# STUDY OF THERMAL STABILIZATION KINETICS IN Zr-Fe LAMELLAR SYSTEM

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## 1. INTRODUCTION

In the present work within the framework of thermo-dynamical approach, which has been successfully applied earlier in creation of stable laminated materials on the base of iron-beryllium system [1], there were studied the thermally induced phase transformations in laminar iron-zirconium systems obtained using the magnetron sputtering.

## 2. EXPERIMENTAL

Thin film samples were prepared of α-Fe with purity of 99.98% rolled to thickness of 5±1 μm and annealed at 850°C during 3 h to eliminate the crystalline lattice defects induced at rolling. The prepared foils were covered with high purity zirconium layer of 2±0.3 μm by magnetron sputtering. The samples were sequentially annealed at 900°C during up to 50 hours.

In our research thicknesses of zirconium layer (2μm) and iron layer (5μm) were selected to assure mean concentration of zirconium in the sample bulk ($C_{Zr} \cong 16.7$ at. %) to be in areas of the phase diagram [2], which consists of pure α-Fe and corresponding intermetallic compounds: $Fe_2Zr$ and $Fe_3Zr$.

As the methods of analysis have been used Mossbauer spectroscopy (MS) and X-ray diffraction (XRD). Mossbauer investigations have been carried out at spectrometer MS1104ME in transmission at room temperature using ~ 50 mCi Rh ($^{57}Co$) as a source of γ-quanta. The calibration of a spectrometer was realized by α-Fe standard sample. The processing of experimental spectra has been done by DISTRI program [3]. The XRD measurements were spent in Bragg-Brentano geometry from both sides of specimen.

## 3. RESULTS

After of two-hour's duration annealing in MS-spectrum (see Fig. 1) on the background of lines from α-Fe the appearance of totality of additional sextets of ferromagnetic phase with essentially smaller hyperfine fields ($\overline{H_n}$ ~ 207 kOe) have been observed.

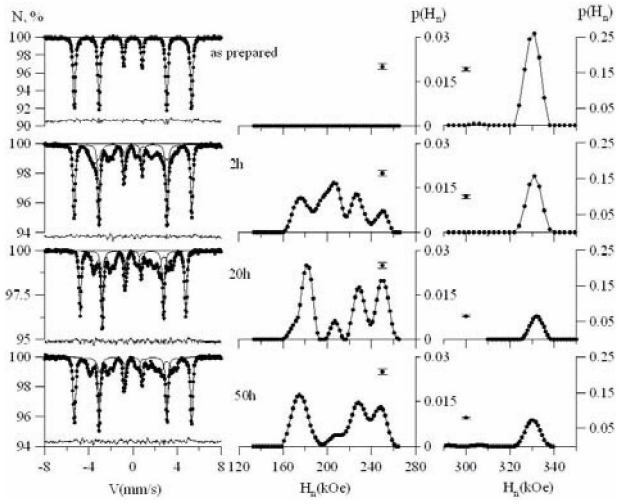



Fig. 1 MS-spectra and restored function $p(H_n)$ of hyperfine field for the lamellar system Fe(5 μm)-Zr(2 μm) after isothermal ($T_{ann}$=900°C) annealing

The functions of hyperfine magnetic field distribution have been restored. It is seen (Fig. 1) that the distribution of hyperfine fields has several maxima whose field values correspond to $Fe_2Zr$ and $Fe_3Zr$ intermetallides. It is known [5, 6], that $Fe_2Zr$ phase consist of two sextets with effective magnetic fields on $^{57}Fe$ $H_n$=190±5 kOe and $H_n$=200±5 kOe nuclei with intensity ratio 1:3. $Fe_3Zr$ phase is characterized by three sextets with $H_n$=180±5, 224±5 and 241±5 kOe fields with intensity ratio 2:1:1. There is a difficulty in determination of contribution value of each phase because the values of hyperfine fields of compounds are very close. This problem can be resolved if the distribution of hyperfine magnetic field for intermetallides considers as experimental spectrum. Fig. 2 presents the processing result of hyperfine field for the lamellar system Fe-Zr by the model of five lines: doublet ($Fe_2Zr$) and triplet ($Fe_3Zr$).

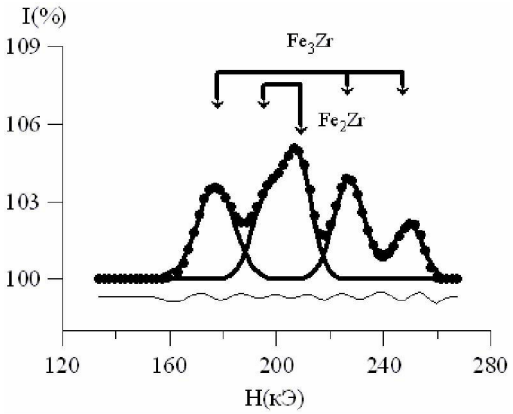



Fig.2 Model decoding result for distribution function of effective magnetic field $p(H_n)$ ($T_{ann}$=900°C, $t_{ann}$=2h)

The distributions for MS-spectra at each stage of thermal annealing were subjected to such processing. On the base of obtained data there were made dependencies of relative intensities of partial spectra α-Fe and intermetallides (see Fig.3).

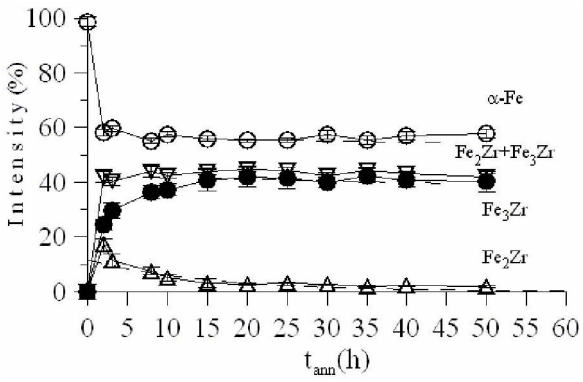



Fig.3 The relative intensities of partial Mössbauer spectra of iron-containing phases versus duration of isothermal annealing at 900°C

As it is seen from the figure, the decreasing of relative contribution from α-Fe from 100 to ~60 % after first annealing has been taken place. During further increasing of annealing time the relative contribution from α-Fe isn't changing. At that the relative contribution from $Fe_2Zr$ intermetallide decreases and from $Fe_3Zr$ increases. As it seen (see Fig.3) the further isothermal annealing will result in thermal stabilization of $Fe_3Zr$ intermetallide on the surface of α-Fe matrix.

Fig. 4 presents x-ray patterns of Fe-Zr lamellar system, from which one can see that 2-hour annealing results in formation of $Fe_2Zr$ (triangles) and $Fe_3Zr$ (black circles) intermetallide compounds. A little quantity of zirconium atoms reacts with oxygen, forming $ZrO_2$ (circles). After 30-h annealing (Fig. 4) there can be seen only $Fe_3Zr$ and $ZrO_2$ reflexes and α-Fe matrixes (black rhombs) on the pattern. The reflexes of $Fe_2Zr$ are disappeared.

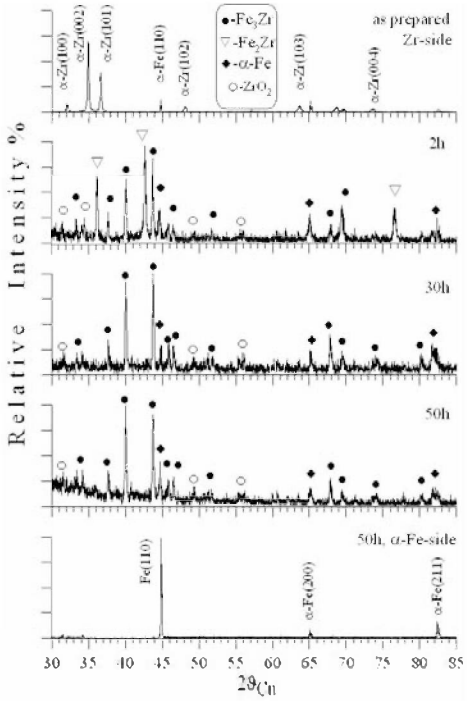



Fig. 4 XRD patterns of the lamellar system Fe (5 μm)-Zr (2 μm) after isothermal ($T_{ann}$=900°C) annealing; 0h , 2h , 30h, 50h and pattern from α-Fe-side at 50h.

At the same time on the XRD pattern measured from the reverse side of the sample annealed at 50 h only lines characteristic for α-Fe have been observed (Fig. 4).

Fig.5 presents normalize intensities (squares) of XRD reflexes of phases versus duration of isothermal annealing. After two-hours annealing practically all free zirconium atoms are interact with iron atoms, forming the intermetallic compounds.

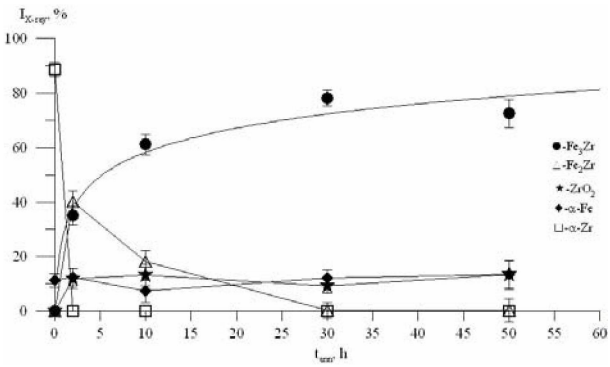


Fig.5 Normalize intensities (squares) of XRD reflexes of phases versus duration of isothermal annealing

Following annealing lead to decreasing the contribution of $Fe_2Zr$ and increasing the contribution of $Fe_3Zr$ phase. The contribution from $ZrO_2$ oxide with increasing of annealing time undergoes insignificant changes. The comparison of Mossbauer and X-ray kinetic curves show the lamination of Fe-Zr system. Since the solid solution of Zr in Fe was not observed, then could say about thermal stability of $Fe_3Zr$ phase on the α-Fe substrate.

## 4. CONCLUSIONS

1) The relative quantity of the phases formed in the bulk of the sample on each stages of annealing was received.

2) The original model decoding of the distribution function of effective magnetic field p (Hn) has been suggested.

3) It is shown, that the relative fraction of α-Fe does not change with increasing of annealing duration while the relative contributions of $Fe_2Zr$ and $Fe_3Zr$ phases are decreases and increases, respectively. The

increasing of annealing duration results to stabilization of $Fe_3Zr$ intermetallide on the surface of $\alpha$-Fe substrate.

4) It is shown that the direction of phase transformations is determined by the change of local concentration of components.

## 5. REFERENCES


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