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X-RAY diffraction and mössbauer studies of fe-57 implantation into the metallic Ta AND Mo

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Abstract. Development of nuclear and thermonuclear energy poses a number of problems for materials scientists related to the creation of structural materials. It should be noted that to date, the material of the fuel rod ducts, which would be able to realize the economical, long-term and safe operation of FNR in full scope, has not been developed. The study of the properties of structural materials for operating and future nuclear plants is still one of the most important scientific and technical challenges. The methods of X-ray diffraction and Mössbauer spectroscopy on ^{57}Fe nucleus were used to study the effect of implanting ^{57}Fe ions of 1 MeV energy and the fluence of $5*10^{16}$ion/cm^2 on the radiation resistance properties of the structural materials of the nuclear industry of metallic Ta and Mo. Mössbauer studies were of two methods: 1) standard transmission geometry (MS) and 2) conversion electrons registration from the material surface (KEMC). Concentration of the implanted Fe atoms was calculated using STRIM software. Two phases formation was found at the result of implantation into Ta and Mo matrices. The main phase in molybdenum (84%) is a solid solution of Fe replacement into Mo. The main phase in tantalum (78%) corresponds to formation of Fe complexes in the matrix of Ta. The obtained results of the study could be used to solve the issues of NPPs safe operation and to improve the efficiency of their operation, enabling the resource characteristics of core materials to be correctly assessed and to predict their behavior at high damaging radiation doses.
Key words: implantation, conversion and absorption Mössbauer spectroscopy, X-ray diffraction, STRIM software, radiation damage.

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Introduction

The development of nuclear and thermonuclear energy makes the material scientists to create the new structural materials that are resistant to various types of radiation. The study of radiation resistance under reactor irradiation conditions is easy to implement through the implantation of atoms on charged particle accelerators.

The alloying of metals upon irradiation with ions with energy of more than 10 keV has a number of special features that distinguish them from alloys synthesized by the metallurgical method.

To begin with, it leads to the formation of atomic mixtures in the surface area of the material, the composition of which is not limited by the principles of thermodynamics.

Secondly, the concentration profile of embedded atoms along the target depth with increasing ion fluence can vary from the portion of a percent to tens of percent. The limiting impurity content in the doped layer is determined by the sputtering coefficient, to a large extent and sometimes by diffusion processes.

Thirdly, a large number of displaced atoms (structural defects) are generated along with the ion-beam doping, which strongly affect the location of the introduced atoms in the crystal lattice and can cause the movement of atoms both at small and long distances.

Fourthly, since ion implantation is an athermal process, thermally activated phenomena can be independently controlled by the target temperature.

This allows (with a high degree of accounting and reproducing rate) to create alloys with the very

different properties in the near-surface area of the implanted material with a length of 0.01 to 1 өm. By changing the type and dose of ions, the target temperature, or the rate of dose collection, it is possible to obtain equilibrium alloys, supersaturated solid solutions, metastable, intermediate phases or amorphous materials [1].

The Mцssbauer effect makes it possible to measure hyperfine electric and magnetic fields on 57Fe nucleus, which are determined by the local atomic environment. With this in mind, the phases in which iron atoms are localized can be determined in a Mцssbauer experiment.

A large number of monographs and original articles have been devoted to the study of the physical and chemical state of matter using Mцssbauer spectroscopy with all the variety of its methodological approaches [2,3]. In [4], the grain boundaries of polycrystalline molybdenum were studied by Mцssbauer emission spectroscopy on ^{57}Co(^{57}Fe) nucleus. The Co atoms have been demonstrated to diffuse along grain boundaries by the interstitial mechanism. The authors show that the effective diffusion coefficient in the boundary regions of crystallites is significantly higher than the volume diffusion coefficient, but the width of the zone of accelerated diffusion does not exceed several atomic layers. Based on the temperature dependences analysis of the isomeric shift of the spectral lines, the grain boundaries and the boundary regions of crystallites are concluded to be enriched with interstitial impurities. Moreover, the degree of this enrichment is greater, the lower the annealing temperature.

This paper provides the samples of metallic tantalum and molybdenum irradiated with ^{57}Fe ions were studied by X-ray diffraction, Mцssbauer spectroscopy, and electron microscopy. These two materials are structural materials that are used in the nuclear energy. Irradiation with ^{57}Fe ions makes it possible to conduct Mцssbauer studies on the atoms that directly create radiation damage. High doses of radiation were applied in the work, creating damage in the surface layer corresponding to approximately 200 displacements per atom. In simular conditions, iron atoms do not dissolve in metallic Ta [5] and to a limited extent dissolve in metallic Mo [6]. The study was aimed to determine which phases or local structures of Fe atoms form in Mo and Ta as a result of powerful irradiation and subsequent radiation annealing.

The two methods for Mцssbauer studies were used: 1) in standard transmission geometry (MS) and 2) with registration of conversion electrons from the material surface (KEMC). In the first case, the Mцssbauer spectra contained information about all implanted Fe-57 atoms. As concerns KEMS, only Fe atoms were recorded that were in the surface irradiated layer with a thickness of about 0.1 өm. The thickness was determined by the yield of ^{57}Fe conversion electrons with energies of 7.3 - 13.6 keV.

Testing

The studied samples were Ta and Mo foils with a thickness of 20 өm. The thickness of the studied foils was selected so as to conduct Mцssbauer measurements in transmission mode (MS). Foils were obtained by repeated cold rolling of the original bulk samples. Recrystallization annealing for the foils was carried out in vacuum $5\cdot10^{-6}$mm of mercury for 2 hours at a temperature of 1000 °C. The purity of the initial Ta and Mo was no worse than 99.9.

Irradiation with ^{57}Fe ions with an energy of 1 MeV was carried out using a UKP-2-1 heavy-ion accelerator at the Institute of Nuclear Physics (Almaty). A special holder made of aluminum was designed and manufactured to carry out irradiation experiments, an appropriate sample of metallic iron was placed inside enriched up to 95% with ^{57}Fe isotope, for its further ionization in a source of heavy ions and subsequent acceleration. The ion current density was maintained in the range of 50 - 100 nA. The ion flux fluence for all samples was $5Ч10^{16}$ ion / cm^2.

X-ray diffraction analysis was carried out using BRUKER D8 ADVANCE diffractometer. The diffraction patterns were measured both from the side of the irradiated surface and from the reverse side in the range of angles 2θ = (20 ч90).

Mцssbauer studies were carried out in two modes: 1) in the standard transmission geometry (MS) and 2) with registration of conversion electrons from the irradiated surface of the material (KEMC). The measurements were carried out on an MS-110Em spectrometer at room temperature. A ^{57}Co radioactive source in a chromium matrix served as the Mцssbauer source of gamma rays. The Mцssbauer spectra were analyzed and processed using the least square method using the SpectrRelax program [7].

The SRIM-2008 software [8] was used to assess the degree of influence of the ion beam on the crystal lattice of Mo and Ta. The results are provided in Table 1.

Table 1 SRIM calculation data

Element	Projective mileage R, nm	vacancies number / ion	Total number of displaced atoms	DPA (CHA)
Ta	272	7670	$3.8Ч10^{20}$	250
Mo	315	6580	$3.3Ч10^{20}$	165

Note that the average range of Fe ions (projective range, R) is approximately the same for all matrices, R ≈ 300 nm. The average number of displacements per atom (DPA) for two matrices is approximately 200.

Figure 1a provides the dependence of the concentration of Fe,*n*(Fe) atoms implanted in a tantalum foil on the *x* distance to the irradiated surface (curve 1, concentration is expressed in at.%).

The calculations were performed taking into account the diffusion of the matrix atoms. The calculated Ta diffusion coefficient turned out to be small and equal to 3.09 Ta atoms / ion. Accordingly, a fluence of $5*10^{16}$at / cm^2 causes a sputtering of a Ta layer with a thickness of 28 nm, which is

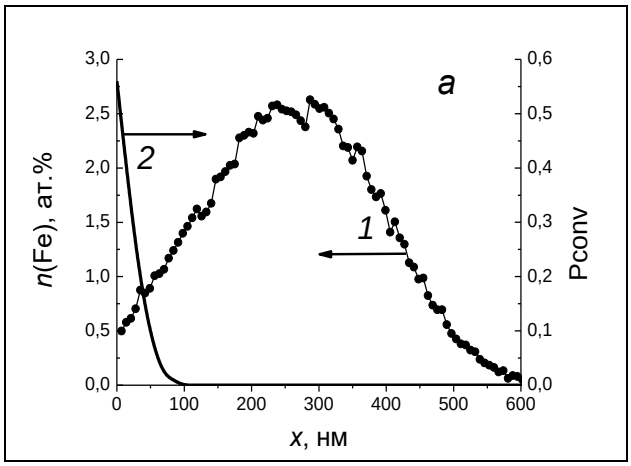

Fig. 1. a Concentration of implanted atoms Fe, n (Fe), in the Ta matrix from the x depth (curve 1); curve 2 – output probability of conversion electrons P_{conv} from x depth (right axis Y)

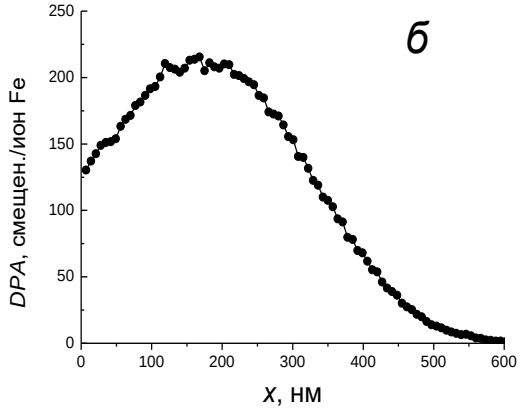

Fig. 1. b DPA parameter - the number of displacements per ion, as a function of x depth

noticeably less than the thickness of the implanted layer. Similar concentration curves were obtained for Mo. The maximum concentration of ^{57}Fe implanted atoms in Ta and Mo is approximately 2.6 at.%.

Figure 1b provides the dependence of the DPA parameter in tantalum on the distance to the irradiated surface, DPA (x). This parameter, equal to the average number of displaced matrix atoms per ion, represents the degree of matrix destruction upon irradiation. STRIM calculations specify only the initial conditions for the problem of material transformation under radiation exposure. The final phases and structure of the material is determined by radiation annealing, which occurs during irradiation.

Mцssbauer spectra of ^{57}Fe in Mo and Ta

Figures 2a and 2b provide the Mцssbauer spectra of ^{57}Fe implanted in Ta. Figure 2a provides the conversion electron spectrum (KEMS). The spectrum corresponds to ^{57}Fe atoms located in the surface layer at a depth of ≈ 100 nm. The probability curve for the exit of conversion electrons from Ta is shown in Fig. 1 a, curve 2 [9, 10].

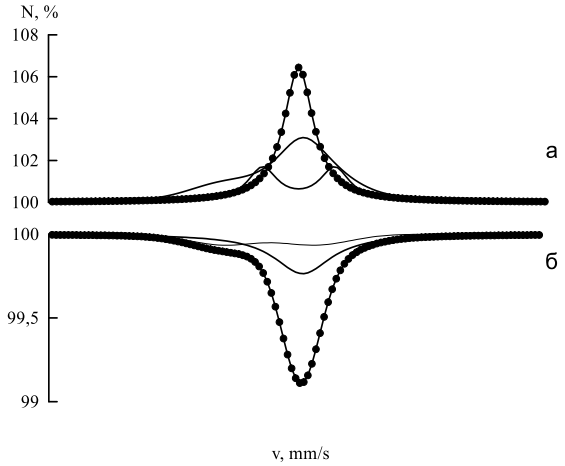

Fig. 2. Mцssbauer spectra of ^{57}Fe implanted in the Ta matrix. a - conversion electron spectrum (KEMS); b - transmission spectrum (MS).

The KEMS spectrum consists of a broadened singlet and a strongly broadened doublet; the intensities of the sub-spectra are 70% and 30%, respectively. Presumably, the doublet corresponds to a strong distortion of the crystal lattice, and the singlet to a less distorted phase.

The Mцssbauer transmission spectrum in Fig. 2b also consists of a singlet and a doublet. A consistent analysis of the conversion electron spectra (KEMS) and transmission spectra (MS) was carried out in the work. When processing transmission spectra (MS), it was taken into account that they should contain a KEMS spectrum, as well as an additional contribution from deeply located Fe atoms. With this in mind, the transmission spectrum processing model contained a KEMS spectrum in which all parameters except the total intensity were recorded, plus an additional singlet spectrum. The parameters of this singlet within the errors coincided with the KEMS singlet.

Figures 3a and 3b provide the Мцssbauer spectra of ^{57}Fe implanted in Mo: the conversion electron spectrum of KEMS is 3a and the transmission spectrum of MS is 3b. The spectra of Mo are similar to those of Ta, and contain singlet and doublet contributions.

The KEMS spectra for both Ta and Mo show that both “doublet” and “single” phases are present near the surface ($x \approx 100$ nm). At large depths, a “singlet” phase is predominantly formed. This phase distribution is especially characteristic of Mo, for

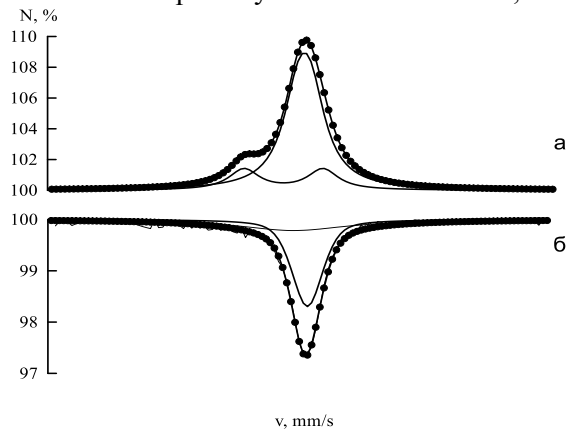

Fig. 3. Мцssbauer spectra of ^{57}Fe implanted in a Mo matrix. a - conversion electron spectrum (KEMS); b - transmission spectrum (MS).

which a single line in the transmission spectra dominates and amounts to 93%. This line in the spectrum of Mo has a width of G_s = 0.27mm / s, which only slightly exceeds the natural width of the Мцssbauer line for ^{57}Fe [11]. This means that the monopic in Mo corresponds to Fe atoms, which replace Mo atoms in the crystal lattice. In other words, even with strong radiation exposure (*DPA* = 200), a solid solution of substitution of Fe in Mo is mainly formed in the Mo matrix.

The KEMS spectrum of the “singlet” phase Ta is greatly broadened, and in the case of the MS spectrum it is described by a small quadrupole splitting QS = 0.17 mm / s. It is known that Fe atoms do not dissolve in Ta at room temperature under equilibrium conditions ~~[12].~~ With this in mind, we can assume that the “singlet” phase is the formation of Fe + Ta + vacancy complexes in the Ta matrix. These assumptions do not contradict the results of [12], in which the Мцssbauer isotope ^{57}Co was introduced into the samples of high purity molybdenum (99.999%) to an average concentration of 0.001 at.%. The diluted MoCo alloy thus obtained was irradiated with protons with an energy of 70 keV to a fluence of $2*10^{18}$ cm^{-2} at a temperature of 300 K, and then isochoric annealing was carried out in the temperature range 300ч1300 K, followed by measurement of γ-resonance spectra. In addition to the single line corresponding to the position of Co in the position of substitution, six additional components were found, five of which (2-6) are symmetrical doublets with the initial width, and component 1 is a single broadened line. Component 1 is attributed to the complexes: (intrinsic interstitial atom) - (Co atom). Components 2–4, which appear after annealing in the temperature range 400ч500 K, bind to complexes (vacancy) — (Co atom), and components 5 and 6 — with nonequilibrium segregation of Co atoms.

The issue of identifying the “doublet” phases for Ta and Mo remains open. Мцssbauer data do not provide clear guidance. We list the possible phases: these are the phases of the Fe-Ta and Fe-Mo diagram, as well as the β-Ta phase. The initiation of cluster formations of Fe + Ta or Mo + vacancies or partial amorphization cannot also be ruled out.

Discussion of the results

The least irradiation effect is observed for metallic Mo. The “singlet” phase in Mo, constituting 84% of the surface layer, is a solid solution of substitution of Fe in Mo, which is confirmed by X-ray diffraction data, which showed a decrease in the lattice parameter from 3.15980 E for the unirradiated side to 3.14194 E for the irradiated one. In the case of tantalum, the “singlet” phase with an intensity of 78% apparently corresponds to the formation of complexes (Fe + vacancy) in the Ta matrix. Such complexes can serve as a source of additional stresses.

As for the "doublet" phases found both in tantalum and in molybdenum, the question remains open. X-ray diffraction performed by the Bragg-Brentano method did not detect additional phases. In particular, the β-Ta phase [13, 14] and phases of the Fe-Ta system: Fe-Ta: Fe_2Ta, Fe_7Ta_6 and Fe_2Ta_3 [15] were not found for the tantalum matrix. For the molybdenum matrix, no traces of the phases of the Fe – Mo system were detected: Fe-Mo: Fe_2Mo, Fe_7Mo_6 and Fe_2Mo_3 [16]. The diffraction patterns measured from the irradiated and back sides practically coincided. For the Ta matrix, a slight broadening of the lines was observed in the diffractogram of the irradiated side; for the Mo matrix, there was practically no additional line broadening. The broadening of the diffraction lines indicates disturbances in the crystal lattice of the surface layer. The “doublet” phases, apparently, refer to the formation of Fe + Ta complexes or Mo + vacancy in the regions of grain boundaries, or are associated with regions of partial amorphization of materials.

Based on the Мцssbauer measurements and the dependence of the n (Fe) concentration on the depth

(Fig. 1a), the localization of phases in Ta and in Mo can be estimated. In the Ta matrix, a “doublet” phase and a “singlet” phase of substitution of Fe in Ta coexist in a 350 nm thick near surface layer; in the layer from 350 to 600 nm, only the “singlet” substitution phase is localized. In the Mo matrix, the “doublet” phase and the “single” monopick of Fe substitution in Mo are localized in the surface layer with a thickness of 250 nm; next is the "singlet" phase of the solid solution of substitution of Fe in Mo.

Conclusions

The effect of the implantation of ^{57}Fe ions with an energy of 1 MeV and a fluence of $5*10^{16}$ion/cm^2 on the properties of structural materials of the nuclear industry[17], Ta, Mo was studied. Implantation took place in a surface layer with a thickness of about 600 nm and corresponded to approximately 200 displacements of the matrix atoms per ion ^{57}Fe.

For metallic Ta and Mo, Mцssbauer spectroscopy also showed the formation of two phases. The main "single" line in the spectrum of molybdenum (84%) is a solid solution of Fe in Mo substitution. The monopik in the tantalum spectrum (78%), taking into account the zero solubility of Fe in Ta, most likely corresponds to the formation of a complex (Fe + vacancy) in the Ta matrix. The nature of the surface “doublet” phases has not been completely established.

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Металл Та мен Мо-де Fe-57 имплантациясын рентгенқұрылымдық және мессбауэрлік зерттеулер

Бедельбекова К. А.

Түйіндеме. Атомдық және термоядролық энергетиканың дамуы материалтанушыларға конструкциялық материалдарды жасаумен байланысты бірқатар мәселелер қойып отыр. Қазіргі таңда ШНР үнемді, ұзақ мерзімді және қауіпсіз жұмысын толықтай жүзеге асыруға мүмкіндік беретін жбэлтері қапшықтарының материалын әзірлеу іске аспағанын атап өткен жөн. Жұмыс істеп тұрған және перспективалық ядролық қондырғыларға арналған конструкциялық материалдардың қасиеттерін зерделеу әлі де маңызды ғылыми-техникалық міндеттердің бірі болып табылады. Рентгендік дифракция және мессбауэрлік спектроскопия әдістерімен ^{57}Fe ядроларында атом өнеркәсібінің металл Та мен Мо конструкциялық материалдарының радиациялық тұрақтылық қасиеттеріне 1 МэВ энергиялы және $5*10^{16}$ion/cm^2 флюенсті ^{57}Fe иондары имплантациясының әсері зерттелді. STRIM программасының көмегімен ендірілген Fe атомдарының концентрациясы есептелді. Матрицаларға Та және Мо ендіру нәтижесінде екі фазаның түзілуі айқындалды. Молибдендегі (84%) негізгі фаза Мо-де Fe ауыстыратын қатты ерітінді болып табылады. Танталда (78%) негізгі фаза Та матрицасында Fe жиынтықтарының түзілуіне жауап береді. Алынған зерттеулер нәтижелері ядролық қондырғыларды қауіпсіз пайдалану мәселелерін шешуге және олардың жұмысының тиімділігін арттыруға пайдаланылуы мүмкін, бұл активті аймақтың материалдарының ресурстық сипаттамаларын дұрыс бағалауға және жоғары зақымдаушы дозаларда сәулелендіру кезінде олардың әрекетін болжауға мүмкіндік береді.

Түйін сөздер: Имплантация, конверсиялық және абсорбциялық мессбауэрлік спектроскопия, рентгендік дифракция, STRIM программасы, радиациялық зақымдану.

Рентгеноструктурные и мессбауэровские исследования имплантации Fe-57 в металлический Ta и Mo

Бедельбекова К. А.

Аннотация. Развитие атомной и термоядерной энергетики ставит перед материаловедами ряд проблем, связанных с созданием конструкционных материалов. Следует отметить, что на сегодняшний день материала чехлов твэлов, который позволил бы в полной мере реализовать экономичную, длительную и безопасную работу РБН, разработать не удалось. Изучение свойств конструкционных материалов для эксплуатируемых и перспективных ядерных установок по-прежнему остается одной из важнейших научно-технических задач. Методами рентгеновской дифракции и мессбауэровской спектроскопии на ядрах ^{57}Fe исследовано влияние имплантации ионов ^{57}Fe с энергией 1 МэВ и флюенсом $5*10^{16}$ion/cm^2, на свойства радиационной устойчивости конструкционных материалов атомной промышленности металлических Ta и Mo. Мессбауэровские исследования проводились по двум методикам: 1) в стандартной геометрии на пропускание (МС) и 2) с регистрацией электронов конверсии из поверхности материала (КЭМС). С помощью программы SRIM была рассчитана концентрация имплантированных атомов Fe. В результате имплантации в матрицы Ta и Mo обнаружено образование двух фаз. Основная фаза в молибдене (84%) представляет собой твердый раствор замещения Fe в Mo. Основная фаза в тантале (78%) отвечает образованию комплексов Fe в матрице Ta. Полученные результаты исследования могли бы применяться для решения проблем безопасной эксплуатации ядерных установок и повышения эффективности их функционирования, позволив правильно оценивать ресурсные характеристики материалов активной зоны и предсказывать их поведение при высоких повреждающих дозах облучения.

Ключевые слова: Имплантация, конверсионная и абсорбционная мессбауэровская спектроскопия, рентгеновская дифракция, программа STRIM, радиационные повреждения.

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