Сокіл» з енергією пучка 0,2…2,0 МеВ. Особливістю каналу є використання магнітного спектрометра, який дозволяє проводити кількісний прецизійний (декілька нм) пошаровий неруйнівний елементний аналіз складу та структури твердих тіл методом пружного розсіювання іонів. Конструкція камер зворотного розсіювання дозволяє реалізовувати повний набір іонно-пучкових методів елементного аналізу. Представлені спектри зворотного розсіювання протонів та іонів гелію від тонкоплівкових зразків. Добре узгодження експериментального та розрахункового спектрів від імплантованого зразка корунду свідчить про дієздатність каналу зворотного розсіювання як аналітичного інструмента.