CHANGES OF ELECTRON DENSITY AND DEFECTS DISTRIBUTION IN BINARY AND TERNARY IRON ALLOYS STUDIED BY POSITRON ANNIHILATION

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Abstract

Properties of binary and ternary iron based alloys doped by different additions were investigated. It was shown for binary alloys containing chromium, molybdenum and tungsten were that results of doping by 0.8% molybdenum and tungsten were similar to those for the sample doped by 9%chromium. Ternary alloys containing chromium and less amounts of molybdenum, tungsten and vanadium were investigated as well. Two types of defects were observed: divacancies and cluster-like defects. It was shown that the electron density in ternary alloys is similar to that in binary alloys containing 0.8% molybdenum.

Keywords: Positron annihilation; Lifetime; Vacancy; Cluster; Dislocation.

1. Introduction

Creation of construction materials with predictable properties is one of the basic problems of nuclear materials science. Steels are known to be such materials for fast neutron reactors. Steels of different classes doped in different ways are used, doping resulting in their properties. These properties determine the behavior of the material in irradiation fields. These peculiarities are of great importance for high-chromium steels since they have properties essentially similar to the expected operational characteristics. A model alloy containing nearly 9% of chromium is considered to be the basic one. Properties of the alloy containing 9% of chromium are compared with the similar properties of alloys, which contained tungsten and molybdenum of fewer amounts must be investigated as materials stopping the defects migration. Since these elements may solvate only at low concentrations, the amounts of the order of 1% are to be investigated. Phase changes were studied in [1-3]. The behavior of nickel addition to the powder pre-alloy of Fe and Mo was studied in work [1], where nickel concentration influence on the alloy properties was shown. The phase content of intra-granular ferrite – martensitic steels regions was investigated in [2]. Different temperature ranges were studied and different phase content was determined. The main result of work [3] was the study of ternary alloys based on magnesium. In this work different phase diagrams were studied as well. The most neighboring investigation was carried out in [4], where the properties of Fe+9%Cr were studied provided different doping. It is necessary to mark that investigation of binary alloys with molybdenum and tungsten was not carried out, although they may be used in radiation fields since such impurities may stop the defects migration.

Different heterogeneities can appear during thermal or mechanical treatment of the material. Those are structure defects of different types. For example two sorts of defects can appear after rolling, these defects being vacancy clusters and dislocation, a dislocation being a drain for vacancies. These defects influence on the further behavior of the material under different conditions of irradiation..

Thus measurements of defects number and sizes allow making predictions on behavior of the material under irradiation. Positron annihilation method based on measurement of the electron structure difference between that in the non-defective volume and in structure defects was used for vacancy- like defects diagnostics during a long time. Positrons diffusion [5-7], different types of defects generated in pure metals

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Dedicated to the memory of Professor Dragana Živković

[8], macroscopic defects – voids [9], annihilation on the crystal boundaries had been studied in different works. But the use of the method for analysis of electron density changes in construction materials is more completed without study of binary alloys, which simulate the behavior of an important component of ternary alloys. The essential peculiarity of results obtained in [11-17] is their qualitative kind as a rule and technique of the defects identification is imperfect.

Systematic study of construction materials was carried out in [11-30]. The following materials were under investigation: steels of ferrite –martensitic class [11-14, 18, 19, 20, 26-30], vanadium alloys [20], NiCr alloys [21-23], oxide dispersion alloys [15] and model alloys [4, 16-17, 24]. Nuclear gamma resonance (NGR) method is used in [25] for short-range phases determination and for setting the correlation between the type of short-range ordering and changes of electron density. It must be marked that experiments with industrial steels, both strengthened and usual, cannot be interpreted unequally, since new phases may be created. That's the reason for carrying out the experiments with other techniques. NGR method was used in [25] as an additional method since it is sensitive to short-range ordering. Nevertheless industrial materials (steels) were investigated in [26-30]. It was shown in [26] that positron annihilation lifetime technique is the most sensitive instrument of studying defects with small number of vacancies. So this technique was used for investigation of post radiation defects. One of the most interesting results is observation of the dependence of positron's lifetime on helium concentration. Besides that defects of different types were observed, their occupation by helium being the reason for lifetime change [30]. It should be marked that use of fast particles [29] may be of interest for space radiation physics. The above mentioned samples were investigated by means of two techniques: measurement of positron's lifetime and Doppler broadening of the annihilation. The most interesting result is observation of the complex Cr+vacancy and the effect of small amounts of carbon resulting in a rather long annihilation component

Model alloys would be investigated in order to predict the changes in the material. It was shown in [24] that small changes of the doping regime may result in significant changes of the material properties. The nature of the changes becomes clear provided using NGR method in combination with positron annihilation [25].The model alloys in [4,17] have only one doping element, so doping by two elements would also be investigated in this research.

The data obtained by two methods were compared, that comparison having been possible to set observation correlation between changes of electron density and short-range ordering. It should be marked that complex approach has been used in all experiments, this approach being based on use of theoretical models for defects diagnostics by means of electron density change observation. This approach contains a systematic error, since one must base on some a priory known parameters, for example, the

defect form and vacancy size. But such an approach may be used for diagnostics of defects sorts.

Investigation of binary alloys based on iron and containing chromium and vanadium as doping elements has been carried out in [24]. Difference in defects distribution has been observed in these alloys, the difference in defects generation and effective electron density distribution was explained in [25], where changes of short – range ordering in these materials had been observed.

It is known [31-33] that the most significant changes in short-range ordering are achieved at chromium concentration of the order of 8%.The necessity for carrying out experiments with chromium concentration higher than 8% [25] has been appeared in connection with this. Influence of other impurities should be investigated as well, tungsten and molybdenum being chosen for this goal in binary alloys.

2. Experiments.
2.1. Technique and instrumentation

Samples were cylindrical weight probes, their sizes were Ø15 and thickness 0.05 mm^3. Their chemical content is shown in table 1.

Table 1. Chemical content and thermal treatment regimes for binary alloys

Alloy	Content of elements, atomic per cent								
	Fe	Cr	Cu	Mn	Mo	Ni	V	W	T^0C
FeW	Basic	0.17	0.22	0.14	0	0.12	0	0.83	750
FeMo	Basic	0.16	0.21	0.12	0.82	0.1	0	0.02	750
FeCr	Basic	8.67	0.2	0.23	0.1	0.09	0	0.02	750
All samples were treated by cold deformation and annealed during 30 hours									

Besides binary alloys ternary alloys FeCrMo, FeCrV и FeCrW have been investigated, their content is shown in table 2.

Table 2. Chemical content of ternary alloys

Alloy	Fe	Cr	Cu	Mn	Mo	Ni	V	W
FeCrMo	Basic	8.5	0.2	0.19	0.88	0.12	0	0.02
FeCrV	Basic	8.4	0.12	0	0	0.6	4.51	0
FeCrW	Basic	8.72	0.21	0.21	0	0.1	0	0.87

Content of doping elements for ternary alloys were prepared using annealing at 1200-1300 C in quartz ampula during 30 hours and cooled within the furnace after rolling. Spectrometers with time resolution 300 ps and high stability of the temporary parameters are required for positron lifetime distribution measurements.

The prepared samples were investigated at the ORTEC spectrometer, which satisfies to these requirements, at the ITEP base. The structure scheme

of distribution measurements spectrometers produced by ORTEC Company is shown in fig.1.

The positron source is set inside the sample under investigation. Such disposition is necessary; else background photon emission due to annihilation in detector probability will sharply increase. Positrons penetrate to the sample and annihilate there. The annihilation and reference – point photons with energies 0.511 and 1.27 MeV are detected by detectors based on solid organic scintillators and photomultiplier time tubes (PMT) [35]. It should be marked that time resolution is basically determined by the detector unit; contributions of the scintillator and its PMT are of one order. Scintillators based on polystyrene and polymethyl metacrylate with different additions that shift the emanation spectrum towards waves with higher lengths are known to be the best of native scintillators [34].

PMT 30, 36, 87,143 are the most often used as time PMT.   Pulses from detectors enter two identical channels, where the amplitude pulse selection for search of pulses according to energies 0.511 and 1.27 MeV and the time binding is made. Each of the channels consists of a constant fraction differential discriminator-shaper for precision binding to the pulse front and a coincidence scheme, the precision binding pulse to the pulses from PMT detecting photons of the specified energy, which are formed at its output. The start pulse is set up to photons with 1.27 MeV, the stop pulse is set up to pulse 0.511 MeV. Pulses from coincidence scheme enter the start and stop entrance of the time to amplitude converter [34].

Time to amplitude converter (TAC) transforms time intervals between start and stop pulses to the output signal amplitude, which then enters for amplitude analysis to analogue-digital transformer and after it - to a computer. The time to amplitude converter use is specified by the fact that immediate coding of the time interval to a digital code in the spectrometer is impossible because of short time interval value (not more than 50 ns) and a short quantum pace (5-30 ps).). Thus the amplitude scale of the analyzer is equivalent to the time scale and the amplitude distribution is linearly connected with distribution of time intervals between detection of start and stop pulses by PMT. High and low voltage sources for PMT and scheme supply and the thermo-stability unit intended or reduction of instrumentation time parameters drift are included into the device as well.

It should be marked that events in the sample may be simulated by events in the source. One must measure lifetime in the source and lifetime in a known sample such as pure iron. Then during fitting the experimental data one must choose such thickness of the lay that the longest component of the annihilation spectrum of the basic sample would be zero. It is necessary to mark that the source lifetime is dependent on the lay material and sometimes is nearly zero . The criterion of coincidence of theoretical and experimental data (x_2) must be minimal. In our experiments the contribution of the layer does not exceed 6% and has 1.98 ns. So it is observed as a constant and may be neglected.

2.2. Discussion of results on binary and ternary alloys

A computer fit of data with the use of a special program POSFIT was carried out after obtaining the experimental data.

Results of fitting of experimental data for binary alloys are shown in table 3.

Table 3. *Results of experiments with binary alloys*

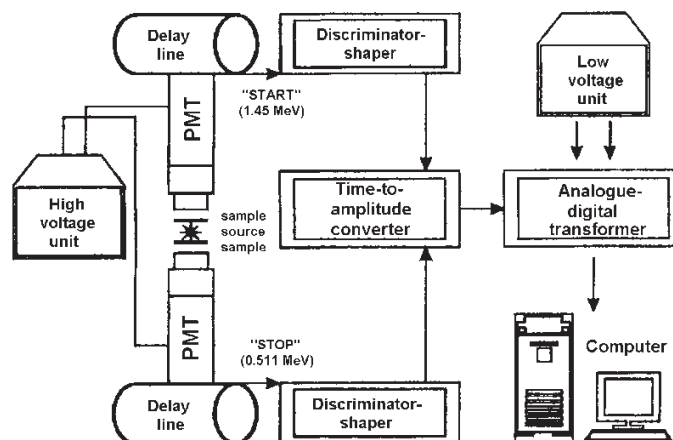| Sample under investigation	τ_1/ps	I_1/ %	τ_2/ ps	I_2/ %	χ^2	Third component
FeCr	123±2	76±2	280±30	24±5	1.1±0.1	Absent
FeMo	122±2	72±2	260±20	27±6	0.9±0.1	Absent
FeW	118±2	70±2	250±20	30±4	1.06±0.10	Absent
FeCr (9% spec.annealed)	118±2	72±2	380±20	27±2	1.02±0.10	Absent

Figure 1. *Structure scheme of the positron lifetime measurement spectrometer*

In table 3 $\tau_{1,2}$ are experimentally measured lifetimes corresponding to reduction of positrons from the non-defective volume and their annihilation in defects.

It may be seen from results obtained:

- The effective electron density in samples containing 8% of chromium and 0.8% of molybdenum is equal, the concentration of "large" defects is similar as well;

- A slight increasing of   for specially annealed at 700C during 150 hours show that the annihilation time of positrons in bulk is less than in the sample, containing 8% of chromium and annealed at 1200C.The annihilation time for the longer component is less in the 8% of chromium. That means partial annealing of the defects

It was supposed that all difference of electron density was connected with the difference in vacancy concentration, vacancies being present in small amounts in any sample. Fitting with fixed parameters corresponding to annihilation in vacancies and "large" defects was carried out in order to obtain results concerning annihilation in vacancies and "large" defects.

The results of fitting for binary alloys shown in table 4 are in the quality agreement with experimental data, obtained for imperfect samples.

In table 4 $\tau_{1,2,3}$ are the times of positron escape from the non-defective-ouldolume and lifetimes in a vacancy and a cluster, $I_{1,2,3}$ are corresponding to these times probabilities of positron stopping corresponding to non-defective volume, vacancy and cluster

It follows from data shown in table 4 that:

- the short-time component may be only found in the 9% chromium alloy, although a great amount of associated vacancies may be considered to be there;

- there is no short component in other samples;

- the influence   of the annealing regime is illustrated as an increase of the "pure" component of the annihilation lifetime, it should be noticed that other components don't reduce;

- there are defects of at least two types in the samples: vacancies or another regions with electron density, which is equal to the density in a vacancy (characteristic lifetime is 171-190 ps) and clusters, which may be located at the boundaries of the regions with homogenies electron density (the characteristic lifetime is 350ps):

- the highest concentration of vacancies is in the sample doped by molybdenum);

- the value of the component according to positron capture into a cluster is the highest for the sample doped by tungsten.

Experiments on measuring positron lifetime in ternary alloys were also carried out at the ORTEC set belonging to ITEP. Fitting was made in two stages like fitting of the results obtained for binary alloys. Results are shown in tables 5 and 6.

It is seen from data presented in tables 5 and 6 that doping by tungsten and molybdenum leads to the same results. Vanadium doping leads to increase of defects concentration, which may be considered to be clusters. One must calculate probabilities of positrons capture in order to make a more accurate estimation.

3. Defects concentration estimate
3.1. The positron capture model

We shall accept the model of positrons capture provided existing of the following regions of positron localization: the non-defective volume and two types of defects, since only three components were found in

Table 4. Results of fitting with fixed parameters.

Material of the sample	τ_1, ps	I_1, %	τ_2, ps	I_2,%	τ_3, ps	I_3,%	χ^2
FeCr	115	61.8±0.3	190	27.2±0.5	350	10.4±0.2	1.08±0.04
FeMo	115	60.0±0.3	190	30.0±0.8	350	10.1±0.2	0.99±0.04
FeW	115	62.1±0.3	190	26.7±0.8	350	11.1±0.2	1.05±0.04
Fe+9%Cr, special	109	48.3±0.3	171	36.8±0.3	389±20	15.±1.	1.04±0.06

Table 5.   Results of ternary alloys investigation experiments (without fixing the decay exponents)

Material of the sample	τ_1/ ps	I_1/ %	τ_2/ ps	I_2/ %	τ_3/ ps	I_3/ %
FeCrMo	129±1	82.5±0.6	316±6	18.0±0.6	1.08±0.05	Absent
FeCrV	128±1	81.7±0.4	314±4	18.3±0.6	1.10±0.05	Absent
FeCrW	129±1	81.8±0.6	317±4	18.2±0.6	1.03±0.04	Absent

Table 6. Results of ternary alloys investigation experiments (with fixing the decay exponents)

Material of the sample	τ_1/ ps	I_1/ %	τ_2/ ps	I_2/ %	τ_3/ ps	I_3 /%	χ^2
FeCrMo	115	60.4.±0.4	190	30±1	350	9.5±0.4	1.07±0.09
FeCrV	115	61.1±0.6	190	28±1	350	10.4±0.5	1.12±0.09
FeCrW	115	60.3±0/4	190	30±1	350	9.7±0.4	1.002±0.098

experiments.

In addition to this we shall accept the defects to be deep potential minima, so we consider positron escape during their lifetime to be impossible. So we get the following differential equations system

$$\frac{dN_1}{dt} = (-\lambda_1 - \mu_{12} - \mu_{13}) \cdot N_1$$

$$\frac{dN_2}{dt} = \mu_{12} \cdot N_1 - \lambda_2 \cdot N_2$$

$$\frac{dN_3}{dt} = \mu_{13} \cdot N_1 - \lambda_3 \cdot N_3$$

Here $\lambda_{1,2,3}$ are the positron annihilation rates in the non-defective volume and in two different types of defects, $\mu_{12,13}$ are positron capture rates to both types of defects. Besides that positron's initial location in the non-defective volume is also considered to be. That means $N_1(0)=N_{10}$, $N_2(0)=0$; $N_3(0)=0$.

$$N(t) = (1 - \frac{\mu_{12}}{\lambda_1 + \mu_{12} + \mu_{13} - \lambda_2} - \frac{\mu_{13}}{\lambda_1 + \mu_{12} + \mu_{13} - \lambda_3})$$

$$\cdot \exp(-(\lambda_1 + \mu_{12} + \mu_{13}) \cdot t) + \frac{\mu_{12}}{\lambda_1 + \mu_{12} + \mu_{13} - \lambda_2}$$

$$\cdot \exp(-\lambda_2 \cdot t) + \frac{\mu_{13}}{\lambda_1 + \mu_{12} + \mu_{13} - \lambda_3} \cdot \exp(-\lambda_3 \cdot t)$$

Both exponents' powers and the coefficients before exponents may be considered to be measured in an experimental research. One can find μ_{12} and μ_{13} using these data. Capture constant is connected with defects parameters in the following way $\frac{\mu_{12,3}}{C_{,3}} = 4 \cdot \pi \cdot R_{2,3} \cdot D_+$, where $R_{2,3}$ are the characteristic defect sizes, C is the defects concentration, D_+ is the positron diffusion coefficient. The type and concentration of defects can be estimated using data of ref [36]*, the positron diffusion coefficient is accepted to be 1 cm²/c in these calculations. The electron density in clusters can't differ more than for 2 times in comparison with electron density in vacancies,

since the lifetime of positrons in vacancies is two times shorter than the lifetime of positrons in a cluster, capture of positrons taking place on the surface of the cluster.

The size of the cluster is accepted to be $1.1R_v$ (R_v is the vacancy radius), ideas of accepting defects sizes ratio for vacancies and clusters will be considered later. All defects are assumed to have spherical shape. Results of calculations for binary alloys are shown in table 7.

We shall estimate vacancies and cluster - like defects concentrations under the following assumptions. Since the precious size of the region of potential irregularity is unknown let's accept the vacancy size equal to the shifted atom size, i.e. to its diameter. Cluster-like generation is considered to be similar to model cluster generated by the static displacements of neighborhood atoms near the doping element along (110) axis, the axis being a face's diagonal of BCC lattice In this case the linear size of the cluster is $1.1\ R_V$, where R_V, is the vacancy radius. Then the number of vacancies in the cluster may be estimated using the formula.

$N = 0.2 \cdot R^3 \cdot 10^{24} = 6$.

Calculation of defects concentration in ternary alloys is made using the same technique as for binary alloys. The calculated results for ternary alloys are shown in table 8.

It follows from data from tables 6, 7and 8 that doping of the alloys by molybdenum and tungsten is i1dentical so the properties are similar to the binary alloy with molybdenum used as a doping element .The only difference is a slight reduction of clusters concentration. The difference of alloys with vanadium is that there are fewer vacancies and the concentration of clusters is slightly higher than in binary alloys. These results also qualitatively agree with the assumption of sphere clusters. But most probably the cluster size must be larger. Really if the assumption of proportionality of the annihilation rate and the electron density means that the cluster volume must be 35/19=1.84 of vacancy size Then the linear distortion must be $(1.841)^{1/3}=1.226$. Such discrepancy may be connected with not account to anisotropy of the distortions (different values of distortions in different crystallography directions) in bcc – lattice [36].

Table 7. *Results of calculations. Relative error is 1%*

Material of the sample	μ_{12}/ 10^8 c^{-1}	μ_{13}/ 10^8 c^{-1}.	C_2/ 10^{15}cm^{-3}	C_3/ 10^{15}cm^{-3}
FeCr	9,33	6.07	2.58	1.53
FeMo	10.3	5.89	2.85	1.48
FeW	9.16	6.49	2.54	1.64

Table 8. *The calculated results for ternary alloys. The relative error of the measurements is 4%*

Alloy	Content of elements, atomic per cent								
	Fe	Cr	Cu	Mn	Mo	Ni	V	W	T^{0}C
FeW	Basic	0.17	0.22	0.14	0	0.12	0	0.83	750
FeMo	Basic	0.16	0.21	0.12	0.82	0.1	0	0.02	750
FeCr	Basic	8.67	0.2	0.23	0.1	0.09	0	0.02	750
All samples were treated by cold deformation and annealed during 30 hours									

3.2. Discussion of the results using the positron capture model

The results obtained may be explained in such a way. It is seen from data shown in table 1 that doping by 8-9% of chromium must result in the rebuilding of short range order types. Maybe doping by molybdenum and tungsten result in the ordering in the same way, but content of the doping elements should be lower. Favorite conditions are created in alloys containing molybdenum for vacancies generation, they are not repulsed to boundaries. That is the reason for observation of increasing of positron capture to vacancies. Tungsten impurity makes greater distortion of the lattice; besides that tungsten atoms preferentially generate complexes in comparison with molybdenum. This may result in the increase of the third (350 ps) and reduction of the second (190 ps) lifetime component.

Generation of complexes of the atom-vacancy type maybe takes place in ternary alloys. Addition of vanadium results in the reduction of vacancies concentration and increase of clusters number. The further increase of clusters doesn't take place since further increase in the number of clusters is not happening, because the accumulation of vacancies is a source for clusters generation.

4. Conclusions and proposals

Thus changes, which take place in binary and ternary Fe-based and Fe-Cr- based alloys, have been investigated by positron annihilation method and it has been shown that at those concentrations of doping elements properties of the alloys must be characterized by stability. Comparing the results with results obtained recently [16-18] one may conclude that these experiments have the advantage of fixed amounts of doping elements

The following proposals should be made in connection with this research.

1. To carry out experiments with samples containing equal amounts of doping additions in order to maintain the more unique interpretation of the experimental data. For example the ternary alloy doped by vanadium has greater amount of impurity in comparison with alloys doped by tungsten and molybdenum.

2. One must exclude the influence of nickel impurity, containing in the alloy doped by vanadium

3. One must solve the problem of comparison of binary alloys, containing different amounts of doping additions. For example chromium content is by an order higher than doping additions of molybdenum and tungsten.

4. It is proposed to carry out experiments with samples having different number of linear dislocations for clearing the nature of cluster – like defects. The samples must be treated by cold deformation.

5. It is necessary to carry out testing measurements for determination of short-range ordering by Mossbauer spectroscopy as had been described in paper [26].

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