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Mössbauer spectroscopy of zirconium alloys

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Abstract Mössbauer investigations of zirconium alloys were examined. Data about the chemical state of iron atoms in the zirconium alloys of different composition has been provided. Mössbauer spectroscopy revealed that small quantities of iron in binary zirconium alloy are in the solid solution α-Zr (up to 0.02 wt.%). Different iron atoms concentration and thermo-mechanical treatments may lead to formation the intermetallic compounds Zr_3Fe, Zr_2Fe, $ZrFe_2$. Adding tin atoms does not affect the formation and shape of Mössbauer spectra of these compounds. Adding Cr and Nb atoms makes significant changes in the shape of Mössbauer spectra and leads to the formation of complex intermetallic compounds. Adding Cu and W atoms, the shape of the binary alloys spectra (Zr-Fe) remains unchanged, but a change in the temperature dependence behavior of the spectral parameters occurs and also, changes to the properties of the alloys.

Keywords Mössbauer effect · Zirconium alloys · Zirconium intermetallic compounds

1 Introduction

Zirconium was discovered in 1895 in the mineral zircon. The element is used in alloys as a structural material in nuclear reactors. Alloys used in Russia, such as E110, E125, E635 have approximate compositions of Zr-1% Nb, Zr-2.5% Nb, Zr-1%

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Nb-0.35% Fe-1.2% Sn, respectively. Zircaloy-2 (Zr-1.2 ÷ 1. 7% Sn-0.07 ÷ 0.2% Fe-0.05 ÷ 0.15% Cr) (wt. %) and Zircaloy-4, M4, M5, NSF are commonly used alloys in the US and Europe. Fe, Sn, Nb, Cr, Cu, Ni are often added to improve mechanical properties and corrosion resistance of the zirconium alloys. Their amounts are varied, (0.05–1.70 wt.%). Iron and tin have Mössbauer isotopes, therefore it is possible to study their states and their effect on the zirconium alloy properties using Mössbauer spectroscopy.

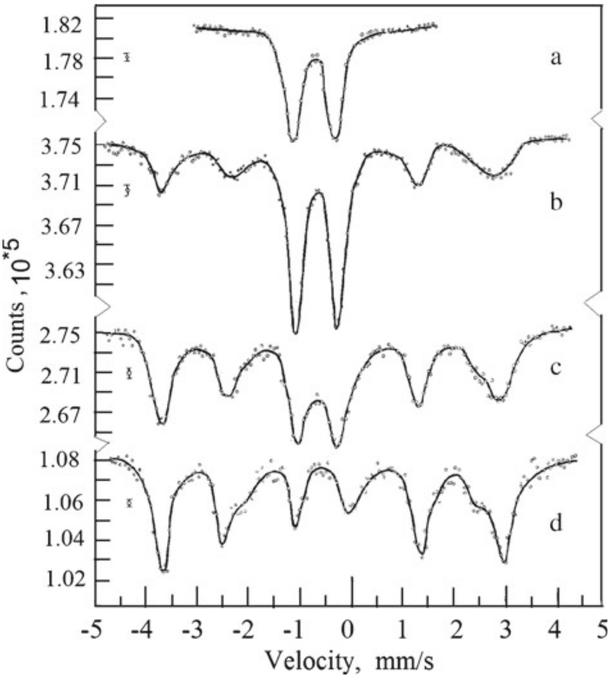
For improving zirconium alloy properties it is important to know the solubility of iron atoms in α-Zr [1], and the types of solid solutions and the structure of intermetallic compounds of zirconium. Alloys are doped with many elements and it is important to know the influence on iron atoms states of these doped elements [2–12]. To obtain the necessary properties, zirconium alloys are treated thermo mechanically and sometimes irradiation is used. Due to low concentrations of Fe and Sn atoms in the alloys sometimes only Mössbauer spectroscopy can provide qualitative and quantitive information. The next question deals with the development of new technologies with changing near surface layer properties of alloys, for example, by ion or laser irradiation. It seems that only Mössbauer investigations can give useful and complete information. The Mössbauer effect allows us to obtain quantitive results about the influence of reactor irradiation and radiation growth. These results could give us the possibility of understanding the correlation between radiation growth and initial state of iron atoms and to obtain quantitive data about phase transformations in alloys during reactor irradiation.

2 Experimental technique

In most research work devoted to zirconium alloys, the alloys were prepared from the iodide zirconium. Melting was carried out in an electro arc furnace in a purified argon or helium atmosphere or by electron-beam melting in a vacuum. After melting, the samples in ingots were exposed to forging with intermediate annealing, hot rolling in a copper cover at temperatures ranging 700–800 °C to a thickness ~3 mm. The next step was cleaning, the removal of copper from the surface of the samples and then cold rollings were carried out to a thickness of 20–150 microns with the intermediate annealing conducted at 580 ÷ 620 °C. Foils were cut from these samples for Mössbauer spectroscopy studies. Considering the possibility of fragile intermetallic compounds after the necessary thermal treatment, a powder from the treated alloys was prepared using a diamond file. Absorbers were prepared for Mössbauer investigations from these powders. Intermediate annealing was carried out in a strong vacuum of $p = 10^{-4} \div 10^{-3}$ Pa.

Mössbauer sources ^{57}Co(Cr), ^{57}Co(Rh) and Mössbauer spectrometers operating at constant speed or constant acceleration were used. The detector with a thin crystal of NaI(Tl) was used for registration of transmission radiation. Proportional gas counters were used for the registration of the scattered X-ray. For registration of conversion electrons, the flowing proportional counters with helium mixed with methane (5–8%) were used. Isomer shifts were given relative to metallic iron and SnO_2.

Fig. 1 Mössbauer spectra ^{57}Fe (obtained at $T = 298$ K) in zirconium alloys with concentration of iron (wt.%Fe): **a** 24.00, **b** 26.15, **c** 37.15, **d** 55.15



3 Intermetallic compounds in binary Zr-Fe alloys

Before Mössbauer investigations only one intermetallic compound $ZrFe_2$ was considered in the Zr-Fe system in accordance with the Hansen phase diagram [13]. There were some predictions about the existence of other intermetallic compounds such as Zr_4Fe, Zr_3Fe, Zr_3Fe_2, and Zr_2Fe but they were not confirmed experimentally. For the first time the existence of intermetallic compound Zr_2Fe has been confirmed experimentally by Mössbauer spectroscopy [2]. In paper [2] the alloys with iron content ranging from 0.09 wt.% Fe to 57 wt.% Fe were investigated. The spectra obtained at room temperature are shown in Fig. 1. It was observed that the spectra of alloys up to 24 wt.% Fe have the shape of a hyperfine quadrupole splitting with spectral parameters $\Delta E = 0.60 \div 0.90$ mm/s, $\delta = -0\ 32 \pm 0.03$ mm/s. After 24 wt.% (33 at%) Mössbauer spectra show a superposition of the hyperfine lines of quadrupole splitting with the above parameters, and two systems of the hyperfine magnetic splitting with the spectral parameters $H_{\mathrm{eff1}} = 19.9 \pm 1.0$ Tl, $H_{\mathrm{eff2}} = 18.6 \pm 1.0$ Tl, $\Delta\ E_1 = 0.48 \pm 0.03$ mm/s, $\Delta\ E_2 = 0.27 \pm 0.03$ mm/s, $\delta_1 = -0.19 \pm 0.03$ mm/s, $\delta_2 = -0.19 \pm 0.03$ mm/s.

With increasing the concentration of iron, the lines intensities of the paramagnetic phase decrease and those of magnetic phase increase. The lines of magnetic hyperfine splitting in accordance with papers [14–16] corresponded to the compound $ZrFe_2$. The authors of [2] concluded that there is another intermetallic compound in the system Zr-Fe, which was named Zr_2Fe.

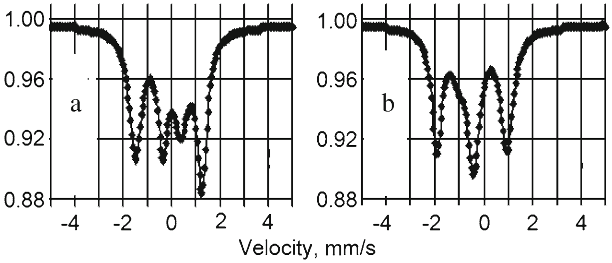


Fig. 2 The spectra of ^{57}Fe (obtained at $T = 298$ K) in the intermetallic compounds of the samples in magnetic fields $H = 4.5$ Tl: **a** Zr_2Fe, $eQV_{zz}/2 = -0.72$ $n = 0$; **b** Zr_3Fe, $eQV_{zz}/2 = +0.80$ mm/s, $\eta = 0.6$

Ten years later using measurements in magnetic fields, Aubertin [3] found that in Zr-Fe there is another intermetallic compound, Zr_3Fe. The spectra are given in Fig. 2. Figure 2 shows that in the magnetic field the spectra of compounds Zr_2Fe and Zr_3Fe are different. The difference is due to the different sign of the electric field gradient at the iron nuclei in these intermetallic compounds. The isomer shift for both compounds is the same $\delta = -0.32 \pm 0.02$ mm/s. Thus, now it is established that in the annealed alloys with small concentrations of Fe atoms, iron is mainly in the form of Zr_3Fe ($\Delta E > 0.75$ mm/s, $\delta = -0.32$ mm/s). Existence of these three intermetallic compounds (Zr_2Fe, Zr_3Fe and $ZrFe_2$ in Zr-Fe system) was proved also by x-ray researches.

Numerous researches [1, 4, 17–22] show that after quenching alloys from β-region, different paramagnetic phases can be formed, including intermetallic compounds with different relative contents of elements. Such formation depends on the Fe atoms content in the alloy (from 0.02 wt.% up to 2 wt.%) and the quenching rate. In some papers [4, 18–20] it was affirmed that supersaturated solid solution Fe in α-Zr (with spectral parameters $\delta = -0.09 \div 0.12$ mm/s $\Delta E = 0.30 \pm 0.03$ mm/s) and intermetallic compound Zr_2Fe are formed. Sometimes the solid solutions is described with a single, $\delta = 0.01 \div 0.028$ mm/s. In other cases several other phases are introduced such as θ-phase ($\delta = -0.369 \pm 0.008$ mm/s), β-phase ($\delta = -0.135 \pm 0.008$ mm/s, $\Delta E = 0.252 \pm 0.008$ mm/s), thermal ω phase ($\delta = -0.561 \pm 0.008$ mm/s) and athermal ω phase ($\delta = -0.589 \pm 0.008$ mm/s). In papers [7, 23–25] phases with similar spectral parameters are interpreted as other intermetallic compounds, and even as an amorphous state of intermetallic compounds [26]. Based on obtained results a table of the intermetallic compounds spectra parameters is created [25]. One can see the differences in interpretation of spectra. The lines with little differences in isomer shift and quadrupole splitting were interpreted with different phases. Using a substantial number of results authors of paper [7] summarized the data and obtained the interdependence of the isomer shift, quadrupole splitting and the iron concentration in the system Zr-Fe. The obtained results are given in Fig. 3.

Thus, the existence of many phases and compounds is described in the Zr-Fe system. There is an overlap of the spectra parameters for different phases and compounds. This phenomenon should be taken into account during the interpretation of experimental data and one must use additional data for a reliable interpretation of experimental results.

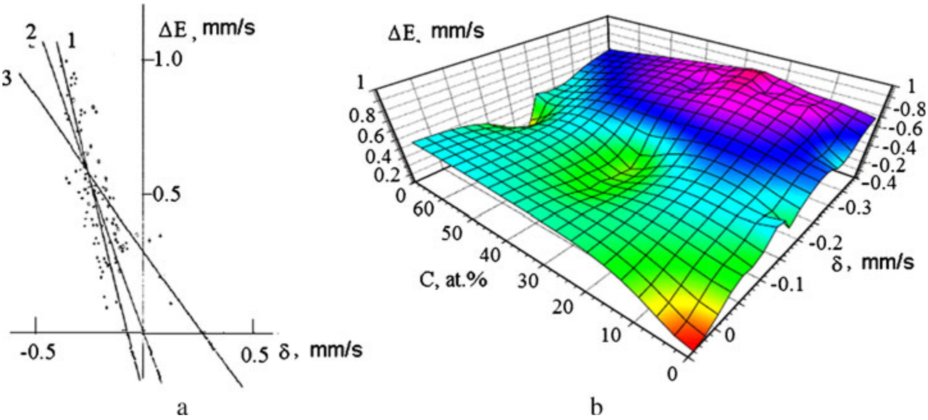


Fig. 3 **a** Interaction between isomer shift and quadruple splitting in alloys with different structure types; **b** diagram concentration, isomer shift, quadruple splitting for zirconium rich alloys $Zr_{1-x}Fe_x$

4 The solubility of iron atoms in the α-Zr

Estimation of the iron atoms solubility in α-Zr was carried out in papers [1, 4, 6, 18–20, 22]. To determine the solubility of iron atoms samples were annealed for a long time (100–320 h) at given temperature and then quenched. Because of annealing, some iron atoms from the supersaturated solid solution formed intermetallic compounds and others remained in the solid solution. Quenching fixed this state. The results obtained from these studies were in accordance with each other. Figure 4 shows the Mössbauer spectra and results of their graphical presentation from papers [1, 22]. The next question concerns the state of the iron atoms in solid solution. Do they form a substitutional solid solution or interstitial? Metals tend to form solid solutions of substitution. However, taking in to account that the size of the iron atoms is smaller than that of zirconium atoms, we can estimate the possibility of interstitial solid solution formation too.

Samples of alloy Zr-0.09 wt.%Fe annealed at 750 °C were saturated with oxygen to clarify this question. Analysis of the data given in [28] and additional calculations allowed to show, that after annealing the iron atoms are in solid solution and in intermetallic compounds and their relative parts correspond to the data given in Fig. 4 from paper [22]. After saturation with oxygen (1.65 at%) solid solution lines have disappeared and only the lines of intermetallic compounds Zr_3Fe, Zr_2Fe, $Zr_2Fe + O$ are visible. Taking into account that oxygen atoms form interstitial solid solution, one can assume that they have replaced the iron atoms from these positions and thus Fe atoms have formed intermetallic compounds. In the case of the iron atoms in the substitution solid solution the probability of their displacement from the lattice of zirconium would be less likely. However, the substitutional solid solutions were obtained by implantation of Fe atoms in Zr [27]. This solid solution is described by components with quadrupole splitting with $\delta = 0.08 + 0.03$ mm/s

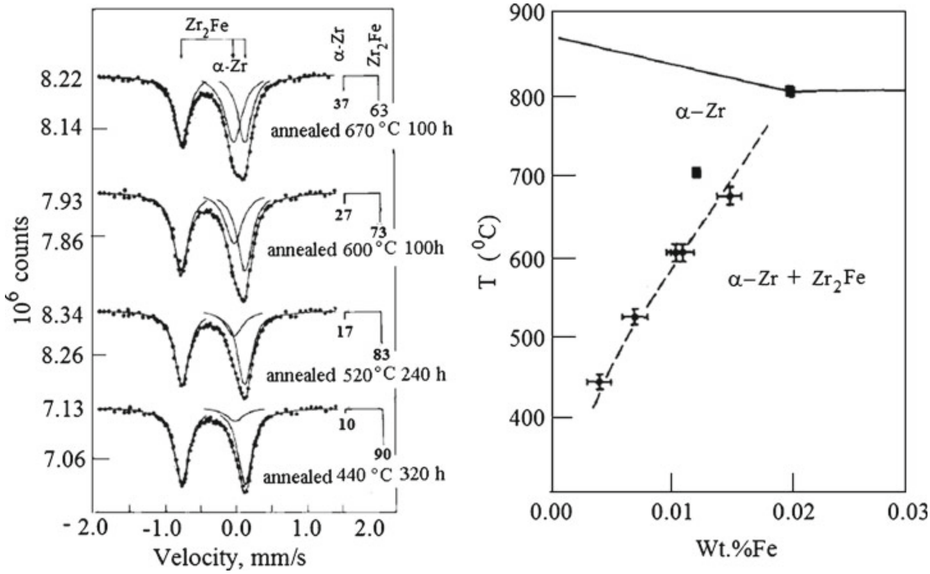


Fig. 4 Mo¨ssbauer spectra of ^{57}Fe in Zr-0.04 wt.%Fe after annealing and quenching (obtained at $T = 298$ K) (*left side*) and results calculations of iron atoms solubility (*right side*)

$\Delta E = 0.10 + 0.05$ mm/s. This state was observed in the quenched sample of the binary alloy subjected to annealing at 350 °C (third state). After a long temperature treatment only the interstitial solid solution is observed.

5 The influence of other elements on the state of the iron atoms

As mentioned above, alloying elements other than Fe atoms such as Sn, Cu, W, Cr, Ni and Nb are used. These elements play an important role in the improvement of the mechanical, corrosion and radiation properties of zirconium alloys.

The effect of tin atoms Adding Sn atoms to Zr-Fe alloys in the Zirconium corner does not change the shape of Fe spectra. According to phase diagram [13] the Sn atoms should be in solid solution and in an intermetallic compound, Zr_4Sn. In papers [4, 5, 8, 28–30] it was shown that in alloys with Sn atoms of concentration up to 3 wt.% are in solid solution in α-Zr with Mössbauer spectra parameters $\delta = 1.6 \div 1.4$ mm/s, $\Delta E = 0.33 \div 0.43$ mm/s. The lines of Zr_4Sn ($\delta = 1.1$ mm/s, $\Delta E = 0.86$ mm/s) are not observed at these concentrations.

The spectral shape corresponds to Zr_2Fe or Zr_3Fe depending on annealing time. However, the influence of tin is determined by the decomposition rate of the supersaturated solid solution of Fe atoms in α-Zr. Paper [28] showed that during annealing of quenched alloy (Zr-1.21 wt.%Fe) there was an increase of the solid solution spectrum area for a short time after $t = 2$ h. In case of presence of Sn atoms in the alloy such phenomena was observed too, but after 16 h of annealing at the same temperatures.

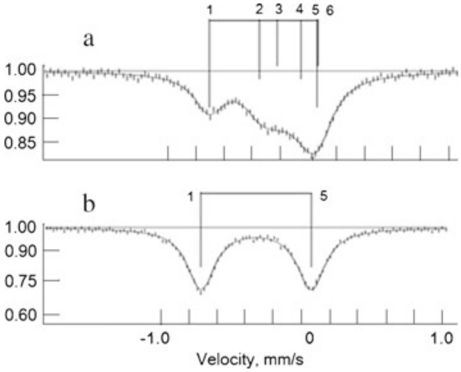


Fig. 5 The spectra of ^{57}Fe (obtained at $T = 298$ K) in the alloy Zr-1.21%Fe −0.81%Cu −0.40%W: **a** after quenching from the beta region; **b** after tempering at $t = 500$ °C 2 h

Table 1 Parameters of Mössbauer spectra of Zr-Fe compounds

Phase	Isomer shit, mm/s	Quadruple split, mm/s	Composition, at% Fe
$ZrFe_2$ (C15)	−0.22	0.46(1)	67
Zr_2Fe ($CuAl_2$)	−0.31(1)	0.75(1)	33
Zr_2Fe (Ti_2Ni)	−0.12(1)	0.30(1)	33
Zr_3Fe	−0.33(1)	0.91(1)	25
α-Zr_4Fe	−0.34(1)	0.85(1)	20
β-Zr_4Fe	−0.3(1)	0.75(1)	20
ZrFe	−0.15(1)	0.32(1)	0.02
ZrFe	0.04(1)	–	0.02

Effect of copper and tungsten atoms Effect of additional doping atoms of tungsten on the iron and tin atoms state is considered in paper [34]. The spectra of the alloy sample with additional doping of copper and tungsten atoms (Zr-1.21% Fe-0.81% Cu-0.40% W) are shown in Fig. 5. Data of fitting are given in Table 2. After quenching, the spectrum (Fig. 5 a) can be fitted by 3 systems of lines of quadrupole splitting: 2 systems of solid solution (2–4, 3–6 (from left to right)) (may be 2–4, according the Table 1 represent Zr-Fe) and one – of Zr_2Fe (1–5). After annealing, only lines of Zr_2Fe are visible (Fig. 5 b). The differences with the binary alloy are as follows: 1) it was not possible to reach $\Delta E = 0.9$ mm/s as for Zr_3Fe in spite of long annealing; 2) it was not possible to obtain only solid solution with quenching procedure (Table 2).

Effect of chrome atoms Chrome atoms are used in Zircaloys, in threefold and fourfold alloys. Influence of the Cr atoms was demonstrated in paper [5] during investigations of Zr-1.0%Fe-1.27%Sn-0.51%Cr (wt.%) alloy. The spectra of the annealed sample at $t = 600$ °C during 2 h are shown in Fig. 6. The chrome atoms considerably change the spectral shape. The fitting of annealed and quenched samples spectra gives two doublets with $\delta 1 = -0.19 \pm 0.02$ mm/s and $\delta_2 = -0.23 \pm 0.02$ mm/s, $\Delta E_1 = 0.29 \pm 0.04$ mm/s and $\Delta E_2 = 0.21 \pm 0.04$ mm/s. These parameters correspond to spectra parameters of particles $Zr(Fe,Cr)_2$ with C14 and C15 structures [6]. It is necessary to note that on the spectrum of the initial sample the lines of Zr_3Fe and Zr_2Fe are not visible. The main feature of this alloy is that after a period of 2 years at room temperature the lines of intermetallic compound Zr_3Fe on spectra

Table 2 Spectra parameters after thermal treatment

Treatment	No. lines	δ mm/sc	Δ E mm/s	Area	W mm/s
Quenching from beta region	1–6	-0.32 ± 0.01	0.77 ± 0.01	44 ± 1	0.264
	2–4	-0.20 ± 0.01	0.30 ± 0.01	30 ± 5	0.264
	3–5	-0.08 ± 0.01	0.29 ± 0.01	26 ± 5	0.264
Annealing	1–6	-0.32 ± 0.01	0.78 ± 0.01	100 ± 1	0.234

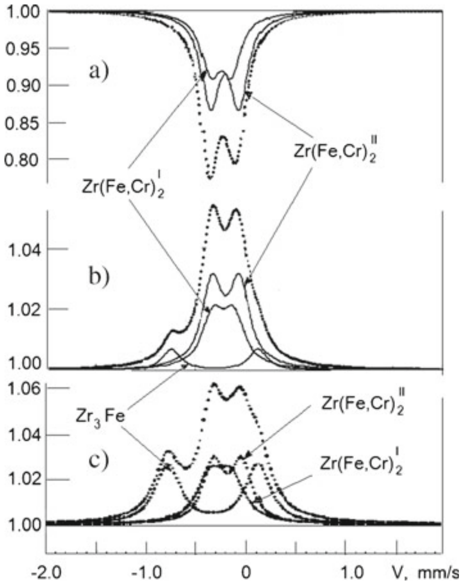


Fig. 6 ^{57}Fe spectra in Zr-1.0 wt.%Fe-1.27 wt.%Sn-0.51 wt.%Cr (obtained at $T = 298$ K) after 2 years: **a**) transmission, **b**) CEMS and **c**) CEMS after 6 years

were detected. The intensity of the lines eventually increased with time, especially on CEMS spectra [10] (Fig. 6 b, c).

Effect of niobium atoms Niobium is included into all main alloys for nuclear industry. Zr and Nb form both solid solutions and intermetallic compounds.

The shape of niobium containing alloys spectra considerably depends on both structure of the alloys and the type of thermal treating. One of the first papers devoted to alloys containing niobium is Tanaka's one [9]. Further research of their influence on the state of Fe atoms state are given in papers [24–26, 31–33, 35–38].

The majority of researchers come to a conclusion that in alloys with niobium, iron atoms can be in solid solution in α-Zr and in intermetallic compounds $(Zr_{1-x},Nb_x)_2Fe$, $(Zr_x,Nb_{1-x})Fe_2$. The spectra of the threefold alloy from paper [35] are given in Fig. 7, and spectra of a fourfold alloy are in Fig. 8.

The spectral parameters of Nb containing samples have a wide range. For example, $(Zr_{1x},Nb_x)_2Fe$, ($\Delta E = 0.5 \div 0.75$ mm/s with $\delta = -0.28 \div 0.34$ mm/s), and for $(Zr_{1-x},Nb_x)Fe_2$ ($\Delta E = 0.20 \div 0.51$ mm/s, $\delta = -0.16 \div 0.28$ mm/s). This is because of the difference in Zr and Nb content in intermetallic compounds.

Fig. 7 ^{57}Fe spectra in Zr-0.31 wt.%Fe-0.51 wt.%Nb (obtained at $T = 298$ K); **a**) after deformation; **b**) after annealing (700 °C, 15 h)

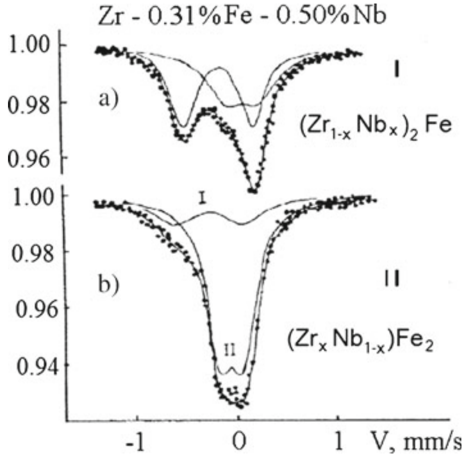
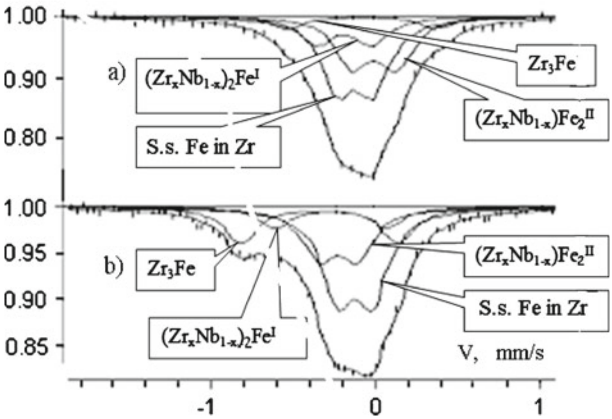


Fig. 8 ^{57}Fe spectra in Zr-0.69 wt.%Fe-1.35 wt.%Sn-0.88 wt.%Nb (obtained at $T = 298$ K); **a**) after quenching; **b**) after annealing (350 °C, 3 h)



Influence of Ni atoms It seems that Ni substitutes Fe atoms in intermetallic compounds Zr_2Fe and Zr_3Fe. Addition of Ni atoms in binary Zr-Fe alloy decrease the value of ΔE to 0.57 mm/s.

6 Redistribution of iron atoms in samples under thermo mechanical treatment and irradiation

Iron atoms under heat treatment tend to move to the surface and as the result, they enrich the thin layers (0.1 microns) on the surface with intermetallic particles. This enrichment can reach 5 wt.%, at initial concentration of 0.35 wt.%Fe [35].

The influence of laser radiation on enrichment of near surface layers of iron is different for the annealed and deformed samples. The reason for such behaviour of iron atoms is explained by fast diffusion of iron atoms and by existence of defects in the deformed samples of alloys [25].

Irradiation with an Ar ions beam with a wide power spectrum with middle ion energy in beam <E> = 10 keV, with current of beam $j = 20 - 30$ μcA/cm^2 and dose of $5 \cdot 10^{17}$ ion/cm^2 leads to a change of ratio of intermetallic compounds types in near surface layers both to disintegration of one and to formation of other compounds [38].

Irradiation by neutrons in reactor conditions was investigated in paper [39]. The samples were irradiated to two fluencies: first $2.2 \cdot 10^{25}$м$^{-2}$ and final $11.0 \cdot 10^{25}$м$^{-2}$. It was shown that:

- under the influence of neutron irradiation there is a redistribution of iron atoms between the phases and it is dependent on initial thermo mechanical treatment;
- in the annealed sample after the first stage of radiation the concentration of iron in solid solution in zirconium and Zr_3Fe compounds increases at the expense of disintegration of intermetallic compounds of $Zr(Nb, Fe)_2$ and $(Zr, Nb)_2Fe$; during the second stage of radiation the phase structure of this sample changes slightly;
- in a cold rolled sample after the first stage of radiation there is an increase of iron concentration in solid solution and in compounds Zr_3Fe and $(Zr,Nb)_2Fe$ at the expense of disintegration of intermetallic compound of $Zr(Nb,Fe)_2$; after the second stage of radiation concentration of iron in Zr_3Fe increases owing to disintegration of $Zr\,(Nb, Fe)_2$ and $(Zr, Nb)_2Fe$ compounds.

7 Conclusion

With the help of Mössbauer spectroscopy, the important characteristics of zirconium alloys were determined. These characteristics included new intermetallic compounds, solubility of iron atoms in α-Zr, changing of phase compositions under influence of different factors like thermo mechanical treatment, oxidation, corrosion tests and irradiation. Some phenomena were discovered on the surface of these alloys. The existence of different types of iron solid solution in α-Zr was distinguished and diffusion of iron atoms and phase transformations, not only under irradiation or thermo mechanical treatment but also at room temperature were shown.

Utilization of Mössbauer spectroscopy will be very useful for obtaining fundamental knowledge in this field of science and technique.

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