



# Mean hyperfine fields at $^{57}\text{Fe}$ in dilute iron-based alloys studied by Mössbauer spectroscopy

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**Abstract.** The room temperature Mössbauer spectra of  $^{57}\text{Fe}$  were measured for numerous dilute iron-based alloys  $\text{Fe}_{1-x}\text{D}_x$  ( $\text{D} = \text{Al}, \text{Co}, \text{Cr}, \text{Mn}, \text{Mo}, \text{Ni}, \text{Os}, \text{Pt}, \text{Re}, \text{Ru}, \text{Ta}, \text{Ti}, \text{V}, \text{W}, \text{Zn}$ ), annealed at 1270 K for 2 h before the measurements. The spectra were analyzed using the Hesse–Rübartsch method in order to determine the mean hyperfine magnetic field  $\langle B \rangle$  at the  $^{57}\text{Fe}$  nuclei as a function of concentration  $x$  of the minority component of the alloy. As the binary alloys are one-phase solid solutions of an element D in iron, a linear relationship between  $\langle B \rangle$  and  $x$  is observed. The result supports the suggestion that Mössbauer spectroscopy is a useful tool for the study of dissolution of different elements in iron.

**Key words:** hyperfine interactions • iron alloys • Mössbauer spectroscopy

## Introduction

Impurity atoms, in iron, have significant influence on the hyperfine field  $B$  at iron nuclei located in the vicinity of the atoms because they modify electron spin and charge densities at the iron nuclei. This phenomenon is easily observed by  $^{57}\text{Fe}$  Mössbauer spectroscopy (MS). However, comparison of data obtained by researchers revealed large discrepancies in values of some parameters derived from the Mössbauer spectra for the same iron systems, such as changes of  $B$  at  $^{57}\text{Fe}$  with one impurity atom in the first ( $\Delta B_1$ ) and the second coordination shells ( $\Delta B_2$ ) of the Mössbauer probe as well as the mean hyperfine field  $\langle B \rangle$  [1–8]. This situation could be explained by different analysis methods of Mössbauer spectra and different procedures of sample preparation applied by various authors. In particular, many of the previous Mössbauer experiments were performed on non-annealed samples [1–3, 5–8]. In view of the fact that frozen-in structural defects (like vacancies for example) existing in as-obtained or mechanically treated ingots could have additional, not-well-determined influence on hyperfine interactions [9], it seems obvious that results obtained by different research studies on corresponding alloys can be compared (similar) only in cases in which well-defined, homogenized and relaxed alloys after a proper annealing process are used. Taking the above discussion into account, we decided to return to the

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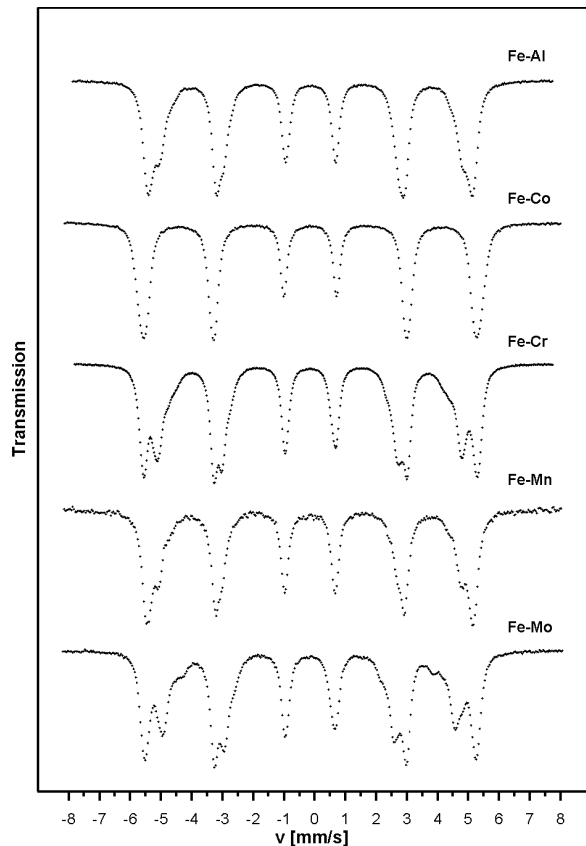
MS measurements concerning hyperfine interactions of iron nuclei in dilute iron-based alloys Fe-D (D = Al, Co, Cr, Mn, Mo, Ni, Os, Pt, Re, Ru, Ta, Ti, V, W, Zn) and collect in one place only those of our results that were obtained for the annealed samples of the aforementioned alloys.

### Experimental details

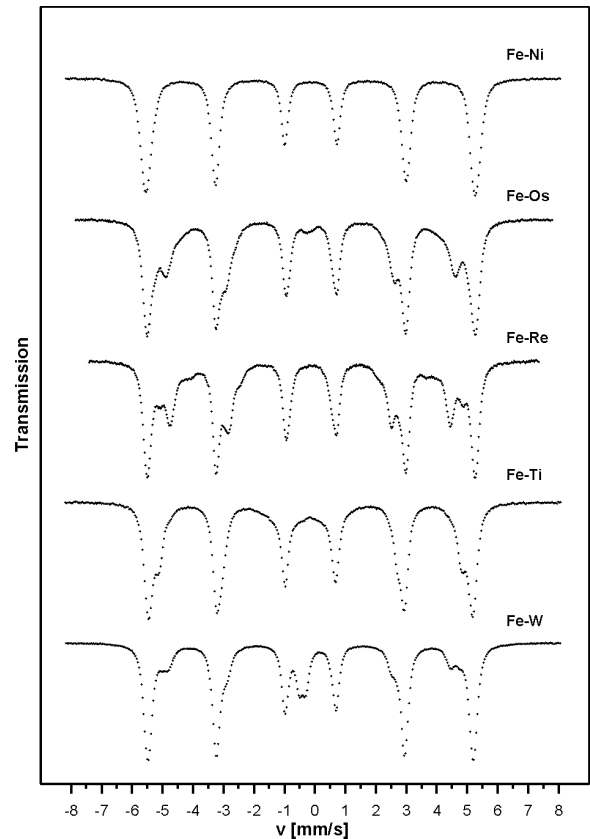
The samples of  $\text{Fe}_{1-x}\text{D}_x$  alloys were melted in an arc furnace under an argon atmosphere and quickly cooled down to the room temperature. The weight losses during the melting process were less than 1%; hence, the compositions of the obtained ingots were close to the nominal ones. The resulting ingots were cold-rolled to the final thickness of about 0.04 mm and thereafter the foils were annealed in the vacuum at 1270 K for 2 h. After that, they were slowly cooled to room temperature for 6 h.

The room temperature measurements of the  $^{57}\text{Fe}$  Mössbauer spectra were performed in transmission geometry by means of a constant-acceleration POLON spectrometer of standard design, using a  $^{57}\text{Co}$ -in-Rh standard source with a full width at half maximum (FWHM) of 0.24 mm/s. Some of the obtained spectra are presented in Figs. 1 and 2.

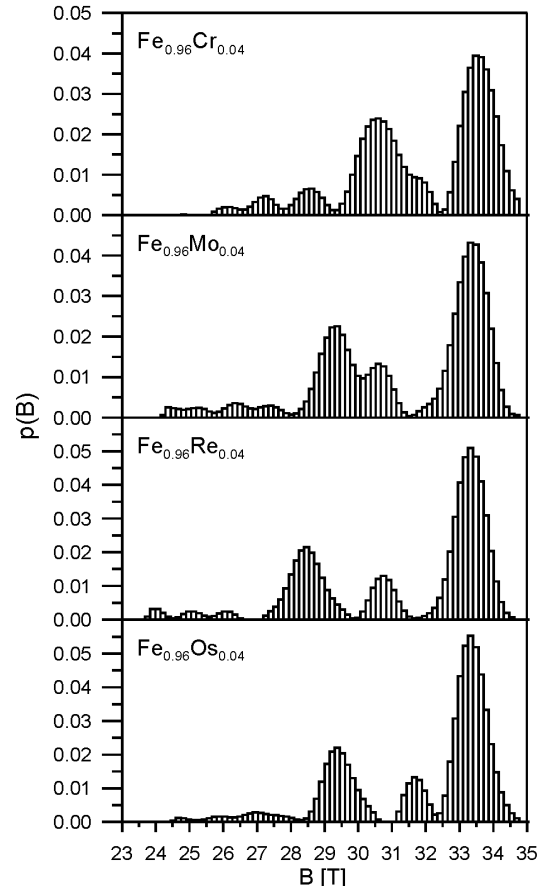
The Mössbauer spectra analysis based on the Hesse-Rübartsch method [10] was made in order to determine the mean hyperfine field  $\langle B \rangle$  at  $^{57}\text{Fe}$  nuclei for the  $\text{Fe}_{1-x}\text{D}_x$  alloys as a function of impurity concentration  $x$  of the element D. Four of the probability distributions of the hyperfine field  $p(B)$



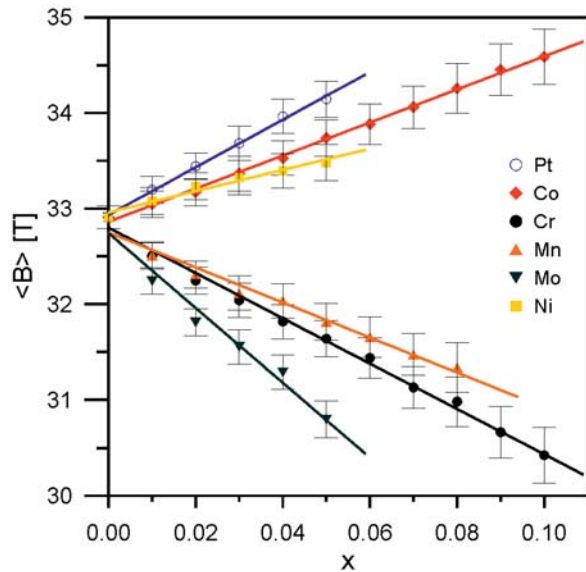
**Fig. 1.** The  $^{57}\text{Fe}$  Mössbauer spectra for the annealed  $\text{Fe}_{0.95}\text{D}_{0.05}$  alloys measured at room temperature.



**Fig. 2.** The  $^{57}\text{Fe}$  Mössbauer spectra for the annealed  $\text{Fe}_{0.95}\text{D}_{0.05}$  alloys measured at room temperature.



**Fig. 3.** The probability distributions of the hyperfine field  $p(B)$  obtained by the Hesse-Rübartsch analysis of the Mössbauer spectra for different iron alloys.



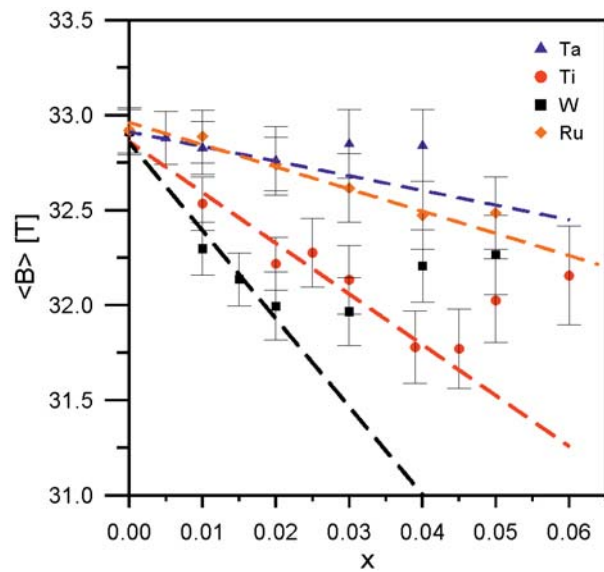
**Fig. 4.** The mean hyperfine fields  $\langle B \rangle$  at  $^{57}\text{Fe}$  in  $\text{Fe}_{1-x}\text{D}_x$  alloys. Solid lines represent values of linear functions  $\langle B \rangle = Ax + B_0$  fitted to the experimental data on  $\langle B \rangle$ .

obtained by the Hesse–Rübartsch method for various Fe-D alloys are presented in Fig. 3.

## Results

It was found that in the case of binary, iron-based alloys with Al, Co, Cr, Mn, Mo, Ni, Os, Pt, Re, V or Zn as a solute, the mean hyperfine magnetic field  $\langle B \rangle$  changes linearly with concentration  $x$  of the impurity (Fig. 4). In other words, the relationship between  $\langle B \rangle$  and  $x$  can be written in the following way:  $\langle B \rangle = Ax + B_0$ , where  $A$  parameter could be interpreted as a total change of the mean hyperfine field due to the corresponding change of the impurity concentration  $d\langle B \rangle/dx$ . The observed proportion of  $\langle B \rangle$  to  $x$  for all studied alloys mentioned earlier speaks in favor of the suggestion that the alloys are one-phase solid solutions of the elements under consideration in iron, and these elements dissolve in iron quite well.

In the case of iron systems with Ru, Ta, Ti and W impurities, the dependence of  $\langle B \rangle$  on impurity concentration  $x$  is more complicated. For very di-



**Fig. 5.** The mean hyperfine fields  $\langle B \rangle$  at  $^{57}\text{Fe}$  in  $\text{Fe}_{1-x}\text{D}_x$  alloys. Dotted lines represent linear function  $\langle B \rangle = Ax + B_0$  fitted to the experimental data on  $\langle B \rangle$  for very diluted alloys.

luted Fe-Ru, Fe-Ta, Fe-Ti and Fe-W alloys, the determined  $\langle B \rangle$  values decrease linearly with  $x$  (Fig. 5). However, in a higher concentration region (for more concentrated systems),  $\langle B \rangle$  tends to increase in a nonlinear way. This fact could be associated with the limited solubility of Ru, Ta, Ti and W in iron [11]. Consequently, the alloys concentrated enough are multiphase, and the solid solution of impurity elements in iron is not the only phase forming the alloys. The concentration  $x$  of these alloys is different from the concentration of impurity elements in iron solid solutions as they are components of the alloys.

The estimated values of the  $d\langle B \rangle/dx$  and  $B_0$  parameters are listed in Tables 1 and 2. Data collected from Ref. [12] are added for comparison. The differences between  $d\langle B \rangle/dx$  values obtained in this work and in Ref. [12] could be attributed to the fact that most of the data collected in Ref. [12] concerned non-annealed samples. This confirms our predictions that the presence of frozen-in structural defects in as-obtained or mechanically treated ingots has additional, not-well-determined influence on hyperfine interactions [9].

**Table 1.** The  $d\langle B \rangle/dx$  and  $B_0$  parameters obtained for a part of the studied binary iron-based systems. Data from Ref. [12] are added for comparison

Impurity	$B_0$ [T]	$d\langle B \rangle/dx$ [T/at.%]	$d\langle B \rangle/dx$ [T/at.%] [12]
Al	32.937(61)	-0.263(17)	-0.209
Co	32.863(15)	0.1728(26)	0.120
Cr	32.801(90)	-0.2368(24)	-0.278
Mn	32.751(49)	-0.183(10)	-0.150
Mo	32.744(99)	-0.391(33)	-0.383
Ni	32.960(32)	0.111(11)	0.160
Os	32.873(33)	-0.255(11)	-0.254
Pt	32.934(20)	0.2489(66)	0.211
Re	32.767(78)	-0.368(26)	-0.373
V	32.945(48)	-0.2679(74)	-0.308
Zn	32.891(30)	-0.051(11)	-0.194

**Table 2.** The  $d\langle B \rangle/dx$  and  $B_0$  parameters, obtained for very diluted Fe-Ru, Fe-Ta, Fe-Ti and Fe-W alloys. Data from Ref. [12] are added for comparison

Impurity	$B_0$ [T]	$d\langle B \rangle/dx$ [T/at.%]	$d\langle B \rangle/dx$ [T/at.%] [12]
Ru	32.961(29)	-0.117(12)	-0.104
Ta	32.912(10)	-0.0771(53)	-
Ti	32.860(69)	-0.267(28)	-0.225
W	32.856(84)	-0.463(63)	-0.391

## Conclusions

The results, for the  $^{57}\text{Fe}$  MS estimation of the mean magnetic hyperfine field at  $^{57}\text{Fe}$  nuclei in binary iron-based alloys  $\text{Fe}_{1-x}\text{D}_x$  with more than a dozen different minority elements D of the alloys, show that the field is simply proportional to concentration  $x$  of the D elements when the alloys are properly annealed, one-phase solid solutions. At the same time, the proportional constant depends on the kind of the solute D and can be helpful in the identification of the solute. On the other hand, a linear relationship between the magnetic field and concentration of the minority elements in the binary iron-based alloys is not observed when the alloys are multiphase systems. Such a situation exists in our case for some iron systems with Ru, Ta, Ti and W. This fact can be used for simple determination of the limits of solubility of unlike elements in iron.

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## References

1. Wertheim, G. K., Jaccarino, V., Wernick, J. H., & Buchanan, D. N. E. (1964). Range of the exchange interaction in iron alloys. *Phys. Rev. Lett.*, *12*, 24–27. DOI: <http://dx.doi.org/10.1103/PhysRevLett.12.24>.
2. Vincze, I., & Campbell, I. A. (1973). Mössbauer measurements in iron base alloys with transition metals. *J. Phys. F*, *3*, 647–663. DOI: 10.1088/0305-4608/3/3/023.
3. Błachowski, A. (2008). Charge and spin density perturbation on iron nuclei by non-magnetic impurities substituted on the iron sites in  $\alpha\text{-Fe}$ . *Acta Phys. Pol. A*, *114*, 1563–1571.
4. Chojcan, J. (2004). A dilute-limit heat of solution of 3d transition metals in iron studied with  $^{57}\text{Fe}$  Mössbauer spectroscopy. *Hyperfine Interact.*, *156/157*, 523–529. DOI: 10.1007/978-1-4020-2852-6\_76.
5. Błachowski, A., Ruebenbauer, K., & Żukrowski, J. (2004). Charge and spin density perturbation on iron atom due to osmium impurity in metallic iron. *Nukleonika*, *49*(Suppl. 3), S67–S70.
6. Błachowski, A., Ruebenbauer, K., & Żukrowski, J. (2006). Spin- and charge-density waves around Ru impurities in  $\alpha\text{-Fe}$  alloys studied by  $^{57}\text{Fe}$  Mössbauer spectroscopy. *Phys. Rev. B*, *73*, 104423. DOI: 10.1103/PhysRevB.73.104423.
7. Błachowski, A., Ruebenbauer, K., Żukrowski, J., & Przewoźnik, J. (2009). Spin and charge density on iron nuclei in the BCC Fe-Mo alloys studied by  $^{57}\text{Fe}$  Mössbauer spectroscopy. *J. Alloy. Compd.*, *482*, 23–27. DOI: 10.1016/j.jallcom.2009.03.177.
8. Błachowski, A., Ruebenbauer, K., Żukrowski, J., Przewoźnik, J., Marzec, J., & Rakowska, A. (2011). Spin- and charge density perturbations and short-range order in Fe-Cu and Fe-Zn BCC alloys: A Mössbauer study. *J. Phys. Chem. Solids*, *72*, 1537–1542. DOI: 10.1016/j.jpcs.2011.08.032.
9. Idczak, R., Konieczny, R., & Chojcan, J. (2014). A study of defects in iron-based binary alloys by the Mössbauer and positron annihilation spectroscopies. *J. App. Phys.*, *115*, 103513. DOI: 10.1063/1.4868471.
10. Hesse, J., & Rübartsch, A. (1974). Model independent evaluation of overlapped Mössbauer spectra. *J. Phys. E: Sci. Instrum.*, *7*, 526–532. DOI: 10.1088/0022-3735/7/7/012.
11. ASM. (1992). *ASM Handbook. Volume 3: Alloy phase diagrams*. Materials Park, OH: ASM International.
12. Błachowski, A., Wdowik, U. D., & Ruebenbauer, K. (2009). Volume effect and electron band modification in  $\alpha\text{-Fe}$  by substitutional impurities. *J. Alloy. Compd.*, *485*, 36–40. DOI: 10.1016/j.jallcom.2009.06.076.