

## INVESTIGATION OF STRUCTURE AND PROPERTIES OF BURY BARRIER LAYERS AT LOW ENERGY CARBON AND OXIGEN IONS

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### ABSTRACT

There is actual investigation of the processes creating the buried barrier layers that prevent diffusion of high-temperature coating materials because of the development of ion and ion-plasma technologies, surface treatment of material.

In the present work we discuss the results on the thermal stability, structure, physical and mechanical properties of the buried barrier layers formed  $\sim 2 \cdot 10^{18}$  ion/sm<sup>2</sup>, by ion implantation of oxygen and carbon (the dose the energy of 1.5 – 2 MeV) accelerator to the DC-60.

Nuclear spectroscopic techniques involving X-ray analysis systematic studies of the influence of the buried barrier layer on the thermally induced processes in the layered system Fe-Be. It is established: the sequence of phase transitions in the surface layers and inside the sample during isothermal annealing. It is shown that the implanted oxygen ions buried barrier layer in the matrix of  $\alpha$ -Fe slow mutual diffusion of beryllium atoms and iron atoms. The kinetics of the process of mutual diffusion of Fe and Be in a solution  $\alpha$ -Fe (Be) for both multi-layered systems with a layer of implanted of oxygen and without it.

The evolution of the distribution of the oxygen implanted layer in the copper and the effect of thermal annealing. It is shown that even at an annealing temperature of  $\sim 200^\circ\text{C}$  in this system is the diffusion of oxygen into the sample of copper. Consequently, the oxygen cannot be used as a subsurface barrier layer in copper, in contrast to iron, where oxygen-implanted layer remains stable at much higher temperatures.

The research phase formation in iron implanted with carbon and deposited on the surface layer of beryllium. It is established that the sample implanted with a layer of carbon formation on the surface FeBe<sub>2</sub> phase begins after 5 hours annealing at 650°C. For the case without implantation – education phase FeBe<sub>2</sub> not fixed.

**Key words:** low-energy ion implantation of oxygen and carbon, buried barrier layers, thermal stability, phase formation

### INTRODUCTION

Ion-beam modification is a promising method effects on materials to make them specific properties. For the directed modification of the surface layers of metallic materials to improve the surface characteristics of widely used meth-

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ods for ion-plasma coatings.

The magnetron deposition is generally used subsequent heat treatment of the material, resulting in improved coating adhesion to the substrate, the formation and homogenization of the phases in the diffusion zone. The initial non-equilibrium distribution of nuclear components and subsequent annealing lead to the spatial orientation of the process of phase formation and the creation of a layered system.

There is necessary to create a thermally stable inhomogeneous distribution of phases over the depth of the sample for practical application of such systems. Elaboration of methods for non-uniform distribution of phases, stable to temperature, is an interesting from a scientific point of view and practically important problem.

One of the promising ways to create a thermally stable layered system is the combination of two methods - ion implantation and magnetron deposition of protective coatings. The method is as follows: in the material created by ion implantation layer (phase differs from the matrix) which is a barrier to the diffusion depth of material deposited on the surface of the coating.

As a result of consistent application of these two methods of forming a layered system - matrix located beneath the barrier layer and a protective coating. This system can be stable until the temperature to which the barrier layer is stable.

Interesting from this point of view, study of the effect of implanted oxygen in the iron (barrier layer) on the thermal stability deposited on the surface of the system of the beryllium layer (protective cover) [1]. The system iron-oxygen-beryllium is as good a model as well as practically important material. It is known that iron is an essential component of many construction materials. At the same time, the unique characteristics of nuclear, radiation and corrosion resistance, high melting point and thermal conductivity led to widespread use of beryllium in the nuclear and missile technology. For a binary system of iron-beryllium characterized by a great variety of structural and magnetic transitions [2, 3].

#### **METHODS OF EXPERIMENT. RESULTS AND DISCUSSION.**

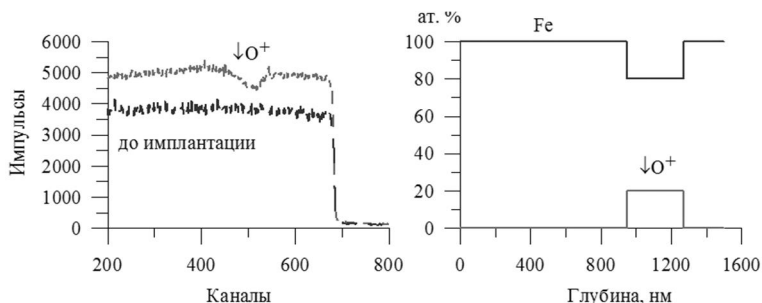
There were prepared by Armco foil - iron, 99.8% purity, a thickness of 10 microns and an area  $S = (10 - 15) \text{ mm}^2$ , with subsequent homogenizing annealing at  $T = 850^\circ\text{C}$  for 3 h for studies. The method of ion implantation in one of the foils Armco iron ions were introduced  $\text{O}^+$  dose of  $1.1 \cdot 10^{18}$  ions/cm<sup>2</sup> at an energy of 1.6 MeV and current density of  $2 \div 3 \text{ mA/cm}^2$ . Using the methods of magnetron deposition of atoms and vacuum sputtering of Be and <sup>57</sup>Fe, were obtained of  $\alpha\text{-Fe: O}^+ \text{-Be}(0.7 \mu\text{m})\text{-}^{57}\text{Fe}$  (0.1  $\mu\text{m}$ ) and  $\alpha\text{-Fe-Be}(0.7 \mu\text{m})\text{-}^{57}\text{Fe}$ (0.1  $\mu\text{m}$ ). Control the thickness of sputtered layers was carried out as a gravimetric method and by Rutherford backscattering of protons. The resulting

systems have been consistent, ie increments  $\Delta t_{\text{otzh}} = 2.0$  h isothermal annealing in a vacuum of  $5 \cdot 10^{-6}$  Hg. Art. at a temperature  $T = 700^\circ\text{C}$ .

There were prepared by the respective thicknesses of massive samples, selected irradiation conditions (dose  $\sim 2 \cdot 10^{18}$  ion/ $\text{cm}^2$ , energy  $\sim 2$  MeV, the type of bombarding particles - oxygen ions) to create a buried barrier layer on the accelerator of the DC-60. The target holder of high energy chamber was prepared and installed.

The distribution of the implanted oxygen atoms in the depth of the layered system (according to RBS). Determine the depth of penetration of oxygen-Fe and the oxygen concentration in the depth of the sample after implantation and appropriate anneal method was used Rutherford backscattering (RBS), proton accelerator complex charge – UKP-2-1.

Carrying POP – research and subsequent data processing program RUMP allows selective in depth, from  $0.03 \pm 0.02$  to  $1.5 \div 2 \mu\text{m}$ , to determine the elemental composition of the sample. To investigate the pattern of iron by RBS with an implanted layer of oxygen placed in the target chamber. The energy of the proton beam was 800 keV. The scattered beam is detected surface-barrier detector located at an angle  $\theta = 135^\circ$  to the beam. Sensitivity of the detector was 15 keV. *Figure 1* shows RBS - spectra before and after implantation, as well as the distribution profile of oxygen in the matrix layer of iron.



**Fig. 1** – RBS-spectra before and after implantation, as well as the distribution profile of oxygen in the matrix layer of iron

Let the rate of energy loss of ions (protons) per unit length is  $dE/dx$ . Then the total loss of energy  $\Delta E$  for the ion, which reached a depth  $t$ , is proportional to  $t$ :

$$\Delta E = \int_0^t (dE/dx) \cdot dx \approx dE/dx \Big|_{\text{in}} \cdot t,$$

where  $dE/dx \Big|_{\text{in}}$  – input - energy lost by the particle before scattering, calculated for the average energy between the initial energy  $E_0$  and  $(dE/dx) \cdot E_0$

–  $t$  at a depth of  $t$  the particle has energy:

$$E(t) = E_0 - t \cdot dE/dx \Big|_{in}$$

After the large-angle scattering particle energy becomes equal to  $E(t)$ ,  $k$  where the kinematic factor  $k$  is determined from the expression:

$$k = [(M_1 \cdot \cos\theta + (M_2^2 - M_1^2 \cdot \sin^2\theta)^{1/2}) / (M_1 + M_2)],$$

$M_1$ ,  $M_2$  – mass of the ion (proton) and the target nucleus, respectively –  $\theta$ , scattering angle.

Measuring the energy of elastically scattered ions at a certain scattering angle  $\theta$  to determine  $M_2$ . By changing the direction of motion, the particle continues to decelerate on the way back to the detector and achieves it, having an energy:

$$\begin{aligned} E_1(t) &= k \cdot E(t) - (t/\cos\theta) \cdot dE/dx \Big|_{exit} = \\ &= -t \cdot (k \cdot dE/dx \Big|_{in} + (1/\cos\theta) dE/dx \Big|_{exit}) + k \cdot E_0 \end{aligned}$$

Then the width of the energy spectrum of  $\Delta E$  particles backscattered film thickness  $\Delta t$ , is:

$$\Delta E = \Delta t \cdot (k \cdot dE/dx \Big|_{in} + (1/\cos\theta) \cdot dE/dx \Big|_{exit})$$

Thus, knowing the width of the energy spectrum of  $\Delta E$  and using foregoing equality is possible to determine the thickness of the film.

Backscattering spectrometry allows to determine the change in composition with depth. The relationship between energy resolution and a resolution  $\delta E_1$  depth  $\delta t$  is given as:

$$\delta t = \delta E_1 / (k \cdot dE/dx \Big|_{in} + (1/\cos\theta) \cdot dE/dx \Big|_{exit})$$

The best sensitivity to the depth we have in the case when the maximum energy loss accompanying the scattering in the depth of the sample. This is achieved by using a sliding geometry can be obtained using standard semiconductor detectors permit to a depth of 2 nm. It is necessary to take into account the factors affecting resolution, such as the final corner of the capture detector, surface roughness, the energy straggling.

As seen from the value of  $k$  depends on the scattering angle  $\theta$ . This change is minimal when the angle  $\theta = 180^\circ$ . The difference of back-scattered particles from the elements with different  $Z$  is also a maximum at a scattering angle of  $180^\circ$ . Therefore, backscattering spectrometry, usually sold at angles  $\theta$  close to  $180^\circ$ . Here, the energy spread introduced by the limited solid angle of the detector is minimal, and the possibility of separating the atoms of the sample with

different masses is greatest. Currently, for this purpose has developed special annular surface-barrier detectors with a hole in the center of the beam.

For processing the spectra of backscattered ions and the analysis of the distribution of elements in depth with the POP method, used the program RUMP. Concentration profile of elements in the program RUMP appears in the form of successive alternating layers varying thickness and composition, the concentration of elements in which is specified as a stoichiometric formula. The number of layers of homogeneous composition is limited to 100, the number of elements in the layer is not limited. In addition to the simulation of the theoretical spectrum and the fitting procedures in interactive mode, the program allows for automatic adjustment of the theoretical spectrum to the experimental with the number of variable parameters, not exceeding 8. The error of measurement of film thickness by fitting the theoretical spectrum under the experimental spectrum is less than  $\Delta t/t \approx 6\%$ .

### **CONCLUSIONS**

For the first time the methods of Mossbauer spectroscopy on  $^{57}\text{Fe}$  nuclei and X-ray analysis of the systematic studies of the influence of implantation layer of oxygen on the processes of diffusion and phase transformations in the iron-beryllium. The sequence of phase transitions in the surface layers and in the volume of the sample during the isothermal annealing, and also defined the characteristic time of phase transformation process  $\text{FeBe}_{2+\delta} \rightarrow \text{Fe}(\text{Be})$ . It is shown that in the implanted layer of oxygen migration processes of beryllium atoms into the iron matrix slowed down, and then there is a layer of oxygen limits the zone of dissolution of beryllium and thus contributes to the processes of phase formation.

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