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THE STUDY OF KINETICS OF DIFFUSION AND PHASE FORMATION IN THE LAYERED IRON-BERYLLIUM SYSTEM

K. A. Kuterbekov,[1] S. A. Nurkenov,[1] S. B. Kislitsin,[2]
T. A. Kuketayev,[3] and T. N. Nurakhmetov[1]

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The methods of Mössbauer spectroscopy with X-ray phase analysis and Rutherford backscattering of protons were used to study the kinetics of diffusion and phase transformations in the layered iron-beryllium system. For the first time, the authors suggested and implemented a method for retardation of diffusion and phase formation processes in the layered iron-beryllium system using the barrier layer. It was established that the barrier layer limits the zone of beryllium dissolution in the area of implanted layer. The impact of the barrier layer on kinetics of thermally induced processes of diffusion and phase transformations in the layered Fe–Be *system was determined using the example of* Fe *(10 μm):* O^+ – Be *(0.7 μm)* – ^{57}Fe *(0.1 μm). The authors suggested and implemented a method for recovery of the distribution function of the admixture atom concentration in the solid matrix–admixture solution on the basis of the X-ray diffraction data. The kinetics of mutual diffusion was determined for* Fe *and* Be *atoms in the* α-Fe(Be) *solution for both sides of the layered systems with a barrier layer and without it using the suggested method for recovery of the distribution function of the* Be *atom concentration. It was established that for the system without a barrier layer, the share of iron atoms ends at* $t_{ann} \sim 5$ *h on the coating side and at* $t_{ann} \sim 7.5$ *h on the iron side, while for the barrier layer case – at* $t_{ann} \sim 20$ *h on the coating side and at* $t_{ann} \sim 40$ *h on the iron side.*

Keywords: barrier layer, diffusion process kinetics, phase formation, thermal annealing, phase transformations, intermetallic compounds.

INTRODUCTION

Ion implantation is an advanced method of surface alloying of materials [1]. Over the past years, this technology has been undergoing intensive development and finding wider application in various areas of science and medicine. The method of ion implantation is based on the insertion (implantation) of accelerated ionized ions and molecules into the solid body, and any ion–target combinations are possible. The depth of ion implantation depends not only on energy, but also on ion mass, as well as mass of the target atoms. The concentration of implanted admixture ions has a certain depth distribution that can be in the general case described by the Gaussian (normal) distribution with the average projected run R_p and standard deviation ΔR_p [2, 3].

Creation of protective and thermally stable materials on the basis of beryllium coatings on iron is a crucial task of today's materials science. Among a large class of intermetallic compounds in the Fe-Be system, beryllides are of practical interest as protective coatings, because they possess a complex of the most important physical, chemical and mechanical properties [4]. Beryllium is one of the most valuable construction reactor materials. Its unique thermal characteristics contribute to the reduction of thermal stresses in the active zone of nuclear reactors. It is not subject to

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[1] L. N. Gumilyov Eurasian National University, Astana, Kazakhstan, e-mail: kkuterbekov@gmail.com; S.Nurkenov@gmail.com; [2] Institute of Nuclear Physics, Almaty, Kazakhstan, e-mail: skislitsin@mail.ru; [3] Y. A. Buketov Karaganda State University, Karaganda, Kazakhstan, e-mail: katkargu@mail.ru. Translated from Izvestiya Vysshikh Uchebnykh Zavedenii, Fizika, No. 10, pp. 67–71, October 2016. Original article submitted October 23, 2015; revision submitted February 24, 2016.

corrosion under stress and has high corrosion resistance in the air, in humid CO_2, in Na, Bi and Pb at temperatures up to 500–600°C. As a construction material, Be surpasses graphite. Stainless steel is stronger than beryllium at any temperature, but it does not have a small cross-section of neutron absorption and thermal characteristics typical for Be. Under the impact of radiations, physical and mechanical properties of beryllium are relatively stable. A rather valuable property of Be for nuclear power equipment is the high ratio of durability to specific weight, especially at elevated temperatures [5–7]. Iron is also the main component of many construction materials. A large variety of structural and magnetic transformations is characteristic for the binary iron–beryllium system [8].

This paper presents the study of kinetics of diffusion and phase formation processes in the layered iron–beryllium system and considers the following tasks:

– impact of the barrier layer on the kinetics of thermally induced processes of diffusion and phase formation in the iron–beryllium system;

– implementation of the method of diffusion and phase formation retardation in the iron–beryllium system using the barrier layer;

– implementation of the method of recovery of the distribution function of the admixture atom concentration in the solid matrix–admixture solution based on the X-ray diffraction data.

Objects of study: armco iron sample (d_{Fe}) = 10 μm, subjected to homogenizing annealing at the temperature T = 700°C in the course of 3 hours, as well as layered systems: Fe (10 μm) – Be (0.7 μm) – ^{57}Fe (0.1 μm) and Fe (10 μm):O^+ – Be (0.7 μm) – ^{57}Fe (0.1 μm).

RESEARCH METHODS. RESULTS AND DISCUSSION

Sample preparation for research was carried out in the standard design vacuum furnace in vacuum P = $5\cdot 10^{-6}$ mm Hg, time to set temperature – 30 min, samples were cooling down with the furnace, temperature measurement error was no more than ±5°C. Substrate thickness was determined using a standard micrometer. Micrometer measurement error was ±1 μm. After rolling, the samples were subjected to homogenizing annealing at 850°C for 3 hours. Experiments on irradiation of these samples were carried out on tandem accelerator UKP-2-1 (Institute of Nuclear Physics, National Nuclear Center, Almaty, Kazakhstan) and heavy ion cyclotron DC-60 (accelerator at L. N. Gumilyov Eurasian National University, Astana). After carrying out the corresponding annealing sessions, at each stage research was performed using the methods of Möessbauer spectroscopy on ^{57}Fe nuclei, X-ray phase analysis and Rutherford backscattering of protons [9].

In this paper, the method of X-ray phase analysis was used to study the layered systems from both sample sides Fe:O^+ – Be (0.7 μm) – ^{57}Fe (0.1 μm) and Fe – Be (0.7 μm) – ^{57}Fe (0.1 μm) on time of isothermal annealing at temperature T_{ann} = 700°C.

The results of X-ray phase analysis were used recover the distribution functions $p(C_{Be})$ of beryllium atoms in iron for the studied systems in the angle range $115° \le 2\vartheta \le 120°$. Recovery of the distribution function was performed using the software complex MSTools [13, 14].

Fig. 1 presents the share of iron atom numbers of diffraction reflection (310) produced as a result of analysis of the distribution function recovery $p(C_{Be})$ for the layered systems Fe:O^+ – Be (0.7 μm) – ^{57}Fe (0.1 μm) and Fe – Be (0.7 μm) – ^{57}Fe (0.1 μm) on the time of isothermal annealing at temperature T_{ann} = 700°C.

Let us consider the following cases: Be-side system. One can see from Fig. 1 that in the Fe – Be system (0.7 μm) – ^{57}Fe (0.1 μm) (without a barrier layer) the share of free iron atoms is practically absent in the case of 5 h annealing. In the system Fe:O^+ – Be (0.7 μm) – ^{57}Fe (0.1 μm) (with a barrier layer) part of the free iron atoms reaches ~20 h of thermal annealing; for Fe-side – approximately 40 h in the system with a barrier layer, and practically ~7.5 h – in the system without a barrier layer.

One can see from Fig. 2 and 3 and that beryllium atoms actively migrate to matrix α-Fe with subsequent formation of the solid beryllium solution in iron α-Fe(Be). Maximum values 2ϑ for both systems in the course of time become ~(117.2 ± 0.1)°. The dependences produced indicate complete dissolution of beryllium atoms in the matrix α-Fe with the increase in time of isothermal annealing.

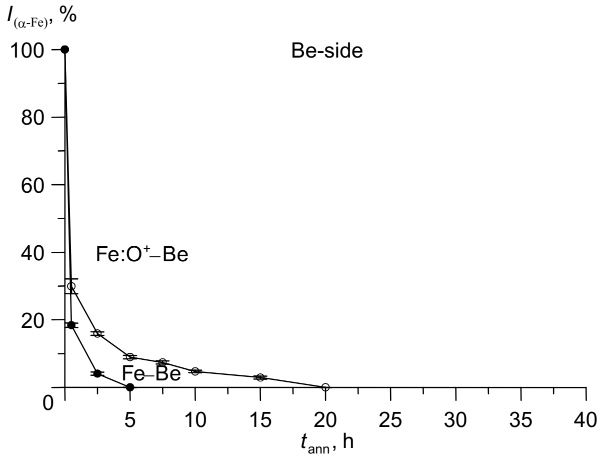


Fig. 1. The share of iron atom number of diffraction reflection (310) produced as a result of analysis of the distribution function recovery $p(C_{Be})$ for the layered systems Fe:O$^+$ – Be (0.7 μm) – ^{57}Fe (0.1 μm) and Fe – Be (0.7 μm) – ^{57}Fe (0.1 μm) on the time of isothermal annealing at temperature T_{ann} = 700°C.

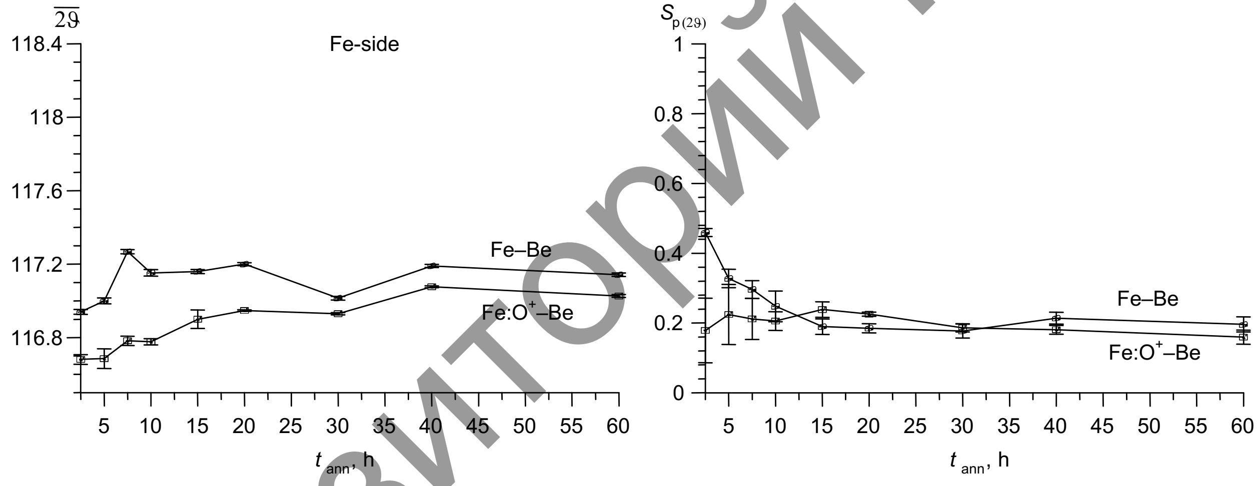


Fig. 2. Dependences obtained by the method of recovery of the distribution function, average value 2ϑ and standard deviation $S_{p(2\vartheta)}$ of X-ray patterns taken on the iron side for the systems Fe–Be and Fe:O$^+$–Be, on the time of isothermal annealing t_{ann} at temperature T_{ann} = 700°C.

Fig. 4 presents the dependences of the average value 2ϑ and standard deviation $S_{p(2\vartheta)}$ of X-ray patterns produced by method of distribution function recovery: *a* – on the side of beryllium coating, *b* – on the side of iron. One can see that with the increase in time of isothermal annealing, the average values of 2ϑ and standard deviation $S_{p(2\vartheta)}$ of X-ray diffraction patterns for both systems become almost identical.

As a result of research, it was established that one observes the same sequence of phase transformations in the systems Fe–Be–^{57}Fe and Fe:O$^+$–Be–^{57}Fe, but in the layered system without a barrier layer the processes of diffusion and associated phase transformations proceed much faster than in the system with a barrier layer:

$$\text{Fe:O}^+ - \text{Be} - {}^{57}\text{Fe} - \alpha\text{-FeBe}_{2+\delta} - \alpha\text{-Fe(Be)},$$

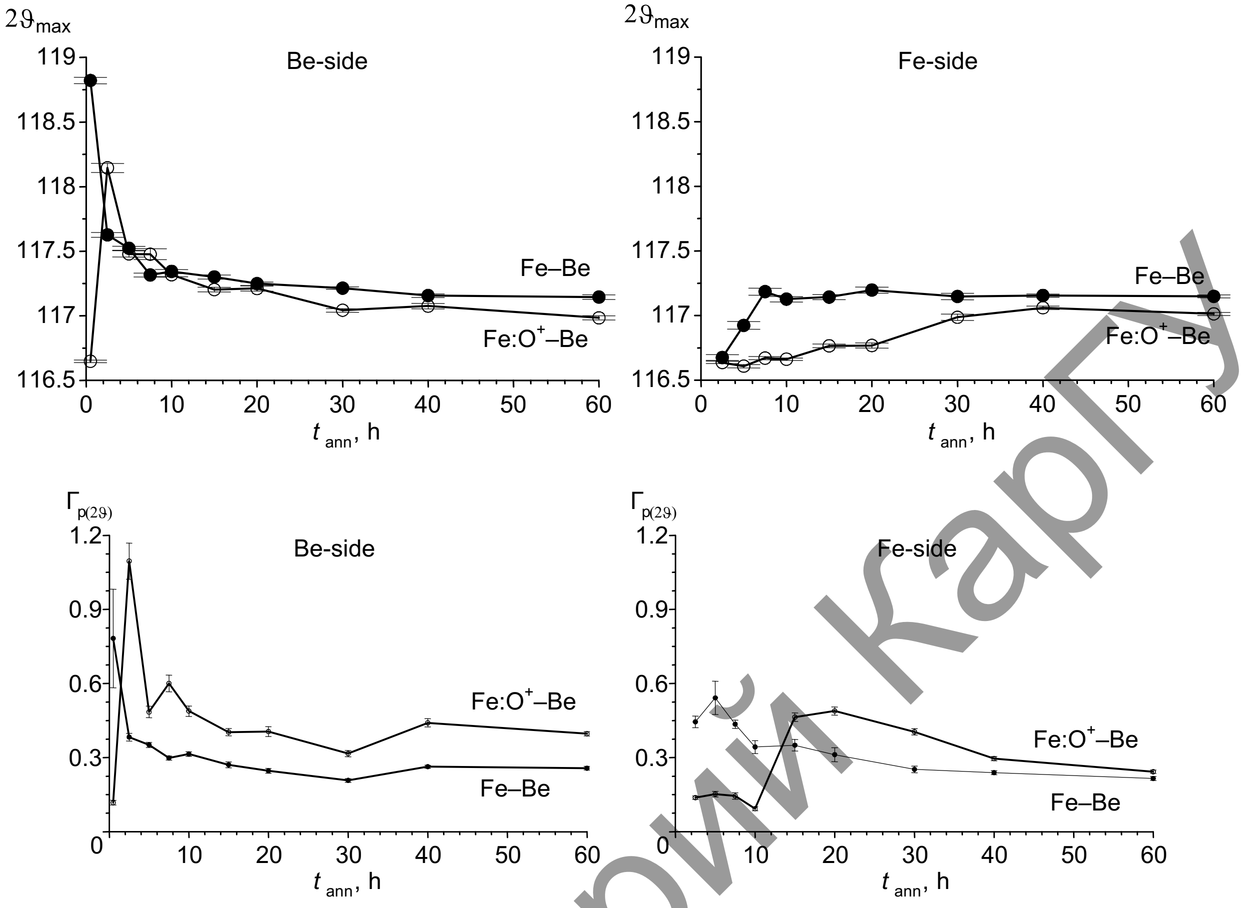


Fig. 3. Dependences produced by the method of recovery of the distribution function, maximum value of 2ϑ and widths $\Gamma_{p(2\vartheta)}$ for the systems Fe–Be и Fe:O$^+$–Be, taken on the side of beryllium coating and iron, on the time of isothermal annealing t_{ann} at temperature T_{ann} = 700°C.

$$\text{Fe:Be} - {}^{57}\text{Fe} - \alpha\text{-FeBe}_{2+\delta} - \alpha\text{-Fe(Be)}.$$

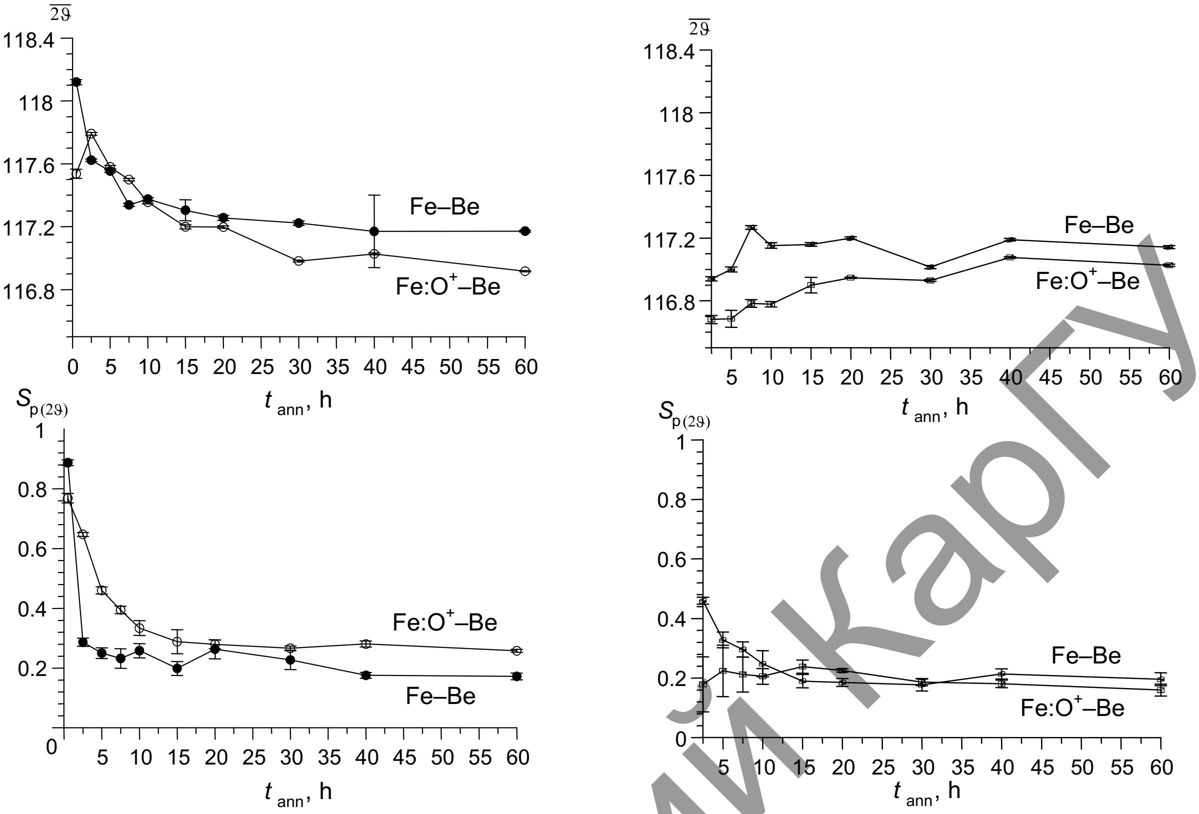
Taking the above-mentioned facts into account, we believe that the experimental data presented indicate the retarded migration of beryllium atoms deeper into the matrix in the barrier layer area.

CONCLUSIONS

With the overall time of isothermal annealing (t = 15 h), according to the data of X-ray diffraction in the system Fe (10 μm):O$^+$ – Be (0.7 μm) – ^{57}Fe (0.1 μm), formation of the solid solution α-Fe(Be) on the side of iron was identified for the first time.

For the first time the method of recovery of the distribution function of the admixture atom concentration was suggested and implemented in the solid matrix–admixture solution.

Kinetics of the mutual diffusion was determined for Fe and Be atoms in the α-Fe(Be) solution for both sides of the layered systems with a barrier layer and without it, using the suggested method of recovery of the distribution function of the Be atom concentration.



a) on the beryllium coating side

b) on the iron side

Fig. 4. The dependences produced by method of recovery of the distribution function, average value 2ϑ and standard deviation $S_{p(2\vartheta)}$ of X-ray diffraction patterns taken on the side of iron for the systems Fe–Be and Fe:O$^+$–Be, on the time of isothermal annealing t_{ann} at temperature T_{ann} =700°C.

It was also established that for the system without a barrier layer, the expenditure of the iron atoms share ends at t_{ann} ~ 5 h on the side of coating and at t_{ann} ~ 7.5 h on the Fe side, while in the case of the barrier layer, the time of irons atom share expenditure reaches around 20 h on the side of coating and around 40 h on the iron side.

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