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STUDY OF MOS STRUCTURES USING NUCLEAR ANALYTICAL METHODS

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Abstract. *The atomic concentrations and depth distribution of elements in MOS (metal oxide semiconductor) structures have been investigated using two nuclear analytical methods: Rutherford Backscattering Spectrometry (RBS) and Elastic Recoil Detection (ERD). The elements with atomic masses in range from hydrogen up to copper were identified. Their depth profiles show that a MOS structure consists of metal (Al) layer, silicon oxide layer and a silicon substrate. The heavy elements Cu, Ti were found at near-surface area of one sample with low concentrations. The transitional area between the silicon substrate and the oxide layer as well as between the metal and oxide layers was noticed. The obtained results provide valuable information about MOS structures and concurrently demonstrate the possibilities of both RBS and ERD methods in material analysis.*

Keywords: Rutherford backscattering spectrometry, elastic recoil detection, multilayer structures.

Classification numbers: 82.80.Yc; 85.30.-z; 01.30.Rr..

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I. INTRODUCTION

The RBS and ERD are the nuclear analytical methods that can be used to determine the elemental composition, atomic concentration, depth distribution of elements contained in near surface area of investigated samples. With the development of accelerator technology, the convenience of detectors with good energy resolution as well as the improvement of computer code for data processing, RBS and ERD methods have been widely applied in various studies of samples that are made from different materials. A lot of papers presented such studies in the recent years. For instance, the combination of RBS and ERD methods provided depth distribution of H, C, O, Si in the study of Cz-Si samples implanted by H_2^+ and annealed under high pressure [1]. The results pointed out variation of concentration and diffusion of H atoms when the temperature, time and pressure of annealing were changed. In an investigation of initial oxidation of AlPdMn quasicrystals [2], RBS method with the 2.5-MeV N ion beam was used in order to measure depth profiles of the metallic components, while ERD method was used for measuring oxygen depth profiles by the beam of 1.3-MeV Ar ions. The results with a high depth resolution proved that a quasicrystal is more corrosion resistant than ordinary materials. For further information about using RBS and ERD methods one can find in the next papers [3–6].

In 2011 at Hanoi University of Science, Vietnam the first tandem accelerator that named 5SDH-2 was installed. The first measurements were carried out for demonstrating possibilities of the nuclear methods on this system, as presented in the following papers [7–9]. However, the studies of applications of these ion beam analysis methods are encountered to many restrictions. Thus, the improvement of these methods using the accelerator has been performed by the international cooperation. Especially, in this study the experiments were carried out on the similar accelerator that named EG-5 at Frank Laboratory of Neutron Physics (FLNP), Joint Institute for Nuclear Research (JINR), Dubna, Russia. The combination of two nuclear methods RBS and ERD were applied in order to determine atomic concentrations and depth distribution for elements in the MOS-structure samples. The MOS structures play an important role in forming semiconductor devices [10]. The investigation of these specimens using the nuclear analytical methods showed a new efficient technique for material analysis could be carried out on the 5SDH-2 accelerator in the future.

II. THE NUCLEAR ANALYTICAL METHODS

II.1. Rutherford Backscattering Spectrometry

There are three main questions about the material characterization that we desire to answer. The first is "*What elements are there in the sample?*", the second is "*What are their atomic concentrations?*" and the third is "*What are the depths of these elements*?". Choosing an analytical method among many different manners depends on what was questioned. However, there is a method, which is able to respond to all these questions, it is named RBS method. Using the RBS method allows to investigate the depth distribution of different elements in near surface layers of solid without their destruction. The wide possibilities of this nuclear method are originated from the basic principles of the elastic collision process between energetic ions and the atomic nuclei of the elements in investigated samples. The fundamental concepts of RBS method were described in detail [11] and a brief introduction will be presented below.

The scheme of backscattering process is shown in Fig. 1. An ion with mass m_1 moving with the velocity of v_0 and kinetic energy E_0 interacts with an atomic nucleus with mass m_2 at rest. After collision, the incident ion moves at a scattering angle of θ respect to the incident direction, with the velocity v_1 and kinetic energy E_1. The nucleus is recoiled at an angle ϕ with velocity v_2 and kinetic energy E_2.

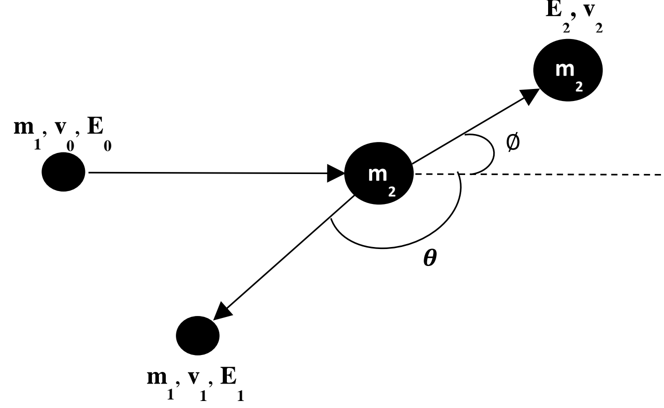

Fig. 1. The scheme of an elastic collision between a projectile with mass m_1, moving with speed v_0, kinetic energy E_0 and an atomic nucleus with mass m_2 at rest.

By applying the laws of energy and momentum conservation, we obtain the energy ratio of incident particle after and before collision that is called *kinematic factor K*:

$$K = \frac{E_1}{E_0} = \frac{m_1 \cdot v_1^2}{m_1 \cdot v_0^2} = \left[\frac{m_1 \cdot \cos\theta + \left(m_2^2 - m_1^2 \cdot sin^2\theta\right)^{1/2}}{m_1 + m_2} \right]^2 \quad (1)$$

We can calculate m_2 from this formula if the other terms were known. This calculation represents the possibility of RBS method in determination atomic mass of elements and allows us to answer the first question.

In a RBS experiment a detector is placed at the scattering angle θ to detect the ions backscattered into the solid angle Ω. In order to calculate *differential scattering cross-section*, the Rutherford's formula is usually used in the most case. That is the reason why this method is called "Rutherford backscattering spectrometry". In the framework of laboratory system, the formula is given as follows:

$$\frac{d\sigma}{d\Omega} = \left(\frac{Z_1 Z_2 e^2}{2E}\right)^2 \cdot \frac{\left\{\cos\theta + \left[1 - (m_1/m_2 \sin\theta)^2\right]^{1/2}\right\}^2}{\sin^4\theta \left[1 - (m_1/m_2 \sin\theta)^2\right]^{1/2}} \quad (2)$$

As we can see at this formula, *differential scattering cross-section* is proportional to Z_2^2, it means that the scattering capability of ions on atoms of heavy elements is much greater than on atoms of light elements. This relation shows that RBS is the method possesses a great sensitivity for heavy elements and less sensitive for light elements. Even if the target atoms are lighter

than incident ions $(Z_2 < Z_1)$, backscattering cannot occur, the elements exist in target that are lighter than the incident ions cannot be found by using RBS method. Usually, the ERD method is combined with RBS to solve this problem.

The solid angle Ω of a surface-barrier detector is usually very small, it could be regarded as a differential solid angle $d\Omega$. Thus RBS method ordinary uses the value of *average differential scattering cross-section*, it also can be called as *scattering cross-section* and is given by:

$$\sigma = \frac{1}{\Omega} \int_{\Omega}^{\frac{d\sigma}{d\Omega}} d\Omega \tag{3}$$

Substitute the formula (3) into the equation of total number of detected particles in the RBS experiment:

$$A = \sigma\Omega \cdot Q \cdot Nt \tag{4}$$

we can deduce *amount of atoms in a unit area* (Nt). This calculation allows us to answer at the second question.

When an incident ion with initial energy E_0 penetrates into solid, it collides with atoms and losses energy proportionally to the distance of path traveled. At a certain depth x, ion can be backscattered and lose more energy when it travels back out the sample. This process is illustrated in Fig. 2.

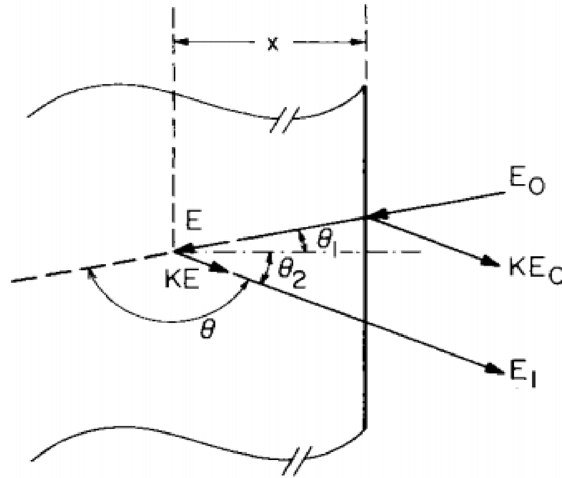

Fig. 2. The schematic representation of backscattering process in a sample, θ_1 and θ_2 are the angles of incident direction and backscattered direction respect to the normal of the sample surface respectively.

The difference between the energy of the ion backscattered at surface and the energy of the ion reached the detector after backscattering at depth of x is determined by:

$$\Delta E = KE_0 - E_1 = \left[\frac{k}{\cos\theta_1} \frac{dE}{dx}\bigg|_{in} + \frac{1}{\cos\theta_2} \frac{dE}{dx}\bigg|_{out} \right] \cdot x \tag{5}$$

where subscripts *in* and *out* refer to the values of energy loss along the inward and outward paths.

We can rewrite (5) by

$$\Delta E = [S] \cdot x, \tag{6}$$

where $[S]$ is the *energy loss factor*:

$$[S] = \left[\frac{k}{\cos\theta_1} \frac{dE}{dx}\bigg|_{in} + \frac{1}{\cos\theta_2} \frac{dE}{dx}\bigg|_{out} \right] \tag{7}$$

The equation (6) can be written in terms of stopping cross-section ε:

$$\Delta E = [\varepsilon] \cdot Nx, \tag{8}$$

where $[\varepsilon]$is called the *stopping cross-section factor; N* is atomic density of elements contained in the layer of thickness x.

We can deduce x from the relation between x and the energy of backscattered particles in the equations (6) or (8). This calculation allows us to answer how depth the elements are. In other words, the RBS method is able to measure the thickness of a thin layer near the surface region of samples.

II.2. Elastic Recoil Detection

The basic concepts of RBS method definitely lead to the possibilities of this method as presented above. However, it is necessary to mention to another method that is usually combined with RBS in order to overcome the limitation of this method in identification of light elements. We usually carry out the RBS measurements together with the other nuclear analytical method that named Elastic Recoil Detection (ERD) method [12]. Because the incident projectiles in this case are heavier than certain target atoms, after collision the incident ions break target nuclei from atomic bond, the light nuclei become recoiled particles. The elastic recoil process is illustrated in Fig. 3. Basically, this process can be described similarly by the principle of an elastic collision as backscattering process, difference point is the recoil particles instead of backscattering ions. The possibilities of ERD method thus come from the basic concepts as well. Energy transfer from incident ions to the target nucleus lead to the concept of *kinematic factor* and relative to the possibility of mass identification. Capability of the interaction between two particles lead to the concept of *scattering cross-section,* represent to the possibility of quantitative analysis. Finally, energy loss of an ion moving through a medium leads to the concept of *stopping cross-section* and lead to the possibility of the depth (or thickness of a thin layer) determination.

In an ERD experiment, a filter is putted in front of the detector in order to stop the incident particles that are scattered at the nuclei of the heavy ions containing in the target. The thickness of the filter is chosen so that the recoil nuclei can be passed through filter with minimal energy loss, while the heavier particles are stopped completely. If incident beam is He^4 ions, only protons, deuterons and tritons can pass through the filter and they are recorded by detector. ERD method is applied in this case as a method for determination of depth profile of hydrogen's isotopes. Usually, ERD measurements are carried out simultaneously with RBS method, this combination allows to investigate the atomic concentration, depth distribution of individual elements having any mass as well as determine the thickness of layers in near surface region of solids.

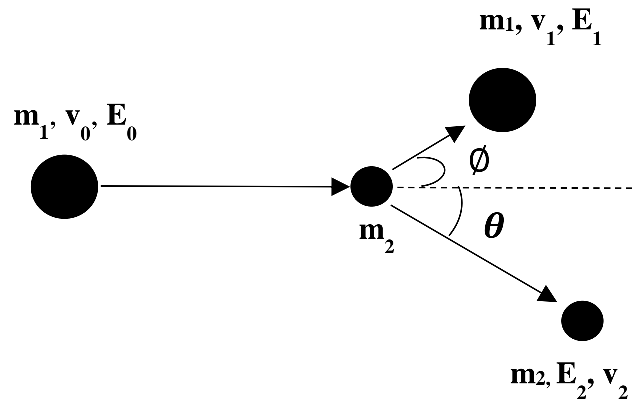

Fig. 3. The scheme of an elastic recoil collision between an ion with mass m_1, moving with speed v_0, kinetic energy E_0, and an atomic nucleus of light element with mass m_2 at rest $(m_2 < m_1)$.

III. EXPERIMENT

In this study the RBS and ERD experiments were carried out simultaneously with the experimental setup is shown in Fig. 4. He^4 ions were accelerated up to energy $E = 2297$ keV by the Van de Graaff accelerator EG-5 at FLNP, JINR, Dubna. The ion beam was used to bombard the studied samples under an angle $\alpha = 75^\circ$ respect to the normal of sample surface. RBS detector was located at backscattering angle $\theta = 135^\circ$ when the first sample was investigated. The backscattering angle was changed at $\theta = 120^\circ$ for the second sample's measurement. The recoiled particles was recorded by the ERD detector that was located at the same recoil angle $\theta = 30^\circ$ for both measurements. An aluminum filter with thickness about 6 μm was putted in front of ERD detector to stop all incident ions scattered in the same direction with the recoiled particles.

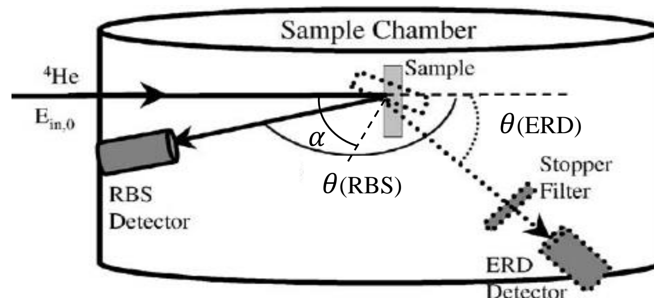

Fig. 4. The experimental setup of the nuclear methods. α is the angle between direction of incident beam and the normal of the sample surface. $\theta(RBS)$ and $\theta(ERD)$ is the backscattering angle and recoil angle of the detector positions respectively.

Depth profiles of all elements that are contained in samples were calculated from the RBS and ERD spectra with the help of the computer code SIMNRA [13]. Using SIMNRA, the imaginative models of MOS structures were calculated, each model consists of some layers containing one or more elements with certain concentrations. In the calculation, the parameters such as the thickness of layers, composition and concentrations of elements were entered in the trial model of the SIMNRA code. These activities were performed repeatedly until the simulated spectrum achieved a good agreement with the experimental spectrum for the same sample. Although the calculated models of samples have many different layers, they simulate for the MOS samples with similar structures as shown in Fig. 5.

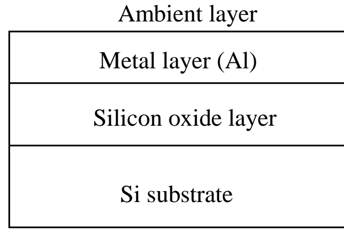

Fig. 5. The drawing of a MOS-structure sample. The ambient layer is a thin film (includes light elements such as H, C, O...) that covers the surface of sample.

The thickness of the model layers can be presented in several different units (typically in [atoms/cm^2], [nm] or [μm]). The thickness in unit [atoms/cm^2] is converted to [nm] as follows:

Firstly, the atomic density is determined by:

$$D = \frac{A}{V}\left[\frac{atoms}{cm^3}\right], \tag{9}$$

where, A is the Avogadro's number, $V = M/\rho$: mol volume (M $[g]$ is mol weight and ρ [g/cm^3] is volume density).

We can rewrite the atomic density by:

$$D = \frac{A\cdot\rho}{M}\left[\frac{atoms}{cm^3}\right]. \tag{10}$$

Then a layer with thickness $t\cdot 10^{15}$ [atoms/cm^2] is transformed to $[nm]$ by the formula:

$$T = \frac{t\cdot 10^{15}}{D} = \frac{t\cdot 10^{15}\cdot M}{A\cdot\rho}\,[cm] = \frac{t\cdot 10^{15}\cdot M}{A\cdot\rho}\cdot 10^{7}\,[nm] \tag{11}$$

IV. RESULTS AND DISCUSSION

The RBS and ERD spectra collected on the first sample are shown in Fig.6a and b, respectively. In RBS spectrum, the peak at energy about 970 keV indicates Heions backscattered on oxygen's nuclei that are contained at surface layer of the sample. The peak near 850 keV corresponds to oxygen atoms contained in silicon oxide layer. The left edge of minimum curve at energy about 1200 keV is related to silicon in the substrate. The kinematic border near 1370 keV indicates the energy of He ions backscattered on aluminum's atoms at the surface layer. In the

ERD spectrum, the border that is near 750 keV is connected with hydrogen atoms at the surface of sample. The low band at energy region between 350 keV and 600 keV indicates hydrogen atoms in the subsurface layers.

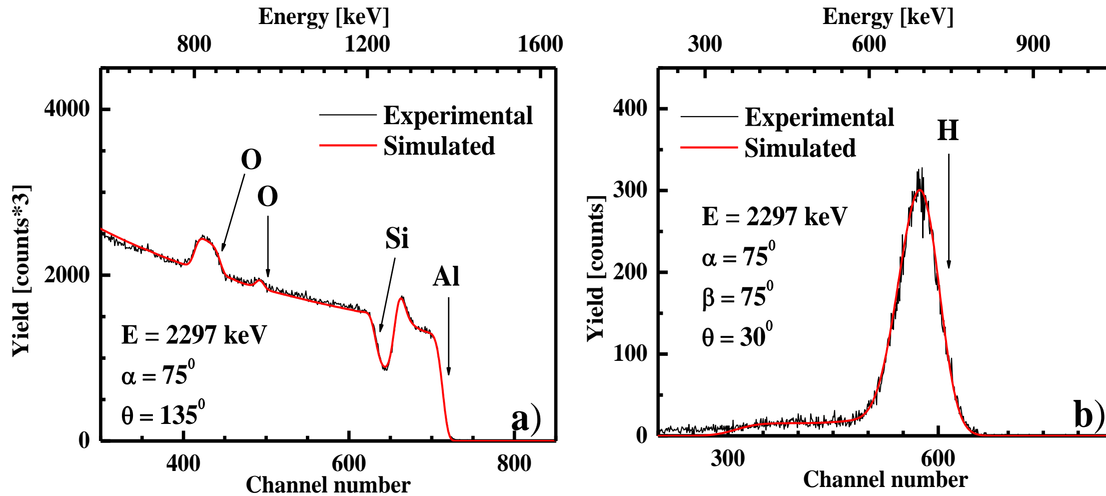

Fig. 6. The RBS (a) and ERD (b) spectra collected from the first sample. The vertical and inclined arrows indicate kinematic borders for atoms in the surface layer and in more depth layer, respectively.

The depth profile of all elements are contained in the first sample is shown in Table 1. The model of this sample consists from 9 layers, which provides a good agreement for the experimental and simulated spectra. The first model layer is an ambient film including light elements H, C, O, they are mixed at the top of aluminum layer. Under this thin film, aluminum layers (model layers 2 - 4) are mixed with hydrogen in low concentrations. If we assume that the atomic density of these layers is near the density of pure aluminum, the total thickness of them can be calculated by equation (11), the calculated result is about 116 nm. At the bottom of these aluminum layers is a transitional region (model layers 5) that is mixed by silicon from the silicon oxide layers below (model layers 6-7). Based on the atomic concentration values of the silicon and oxygen, we expect that these elements are possibly relevant to the chemical compound SiO_2. So, the value of density as pure $SiO_2$was used for the calculation of the total thickness of the model layers 6 and 7, and the calculated result is about 50 nm. All seven model layers are placed on a substrate which is a thick pure silicon layer (model layer 9) with the top (model layer 8) is silicon mixed with hydrogen in 0.7 at.% concentration.

The RBS spectrum collected from the second sample is presented in Fig. 7a. This spectrum has a visible peak at energy near 830 keV, this peak is connected with He^4 ions scattered on carbon atoms at surface layer. The peak at energy near 1100 keV is related to oxygen at surface of aluminum layer. The edge at energy about 1400 keV is connected with Si atoms in the substrate. Aluminium atoms at the surface of sample are indicated by the sharp border at energy near 1500 keV. In this sample two more heavy elements were found in low concentrations, they are indicated by the peaks near 1800 keV and 1900 keV. These peaks are connected with He^4 ions

Table 1. Depth profiles of all elements are contained in the first sample.

Number of layers	Thickness [10^{15} atoms/cm^2]	Atomic concentration [at.%]				
		H	C	O	Al	Si
1	120	33.00	2.00	25.00	40.00	0.00
2	440	10.00	0.00	0.00	90.00	0.00
3	115	0.70	0.00	4.30	95.00	0.00
4	140	0.70	0.00	0.80	95.00	3.50
5	140	0.70	0.00	50.30	20.00	29.00
6	240	0.70	0.00	56.30	14.00	29.00
7	110	0.70	0.00	62.61	0.00	36.69
8	900	0.70	0.00	0.00	0.00	99.30
9	4000	0.00	0.00	0.00	0.00	100.00

backscattered from Ti and Cu nucleons, respectively. The ERD spectrum collected from this sample is shown in Fig. 7b, the kinematic border at energy near 700 keV is connected with hydrogen at surface layer of sample.

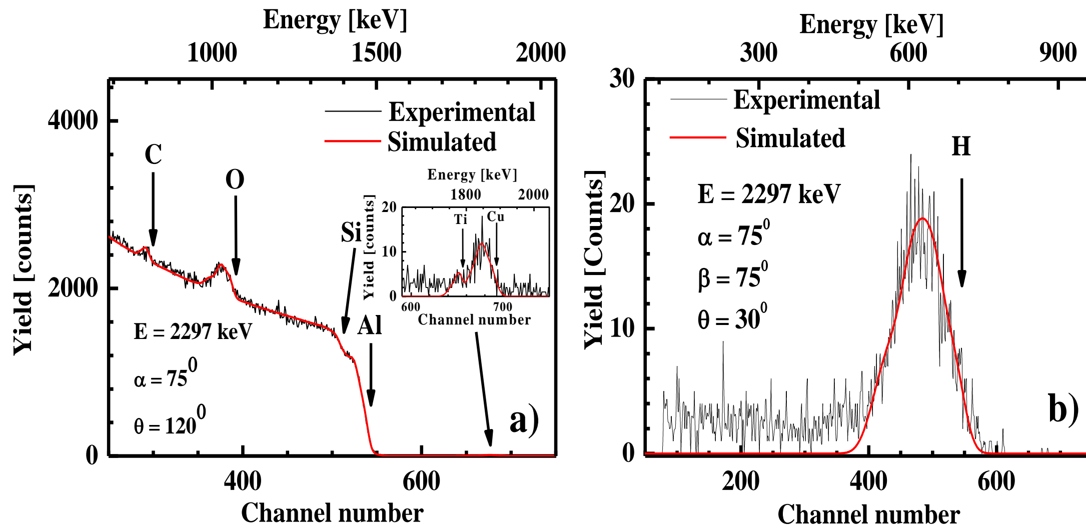

Fig. 7. The RBS (a) and ERD (b) spectra collected from the second sample. The vertical and inclined arrows indicate kinematic borders for atoms in the surface layer and in more depth layers, respectively.

Table 2 presents the atomic concentrations for all elements and thickness of 10 model layers in the second sample. The model layers 1, 2 consist of aluminum mixed with light elements H, C, O and more heavy elements Ti, Cu. The heavy elements Ti and Cu were found in surface region of this sample with very low concentrations, changing of Ti concentrations is within 0.05 - 0.2 at.% and within 0.02 – 0.2 at% for Cu. Under the thin metal film there are layers of silicon oxide (model layers 3-4), their atomic concentrations can be considered as the chemical compound SiO_2. The

transitional region (model layers 5-6) under SiO_2 layers contains more concentrations of silicon that diffuse from the Si substrate. At the top of Si substrate (model layer 7), hydrogen was found with the maximum concentration (4.6 at.%). In more depth (model layers 8,9), Si is mixed by hydrogen with decreasing concentration up to zero in Si substrate (model layer 10).

Table 2. Depth profiles of all elements are contained in the second sample.

Number of layers	Thickness [10^{15} atoms/cm^2]	Atomic concentration [at.%] H	C	O	Al	Si	Ti	Cu
1	110	1.0	53.78	32.0	13.0	0.0	0.20	0.02
2	190	1.8	22.05	41.0	15.0	20.0	0.05	0.10
3	80	2.0	0.00	65.0	0.0	32.8	0.00	0.20
4	70	2.5	0.00	49.4	0.0	47.9	0.00	0.20
5	170	4.3	0.00	26.1	0.0	69.5	0.00	0.10
6	120	4.5	0.00	20.5	0.0	75.0	0.00	0.00
7	200	4.6	0.00	3.0	0.0	92.4	0.00	0.00
8	300	2.3	0.00	0.0	0.0	97.7	0.00	0.00
9	150	1.0	0.00	0.0	0.0	99.0	0.00	0.00
10	5000	0.0	0.00	0.0	0.0	100.0	0.00	0.00

A graphical representation for changes in atomic concentrations of H, C, O, Al, Si relative to depth in both samples is shown in the Fig. 8. The combination of both analytical methods allows to investigate the content of elements in the near-surface layers with the maximum depth about 1 μm. Depth profiles of elements were measured with a depth resolution about 2 nm (the result was calculated at surface of aluminum layer in the first sample).

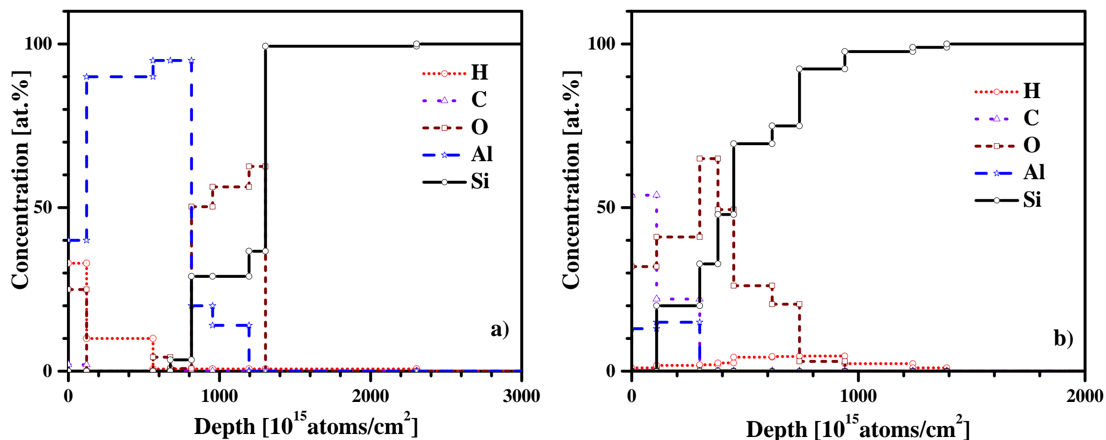

Fig. 8. The graphical representation of the depth profiles for different elements in the first (a) and the second sample (b).

CONCLUSIONS

The study of MOS structures using the nuclear analytical methods were presented in this paper. Depth profiles of different elements in the samples were investigated and described in detail. The combination of the RBS and ERD methods allows to identify any element with atomic masses of elements in range from H up to Cu. The minimum value of atomic concentrations that could be determined is about 1 at.% for light elements and achieved to 0.02 at.% for heavy elements. Using 2297-keV He ions allows to investigate the depth distribution of elements at the maximum depth of about 1 μm. The near-surface area of MOS structures were analyzed at a depth resolution of about 2 nm. The pointed characteristics of the nuclear analytical methods depend on the element contents of the investigated samples. In addition, it should be noted that, the experimental conditions (such as energy and atomic number of the ion beam) can be changed to get the optimal analytical results.

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