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EFFECT OF IRON ON EVOLUTION OF THE STRUCTURE OF ALLOY Zr-1%Nb UNDER ION IRRADIATION

M.M. Pylypenko[1,2], R.L. Vasilenko[1], A.O. Drobyshevska[1]
[1]National Science Center "Kharkov Institute of Physics and Technology", Kharkiv, Ukraine;
[2]V.N. Karazin Kharkiv National University, Kharkiv, Ukraine
E-mail: mpylypenko@kipt.kharkov.ua

This paper presents experimental data of the study of the influence of Fe alloying on the changes of the structure of the Zr-1%Nb alloy and evolution of the structure of this alloy under ion irradiation. It is shown that small additions of Fe to the Zr-1%Nb alloy lead to the change its structure due to the formation of Laves phase precipitates. Influence of ions Ar^{2+} irradiation on parameters of dislocation loops in alloy Zr1%Nb with Fe content 0.012…0.192 wt.% is studied by methods of transmission electron microscopy. Irradiations was carried out by Ar^{2+} ions (accelerator ESU-2) with energy 1.4 MeV, at temperature T = 390 °C. Dependence of density and size of dislocations on Fe content and on irradiation dose are obtained.

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INTRODUCTION

Zirconium (Zr) alloys have widely been used as the fuel cladding materials in water-cooled nuclear reactors of both pressurized water reactor and boiling water reactors over the past several decades. To achieve better economic and safety performance, higher burn-up fuel designs are required by the industry [1], and hence the improvement of existing zirconium alloys and the design of news one with longer operating lifetimes in the core.

The Zr-1%Nb alloys are the materials for cladding and structures and used in pressurized water reactors WWERs and provide reliable operation of fuel elements and fuel assembles in existing reactors and serves as a basis for new modifications. The high corrosion and radiation resistance of zirconium elements is provided by the chemical composition, structure, and phase composition of the alloys. Numerous works [1–3] have shown that Fe alloying of zirconium is promising in developing advancing alloys. The increase of iron (Fe) content in the zirconium alloy provides the material of the cladding tubes with the required resistance to creep and strengthening under irradiation. In addition, the Fe alloying of the Zr-1%Nb alloy increases its corrosion and radiation resistance in the conditions of the operation of a nuclear reactor.

At the same time it was found that as a result of additional Fe alloying the technological efficiency of the Zr-1%Nb alloy decreases, which requires the development of a new deformation-thermal scheme for cladding tube production. Therefore, determination of the optimal Fe content in the Zr-1%Nb alloy is a prerequisite for providing the technological efficiency of the alloy and improving its performance properties. Previously, long-term corrosion tests of samples of the Zr-1%Nb alloy with different Fe content in water of a chemical composition similar to the primary coolant in reactor WWER-1000 (temperature 350 °C, pressure 16.5 MPa) were carried out [4]. Research has detected that the optimal Fe content in the Zr-1%Nb alloy is 0.10 wt.%; the alloy with this Fe content has the highest corrosion resistance under operating conditions of the WWER-1000 core. Increasing or decreasing of the Fe content in the Zr-1%Nb alloy leads to an increase its corrosion rate. This Fe content too provides acceptable values of microhardness of the alloy [4].

The irradiation induced growth (IIG) phenomenon is a significant research area for zirconium alloys, which are used as cladding and structural materials in fuel assemblies in reactor-cores [5–7]. IIG refers to the volume-conservative shape change, which occurs during neutron irradiation; the cladding expands in the axial direction and shrinks in the radial direction [8]. This growth is one of the main limiting factors in the lifespan of fuel assemblies.

IIG is well correlated to the evolution of dislocation loops in the material. At low fluences, irradiation-induced point defects collapse into vacancy and interstitial a-type dislocation loops on mostly first order prismatic planes with a Burgers vector of $b = \frac{1}{3}\langle 11\bar{2}0\rangle$ [9]. Upon further irradiation, c-component vacancy dislocation loops form on the basal plane, which have a Burgers vector of $b = \frac{1}{6}\langle 20\bar{2}3\rangle$ [10].

Alloy chemistry can significantly alter the observed irradiation induced growth and particularly the point at which breakaway growth occurs. That's why, the role of Fe has been investigated with particular interest, as Fe dissolves from pre-existing second phase particles (SPPs) during irradiation and is a fast diffusing element in Zr, potentially sitting interstitially within the Zr lattice [11, 12] and interacting with point defects generated during irradiation [13].

Previous research has suggested that an increased interstitial solute content in the Zr-matrix correlates to the formation of loops [10, 12]. However, Fe has been found to form clusters or precipitates and segregates to and loops [12, 14, 15]. As loops are frequently found near Fe-containing SPPs, Fe has been suspected to intensify the breakaway growth phenomenon. More recently, however, increasing the Fe content in Zr-Nb-Sn-Fe alloys has been correlated with improved resistance to irradiation growth [11].

Niobium (Nb) is important alloying element in some Zr-alloys, which to improve the performance of zirconium alloys regarding corrosion and dimensional stability [16–18]. Delayed c-loop formation has been reported for Nb-containing alloys such as E635 (Sn 1.1…1.4%, Nb 0.9…1.2%, and Fe 0.3…0.47%) and M5 (Nb 0.8…1.2%). According to equilibrium phase diagram of Zr-Nb alloy, the thermally stable status of a binary zirconium alloy with a few percent of Nb is the solid solution of Nb in the α-Zr matrix when the temperature is below the monotectoid reaction temperature [19]. Niobium will precipitate as a β phase in the α-Zr grain matrix.

In Zr-Sn, Zr-Nb, and Zr-Sn-Nb alloys the addition of more Fe led to a decrease in growth strain [20]. Shishov et al. also showed data from the BOR-60 test reactor, which indicated that increased Fe leads to a lower IIG strain in variants of the E635 Nb containing alloy [11].

While most studies have focused on understanding the role of Fe on c-loop formation little work has focused on the effect that Fe might have on a-loop formation. A detailed understanding of the early formation and evolution of a-loops with increasing dose is vital if one considers that c-loop formation is related to a-loop evolution. For instance, it has been proposed that vacancy a-loops are involved in the mechanism for c-loop formation [9, 12], supported by an observed anti-correlation for a-loops and c-loops seen by Topping et al. [14] and the decrease in a-loop density after c-loop nucleation [12].

The main source for alloying elements to be redistributed during irradiation is the SPPs as they slowly dissolve by leaching out either all or some of their elements into the matrix.

The incubation period of the accelerated growth stage is determined by the α-matrix composition, the phase state and the initial dislocation structure. Neutron irradiation leads to a redistribution of alloying elements between the matrix and the precipitates, and to changes in the α-solid solution composition. Interestingly, the addition of 0.04…0.10 wt.% Fe and ≤0.15 wt.% O to E110 (Zr-1%Nb) causes the onset of breakaway growth to delay to a higher dose of > ~15 displacements per atom (dpa) [11].

The aim of this work is to study influence of Fe micro alloying on structure and the radiation properties of the Zr-1%Nb alloy.

MATERIAL AND EXPERIMENTAL PROCEDURES

The Zr-1%Nb alloy based on magnesium-thermal zirconium was used for obtaining experimental samples. Optimization processes of melting of the Zr-1%Nb alloy based on magnesium-thermal zirconium by methods of electron beam melting in laboratory conditions are described in detail in [21].

The vacuum-arc melting method was applied to obtain a homogeneous zirconium alloys with Fe micro-additives. A pure Fe after refining by electron beam melting was used for alloying. The obtained samples were contained Fe from 0.012 to 0.192 wt.% with an interval of 0.03 wt.%. The oxygen content in all samples of Zr-1%Nb alloy was 0.1 wt.%. The Zr-1%Nb alloy samples in the form of 1 mm thick plates were prepared by rolling in a vacuum with intermediate annealing. After rolling all samples were annealed in the vacuum of 10^{-5} mm Hg at the temperature of 580 °C for 3 h.

Investigations of surface microstructure were performed using scanning electron microscope (SEM) JSM-7001F equipped with the system for energy-dispersive X-ray spectroscopy INCA Energy 350. Microhardness was measured by the device PMT-3.

Samples of the Zr-1%Nb alloy for transmission electron microscopy (TEM) studies were prepared as disks of 3 mm in diameter. Thin foils were obtained by mechanical thinning of the disks down to 130 μm followed by electropolishing and short-term annealing.

Irradiation experiments were conducted in the accelerating-measuring system ESU-2 [22]. The samples were irradiated with a 1.4 MeV Ar^{2+} ion beam at irradiation temperature of 390 °C and an irradiation doses 5 and 15 dpa. The depth distribution profiles of damage and concentration of Ar atoms implanted in Zr-1%Nb alloy irradiated by 1.4 MeV Ar^{2+} ions were calculated by SRIM 2003. Based on these profiles, the irradiation parameters were calculated, and the study area was selected, in which, on the one hand, the damaging dose is high, and, on the other hand, the number of implanted atoms is small. The area that meets the above requirements is located at a depth of 0.1...0.15 μm from the sample surface.

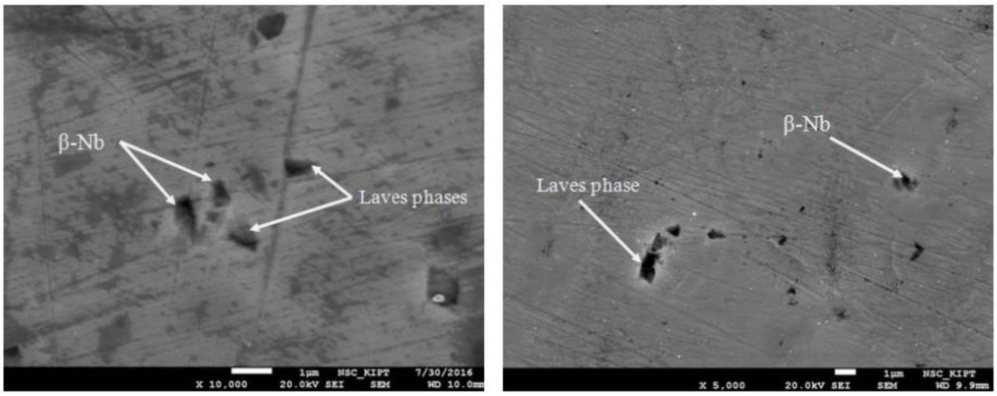
Samples were investigated in JEM-2010 microscope. X-ray energy dispersive spectroscopy (EDS) equipped with TEM was used to identify the chemical composition of the precipitates. Microstructural and precipitates parameter data were extracted using conventional techniques conducted on JEM-2100 TEM, employing standard bright-field techniques.

EXPERIMENTAL RESULTS AND DISCUSSION

The properties of alloys are determined by their structural-phase state and even small changes in the composition of Zr-Nb alloys lead to significant changes due to the formation of different types of precipitates and changes in the matrix composition [23, 24]. The kinetics of release of new phases in α-Zr is determined by the composition of the alloy, the degree of supersaturation of the solid solution α-Zr, the composition and the crystal structure of the phases. The alloying elements have a low solubility in α-Zr (0.005...0.02% Fe, ~ 0.5% Nb) and are precipitated as SPPs with sizes of 50...500 nm. The composition and type of precipitates are determined by the degree of solubility of the main alloying elements (Nb and Fe) in α-Zr and their total content in the alloy [25].

Metallographic studies of the structure of samples of the Zr-1%Nb alloy with different Fe content showed that the samples have a two-phase structure: α-matrix and precipitates of the second phase. The additional Fe alloying of the Zr-1%Nb alloy leads to the formation of precipitates the density of which increases with increasing Fe content. Sizes and morphology of SPPs are close and to distinguish there are difficult. The study of the chemical composition of particles using the X-ray spectral microanalysis confirmed the presence of two types of precipitates. The Fe content in the matrix is 0...0.1%, Nb – 0.3...0.7%.

The microstructures of the Zr-1%Nb alloy samples depending on the composition are presented in Fig. 1. The fine particles of β-Nb precipitate and a small number of larger precipitates – the Laves phases Zr(Nb, $Fe)_2$ are present in structure of alloy samples with the Fe content of up to 0.042 wt.%. The concentration of Laves phases is much lower than the concentration of β-Nb particles. The increasing the Fe content in the alloy from 0.042 to 0.072 wt.% lead to the insignificant increase in the concentration of Laves phase precipitates. A significant increase in the number of precipitates and their sizes occurs when Fe alloying up to 0.162 wt.% and more. The average size of the β-Nb precipitates is 40...50 nm. The precipitates of Laves phase are slightly larger, their average size is 80...100 nm [26].



a *b*

Fig. 1. The microstructure of the samples of the alloy Zr-1%Nb with Fe content 0.072 (a) and 0.162 wt.% (b)

Studies show that the number of Laves phase precipitates significantly depends on the Fe content in the alloy. Due to the low solubility of Fe in α-Zr, almost all of it is concentrated in the Laves phases.

The microhardness values of Zr-1%Nb alloys samples were increased with increasing the Fe concentration in alloys: the microhardness value for the initial samples (0.012 wt.%) are 1720 MPa, the samples with 0.192 wt.% Fe – 1880 MPa. In the the area near the concentration of 0.1 wt.% Fe, the microhardness value of the alloys are 1750 MPa [26].

The effect of irradiation with Ar^{2+} ions on the parameters of dislocation loops in Zr-1%Nb alloy with different Fe content was investigated by methods of transmission electron microscopy.

Studies have shown that dislocation loops under these conditions have an elliptical shape. In this case, starting from dose of 5 dpa, the loops begin to interact with each other, forming elements of the dislocation network. Up to a dose of 15 dpa, only a-type loops are observed, c-type loops are not detected. Irradiation of an alloy with Fe additions leads to the formation of radiation-induced a-type loops and the formation of a dislocation network due to the interaction of the loops at a dose of 15 dpa.

Fig. 2 presents the structure of alloys Zr-1% Nb, which contain 0.012 and 0.102 wt.% Fe after irradiation. Data on the dependence of the size of the loops on the radiation dose are given in the Table, which shows that the size of the loops and the loop density increases with increased the dose of damage.

Data on the dependence of the loops sizes on the Fe content and radiation dose are shown in Fig. 3. It is seen that the size of the loops decreases with increasing concentration of Fe in the alloy; the size of the loops increases with the dose of damage, and with increasing dose the growth rate of the loops decreases (tendency to saturation). This is probably due to the fact that large loops are involved in the formation of a dislocation network, and newly emerging dislocation loops make a significant contribution to the decrease in the velocity of average radius of loop with increasing dose.

Parameters of radiation-induced dislocation loops
in Zr-1%Nb alloys with different Fe contents

Fe content, wt.%	0.012		0.102		0.192	
Dose, dpa	5	15	5	15	5	15
Loop size, nm	22.2	28.1	19.2	26.8	17.6	25.2
Loop density, m^{-3}	$1.6\cdot10^{21}$	$2.1\cdot10^{21}$	$1.8\cdot10^{21}$	$2.2\cdot10^{21}$	$1.9\cdot10^{21}$	$2.3\cdot10^{21}$

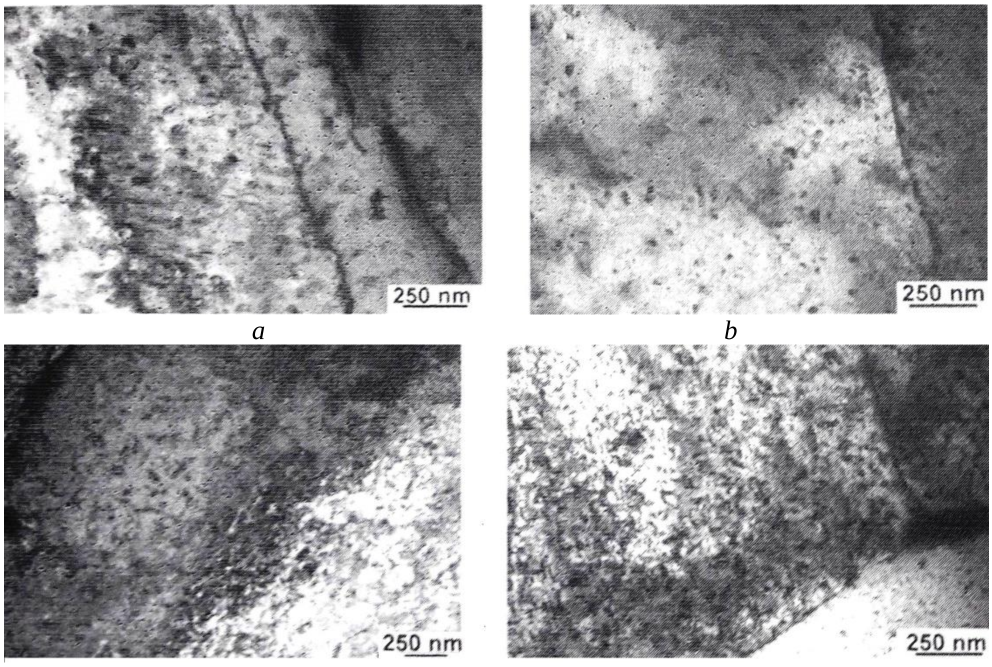


Fig. 2. Microstructure of the Zr-1%Nb alloy with Fe content 0.012 (a, b) and 0.102 wt.% (c, d) irradiated to dose of 5 (a, c) and 15 dpa (b, d)

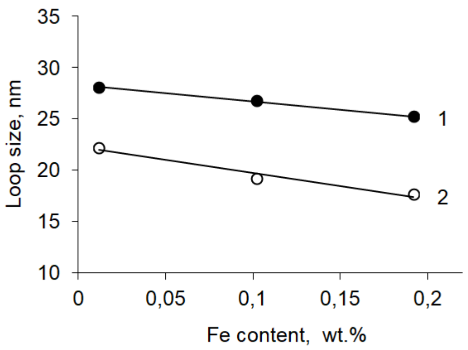


Fig. 3. Dependence of the average loop size on the Fe concentration in the Zr-1%Nb alloy at irradiation doses of 15 (1) and 5 dpa (2)

Studies of the microstructure evolution of Zr-1%Nb alloys with different Fe contents made it possible to reveal the effect of Fe on the processes of formation and development of radiation-induced interstitial dislocation loops. It can be seen from the data obtained that the increase in the Fe concentration leads to a decrease in the size of interstitial dislocation loops and an insignificant increase in their density.

X-ray microspectral studies of the composition of precipitates in samples of Zr-1%Nb alloys with Fe additions after irradiation to a dose of 15 dpa showed that, the precipitates of the Laves phase contain only 1…8% Fe; the content of Fe in the precipitates of the Laves phase was 30% before irradiation. This indicates the forced infiltration of Fe into the solid solution and its possible participation in the formation of secondary radiation-induced fine precipitates, which retard the formation of c-type dislocation loops responsible for accelerated the radiation growth of the zirconium alloy.

In accordance with the theory of anisotropic diffusion of point defects in alloys with an hcp structure [13], it can be assumed that the formation of a atom of Fe-vacancy complex leads to the formation of a rapidly migration conglomerate, which rather quickly annihilates with a vacancy zirconium atom. Accelerated annihilation of the vacancy-Fe-interstitial Zr atom complex compensates for the anisotropic diffusion phenomenon and inhibits radiation growth. Therefore, we can conclude that the Fe alloying of the zirconium-niobium system alloys contributes to the suppression of the phenomenon of radiation growth in commercially effective ranges of irradiation doses.

CONCLUSIONS

The evolution of the structure of the Zr-1%Nb alloy with increasing Fe content has been studied. It is shown that small Fe additions to the Zr-1%Nb alloy lead to a change its structure due to the formation of precipitates of second phase – Laves phases. It was found that the number of Laves phase precipitates is determined by the Fe content in the alloy and increases with increasing Fe content. The microhardness values of the alloy Zr-1%Nb increases with increasing Fe content in alloy.

Irradiation of the Zr-1%Nb alloy showed that increasing the Fe concentration leads to the decrease in the size of the interstitial dislocation loops and a slight increase in their density. Fe, leaving the Laves phase under irradiation into the matrix, forms secondary fine precipitates and delays the formation of c-type dislocation loops responsible for the acceleration of radiation growth. The Fe alloying of the alloys of the system Zr-Nb contributes to the suppression of the phenomenon of radiation growth in commercially effective ranges of radiation doses.

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ВПЛИВ ЗАЛІЗА НА ЕВОЛЮЦІЮ СТРУКТУРИ СПЛАВУ Zr-1%Nb ПРИ ІОННОМУ ОПРОМІНЕННІ

М.М. Пилипенко, Р.Л. Василенко, А.О. Дробишевська

Представлено експериментальні дані дослідження впливу легування залізом на зміни структури та еволюції структури сплаву Zr-1%Nb під час іонного опромінення. Показано, що невеликі додавання Fe до сплаву Zr-1%Nb призводять до зміни його структури за рахунок утворення виділень фази Лавеса. Методами просвічуючої електронної мікроскопії досліджено вплив опромінення іонами Ar^{2+} на параметри дислокаційних петель у сплаві Zr-1%Nb з вмістом заліза 0,012…0,192 мас.%. Опромінення проводили іонами Ar^{2+} (прискорювач ЕСУ-2) з енергією 1,4 МеВ при температурі T = 390 °C. Отримано залежності щільності й розміру дислокацій від вмісту заліза і дози опромінення.