STABILITY IN WIDE TEMPERATURE INTERVAL COATING OF BERYLLIUM ON IRON OBTAINED BY MAGNETRON SPUTTERING METHOD

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INTRODUCTION

Progress in present-day materials techniques has led to the construction of novel materials operating under extreme conditions of high temperatures, intensive mechanical loads, aggressive and contacting environments, and external irradiation. Due to various surface and volume requirements, traditional technologies for the production of materials with various compositions and surface or bulk structure cannot be used. One possible way to solve this problem is to create and apply protective coatings on selected materials. Indeed, that is what is commonly done nowadays. In fact, high-quality coatings are most frequently produced with ion-beam technologies, wherein surfaces, and surface properties, are modified by ion implantation or deposition of protective coatings via an ion-plasma sputtering technique.

In this paper we present a technique for obtaining stable coatings of beryllium on iron and investigate the coatings' stability under irradiation by alpha particles. The methodology for production of such coating is based on a thermodynamic approach developed by the present authors [1,2].

PRODUCTION OF STABLE BERYLLIUM COATINGS ON IRON FOILS

In the thermodynamic approach, a high temperature-stable beryllium-enriched layer on iron can be created when the material is a saturated solid solution of beryllium in alpha iron. In this material, any surface-deposited beryllium will have negligible diffusion penetration into the iron volume; thus a structure with a high content of beryllium (e.g., the $FeBe_2$ phase) will be formed at the surface. According to the Be-Fe equilibrium phase diagram (Figure 1), beryllium concentration is 10-12% in alpha iron at temperature 750 °C. Therefore, if we first create a saturated Be-Fe solid solution in the α-iron, then any further deposited beryllium layer over this structure should be stable up to 750 °C.

Another way to create a stable coating is to deposit a thin layer of beryllium on both sides of a thin layer of α-iron then heat to 750 °C. The thickness of the beryllium layers and the duration of heating should be such that, during annealing, a beryllium saturated solid solution is generated, and on the surface remains an excess of beryllium in the form of enriched $FeBe_2$ phase. We have created such samples using 10 μm α-iron foil and find that the thickness of the beryllium layers should not be less than 1 μm on each side.

The specimens were made of α-Fe foils (enriched by ^{57}Fe isotope up to 89 at. %), rolled to ~10 μm thickness and subjected to homogenization annealing in vacuum at 800 °C for 2 hours. The Be was deposited onto the iron foil via magnetron sputtering [3]. The beryllium layer was deposited to 1.3 μm on one side of the specimen (side A) and was deposited to 0.9 μm on the other side (side B). Samples underwent a series of isothermal annealing at temperature 720 °C for 0.5, 1, 2, 5, 10, 15, 20, 50 and 100 hours. The annealing vacuum was $5 \cdot 10^{-6}$ mmHg. After each annealing period the specimen structure was determined by the various techniques of: conversion-electron Mossbauer spectroscopy (CEMS) in back scattering geometry; the γ-ray technique in absorption geometry; and X-ray diffraction pattern. Control of deposited Be layer depth as well as determination of the iron concentration profile over the depth have been implemented by the Rutherford backscattering method (RBS) using the electrostatic accelerator UKP-2-1 at the Kazakhstan Institute of Nuclear Physics.

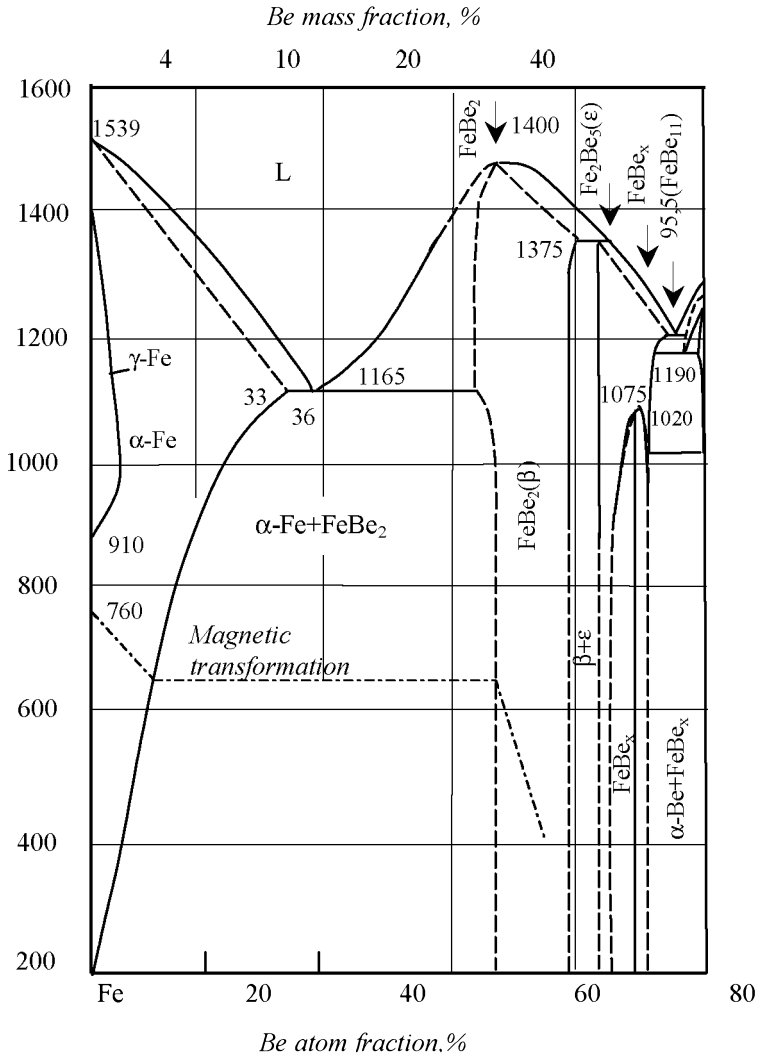


Figure 1. Equilibrium phase diagram for the Fe-Be system.

The structure of the specimen surface was studied by means of optic (OM) as well as scanning electron microscopy (SEM).

As a result of the annealing described above, a phase-stable Be-Fe-Be system formed along the depth of the specimens. $FeBe_2$ phase with ~66% beryllium concentration formed near the surface on both sides (A and B) of specimens. The $FeBe_2$ phase thickens along the depth of the specimens for ~0.7 μm. Solid solution of beryllium in alpha-iron with ~10% beryllium content is in the form of a matrix. For various Be content, a stable phase is achieved with ~10 hours annealing time. Further annealing up to 100 hours at temperature 720 °C does not change the structural phase distribution in the specimens.

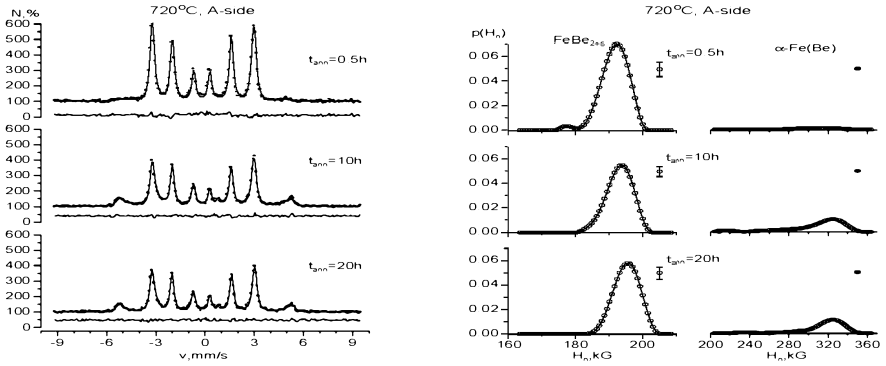
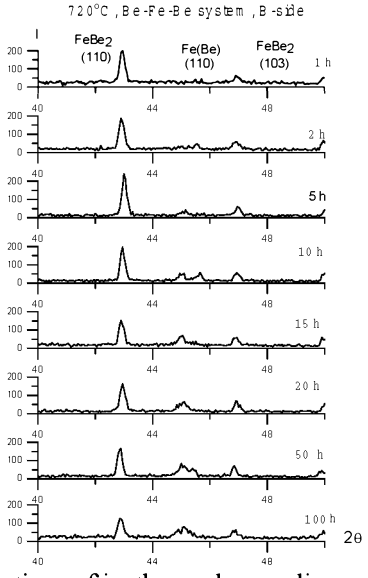


Figure 2. Mossbauer CEMS spectra taken from side A of Be-deposited samples (Fig. a, at left) and the restored distribution functions of the partial-spectra hyperfine interaction parameters (Fig. b, at right): magnetic field $p(H_n)$ for ^{57}Fe nuclei in the Be-Fe-Be



system after appropriate time of isothermal annealing.

Figure 3. The X-ray diffraction patterns for the Be-Fe-Be system after appropriate time of isothermal annealing.

Figures 2 and 3 show results from Mossbauer spectroscopy and X-ray diffractometry, to explore the thermal stability of the generated distribution of phases in the samples. The data clearly shows that the surface structures do not change for annealing time above 10 hours.

In brief, using the procedure described above, sputtering layers of beryllium onto both side of thin foils of iron, followed by isothermal annealing at 720 °C, one can obtain stables structures for 10 hours (or more) of annealing. A saturated solid solution of beryllium-iron is formed in a matrix and enhanced with beryllium layers of $FeBe_2$ near the surface of the foil. The depth distribution of Fe and Be relative to the foil surface is shown in Fig. 4, and is confirmed by Mossbauer spectroscopy and X-ray diffractometry techniques.

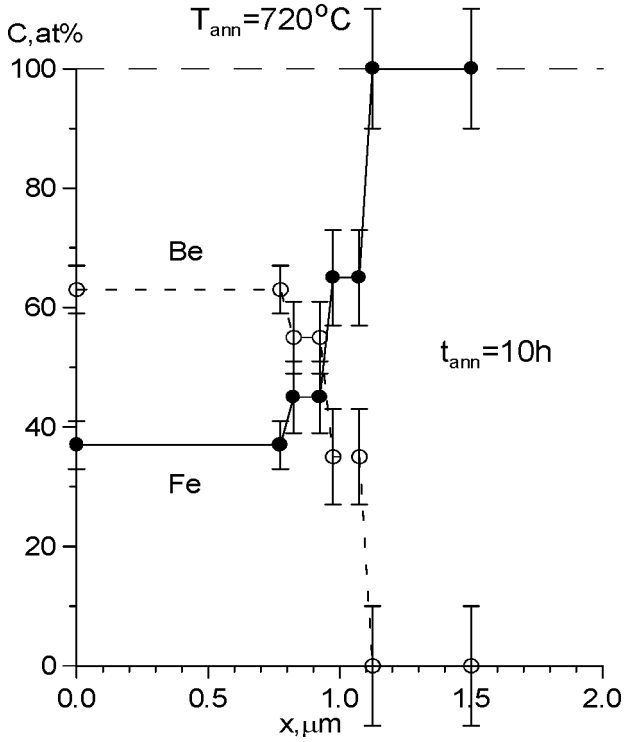


Figure 4. The depth distribution of Fe and Be concentration versus depth of specimen (side A) after 10 hours of annealing, characterized by RBS method.

INVESTIGATIONS OF STABILITY OF THE BE-FE-BE SYSTEM UNDER ALPHA-PARTICLE IRRADIATION

Samples with stable beryllium coating was irradiated by alpha particles from the Wezuvii accelerator (at the Kazakhstan Institute of Nuclear Physics) to test for radiation resistance, e.g., stability of the phase structure, formation of gas bubbles etc. The irradiation scheme is shown in Figure 5. Samples were irradiated under the following conditions:

- Energy of irradiating particles: 330 keV,
- Total dose: $3 \cdot 10^{16}$ particles /cm^2,
- Temperature of irradiation: not more than 100 °C.

The main purpose of the present was to determine the possible dissolution of the beryllium-enriched $FeBe_2$ phase due to irradiation. Therefore the energy of the alpha particles was selected so that longitudinal particle straggling was 1.09 microns, i.e. maximum radiation damage was concentrated near the surface region enriched by beryllium. A computer program known as "TRIM" [5] was used to calculated the projected range of the alpha particles. These values are shown in Table 1. So, alpha particles with 330 keV energy were expected to satisfy to these requirements.

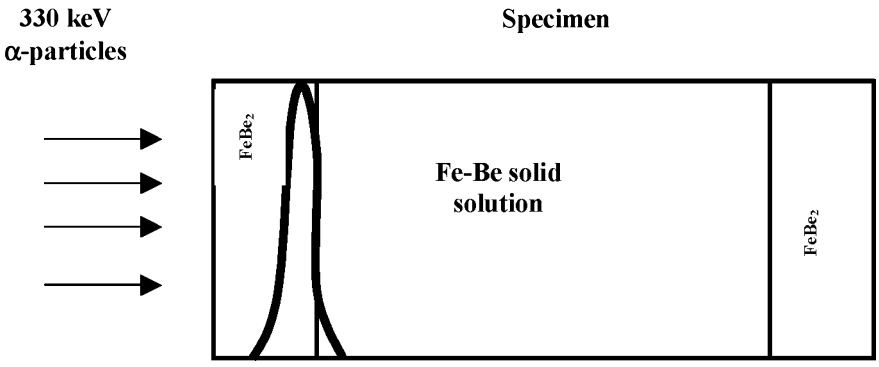


Figure 5. Scheme of specimen irradiation with the Wezuvii accelerator.

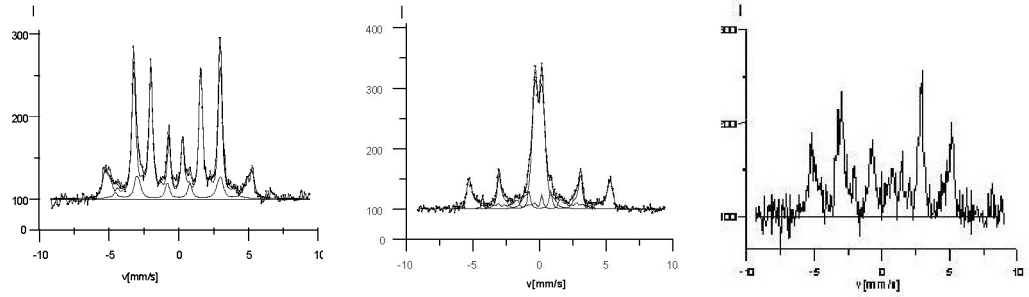
Table 1. Projected range of alpha particles in Fe-Be target

Ion = He (Mass = 4) Target = Fe(34 %) + Be(64 %) Density = 3.9244E+00 g/cm3 Stopping Units = keV / micron					
Ion Energy	dE/dx Elec	dE/dx Nuclear	Projected Range	Longitudinal Straggling	Lateral Straggling
330.00 keV	4.115E+02	1.571E+00	1.09 μm	1191 Å	2126 Å

It is necessary to note that the total irradiation dose was insufficient to explore the formation of gas porosity in the samples. This problem will be the subject of future investigations. The present concern is only the structural changes induced by the alpha particle irradiation. The structure of an irradiated sample, both before and after annealing, was studied by CEMS, X-ray, RBS, OM and SEM techniques.

OM and SEM investigations of the structure of the irradiated surface show that the irradiation up to the dosage used does not induce noticeable changes in the surface texture. CEMS and X-ray investigations shows there are short-range disturbances in the degree order in the $FeBe_2$ phase; see for example Fig. 6.

Figure 6 shows the SEM spectra of non-irradiated (a), irradiated (b), irradiated and annealed specimens (c). From a comparison of the spectra in Fig. 6a and 6b, one can see the disturbance of the short-range order degree or partial amorphization of $FeBe_2$ phase structure. Consequent annealing at 700 °C (Fig. 6c) results in the recovery of the phase structure. Investigation using X-ray diffractometry confirm results of the SEMS study. RBS measurements of the depth distribution of Be and Fe near the irradiated surface shows that there are no visible changes of beryllium and iron content in that region (Fig. 4). It is, therefore, possible to conclude that irradiation in small doses does not disturb the phase structure of $FeBe_2$, and migration of beryllium into the matrix of the sample is not observed.



a) Conversion electrons Mossbauer spectra (side A) before irradiation

b) Conversion electrons Mossbauer spectra (side A) after irradiation

c) Conversion electrons Mossbauer spectra (side A) of irradiated sample after 5 hours annealing

Figure 6. SEM spectra's of non irradiated (a), irradiated (b), and irradiated and annealed specimen (c).

CONCLUSION

A method, supported by the thermodynamic approach, is suggested for the creation of protective coatings stable at high temperatures. With the method, we have produced a beryllium coating on thin iron foil stable at temperatures up to 750 °C, by means of magnetron sputtering method.

Samples were further subjected to alpha particle irradiation at room temperature. Doses up to $3 \cdot 10^{16}$ particles/cm^2 did not result in the destruction of the beryllium coating.

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