THE ⁵⁷Fe MÖSSBAUER ISOMER SHIFT IN INTERMETALLIC COMPOUNDS OF IRON

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Received 13 September 1985

A large number of intermetallic compounds in which Fe is combined with transition metals (Ti, Zr, Hf, Th, V, Nb, Ta, Mo) and s,p metals or metalloids (B, Al, Ga, Si, Ge, Sn, As, Sb) were investigated by means of X-ray diffraction and ⁵⁷Fe Mössbauer spectroscopy. The lattice constants of all compounds were determined. The isomer shift in all these materials was analysed in terms of the model of Miedema and Van der Woude by the same empirical method used earlier in investigations on amorphous Fe-base alloys.

1. Introduction

Amorphous alloys have the favourable property of a large compositional freedom, which makes it possible to prepare single phase materials over often quite extended concentration ranges. In a previous investigation we used this compositional freedom of amorphous $A_{1-r}Fe_r$ alloys to study the ⁵⁷Fe isomer shift systematically as a function of Fe concentration (x) and as a function of the second component A [1]. We found that the ⁵⁷Fe isomer shift in all these materials can satisfactorily be described in terms of the model of Miedema and Van der Woude [2] using a single set of two universal parameters for which we derived empirical values. In the study reported here we investigated in how far this analysis can be applied to the rather large class of intermetallic compounds of Fe.

Several intermetallic compounds of Fe considered in this study had already been investigated by ⁵⁷Fe Mössbauer spectroscopy before. However, in many cases the interest was in hyperfine fields and less in isomer shifts (IS). Values of the isomer shifts had been reported frequently without further specification as to the reference material. For this reason and also with a view to reduce the experimental error associated with differences in calibration and fitting of the spectra within a given series of A-Fe compounds, we prepared most of the compounds again and measured their ⁵⁷Fe Mössbauer spectrum. Since our main interest was in obtaining an accurate value of IS at room temperature we restricted ourselves as far as possible to materials which are still paramagnetic at room temperature, and hence do not show magnetic splitting of the Mössbauer lines.

2. Experimental procedures and results

The intermetallic compounds were prepared by arc melting from pure starting materials of at least 99.9% purity. Most of the samples were vacuum annealed after arc melting and some of them were quenched in water after the annealing treatment. The composition of the samples investigated and the corresponding annealing treatment are indicated in tables I and II. The Fe-As compounds were not prepared by arc melting, owing to the high vaporization rate of As. In these cases powdered As and Fe were thoroughly mixed and heated in an evacuated quartz vessel, first at 700°C and subsequently at

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Preparatory conditions (annealing treatment in hours, annealing temperature in $^{\circ}$ C; q means quenched after annealing) and crystallographic properties of several intermetallic compounds of Fe with transition metals

Compound	Heat treatment	Structure	Lattice constants (nm)
FeTi	melt spun ribbon	CsCl (cub)	a = 0.2972
Fe ₂ Ti	340 h 1000°	MgCu ₂ (hex)	$a = 0.4757, \ c = 0.7829$
FeZr ₃	650 h 800°	Re ₃ B (or)	a = 0.3324, b = 1.099, c = 0.8810
FeZr ₂	500 h 875° (q)	$CuAl_2$ (tetr)	$a = 0.6372, \ c = 0.5583$
Fe ₂ Zr	50 h 1000°	MgCu ₂ (cub)	a = 0.7074
FeHf ₂	as-cast	Ti ₂ Ni (cub)	a = 1.2038
Fe ₃ Th	700 h 1100°	PuNi ₃ (rhomb)	a = 0.5213, c = 2.511
Fe ₅ Th	700 h 1150° (q)	CaCu ₅ (hex)	$a = 0.5110, \ c = 0.4054$
V0 60 Fe0 40	450 h 900° (q)	FeCr (tetr)	a = 0.9028, c = 0.4653
V _{0.50} Fe _{0.50}	700 h 900° (q)	FeCr (tetr)	$a = 0.8965, \ c = 0.4633$
$V_{0.40}Fe_{0.60}$	700 h 900° (q)	FeCr (tetr)	a = 0.8900, c = 0.4606
V0 36Fe0 64	700 h 600° (q)	FeCr (tetr)	a = 0.8888, c = 0.4608
Fe ₇ Nb ₆	170 h 1050°	Fe ₇ W ₆ (rhomb)	$a = 0.4927, \ c = 2.6776$
Fe ₂ Nb	340 h 1000°	MgZn ₂ (hex)	$a = 0.4835, \ c = 0.7881$
FeTa	as-cast	Fe ₇ W ₆ (rhomb)	a = 0.4923, c = 2.7006
Fe ₂ Ta	170 h1050°	MgZn ₂ (hex)	a = 0.4828, c = 0.7878
Fe ₅ Mo ₃	3 h 1250° (q)	Fe ₅ Mo ₃ (rhomb)	a = 1.0956, c = 1.9353
Fe7M06	as-cast	Fe ₇ W ₆ (rhomb)	
Fe ₇ W ₆	1000 h 1150° (q)	Fe ₇ W ₆ (rhomb)	

Table II

Table I

Preparatory conditions (annealing treatment in hours, annealing temperature in °C) and crystallographic properties of several intermetallic compounds of Fe with s,p elements

Compound	Heat treatment	Structure	Lattice constants
FeAl ₃	50 h 1000° (q)	FeAl ₃ (monocl.)	a = 1.549, b = 0.808, c = 1.248 $\beta = 107.40^{\circ}$
Fe ₂ Al ₅	100 h 900°	Fe ₂ Al ₅ (o.rh)	$a = 0.7644, \ b = 0.6411, \ c = 0.4220$
FeAl ₂	100 h 900°	$FeAl_2$ (tricl.)	a = 0.488, b = 0.646, c = 0.880 $\alpha = 91.70^{\circ}, \beta = 73.3^{\circ}, \gamma = 96.90^{\circ}$
FeAl	50 h 1000°	CsCl (cubic)	a = 0.2907
FeGa ₃	100 h 750°	FeGa ₃ (tetr)	a = 0.6256, c = 0.6560
FeSi ₂	300 h 1000° (q)	FeSi ₂ (tetr)	a = 0.2696, c = 0.5142
FeSi	as-cast	FeSi (cub)	a = 0.4487
FeGe ₂	100 h 750°	$CuAl_2$ (tetr)	a = 0.5905, c = 0.4958
FeGe	700 h 630°	CoSn (hex)	a = 0.5000, c = 0.4054
Fe ₂ As	20 h 800°	Cu ₂ Sb (tetr)	$a = 0.3634, \ c = 0.5985$
FeSn ₂	1000 h 480°	$CuAl_2$ (tetr)	a = 0.6542, c = 0.5326
FeSn	100 h 440°	CoSn (hex)	$a = 0.5297, \ c = 0.4481$
FeAs ₂	20 h 800°	FeS ₂ (o.rh)	a = 0.5300, b = 0.5985, c = 0.2882
FeAs	20 h 800°	MnP (o.rh)	$a = 0.5440, \ b = 0.3370, \ c = 0.6030$
FeSb ₂	250 h 580° (q)	FeS ₂ (o.rh)	$a = 0.5841, \ b = 0.6552, \ c = 0.3207$
Fe0.56Sb0.44	1000 h 720° (q)	NiAs (hex)	$a = 0.4124, \ c = 0.5173$

800°C, followed by annealing for 20 h at 800°C.

All samples investigated were examined by means of X-ray diffraction using a Philips X-ray



Fig. 1. 57 Fe Mössbauer spectra of FeTi (top) and FeGe₂ (bottom) at room temperature.

Table III

Average isomer shift relative to α -Fe (δ IS), and hyperfine field (H_{eff}) observed in various intermetallic compounds at room temperature. The quantity f_A^{Fe} represents the fractional area of contact between Fe atoms and A atoms (see main text)

Compound	δ IS (mm/s)	$H_{\rm eff}$ (T)	$f_{ m A}^{ m Fe}$	Ref.
FeTi	- 0.154	0	0.84	this work
Fe ₂ Ti	-0.270	0	0.57	this work
FeZr ₃	- 0.319	0	0.97	[5]
FeZr ₂	-0.321	0	0.96	151
Fe ₂ Zr	- 0.179	19.6	0.65	[6]
FeHf ₂	-0.14	0	0.96	this work
Fe ₃ Th ₇	-0.22	0	0.98	[7]
Fe ₃ Th	- 0.17	16.5	0.58	[8]
Fe ₅ Th	-0.13	20.2	0.38	[8]
$V_{0.60}$ Fe _{0.40}	-0.25	0	0.90	[9]
$V_{0.50}$ Fe _{0.50}	- 0.19	0	0.78	[9]
V _{0.40} Fe _{0.60}	-0.17	0	0.63	[9]
V _{0.36} Fe _{0.64}	-0.19	0	0.57	[9]
Fe7Nb6	- 0.224	0	0.79	this work
Fe ₂ Nb	- 0.245	0	0.57	this work
FeTa	- 0.20	0	0.84	this work
Fe ₂ Ta	-0.242	0	0.57	this work
Fe5Mo3	-0.211	0	0.64	this work
Fe7M06	-0.238	0	0.76	this work
Fe ₇ W ₆	- 0.236	0	0.76	this work

Table IV

Average isomer shift relative to α -Fe (δ IS) and hyperfine field (H_{eff}) observed in the course of the present investigation in various intermetallic compounds at room temperature. The quantity f_A^{Fe} represents the fractional area of contact between Fe atoms and A atoms (see main text)

Compound	δIS (mm/s)	$H_{\rm eff}$ (T)	$f_{\rm A}^{ m Fe}$
FeAl ₃	0.21	0	0.96
Fe ₂ Al ₅	0.24	0	0.96
FeAl ₂	0.23	0	0.95
FeAl	0.26	0	0.83
FeGa ₃	0.28	0	0.96
FeSi ₂	0.24	0	0.95
FeSi	0.28	0	0.79
FeGe ₂	0.31	0	0.95
FeGe	0.02	12.2	0.83
FeSn ₂	0.52	11.2	0.96
FeSn	0.43	11.4	0.91
	0.39	10.3	
FeAs ₂	0.29	0	0.96
FeAs	0.47	0	0.87
Fe ₂ As	0.57	11.4	0.70
	0.37	9.2	0.60
FeSb ₂	0.46	0	0.96
Fe _{0.56} Sb _{0.44}	0.42	0	0.87

powder diffractometer (type PW 1050/25). We used CuK_{α} radiation in conjunction with a graphite crystal monochromator. The crystal structures found for the samples are in agreement with those reported in the literature [3, 4]. The lattice constants of all compounds were determined and are included in tables I and II. They are in satisfactory agreement with literature data [3, 4].

The ⁵⁷Fe Mössbauer spectra were obtained by means of a standard constant-acceleration type spectrometer equipped with a ⁵⁷Co-Rh source. Some typical examples of Mössbauer spectra are shown in fig. 1. Values of the isomer shift (relative to α -Fe) and hyperfine field (H_{eff}) are listed for all compounds in tables III and IV.

3. Discussion

As mentioned in the Introduction, the authors had earlier reported a successful analysis of the ⁵⁷Fe Mössbauer isomer shift for a variety of different amorphous Fe alloys on the basis of the model of Miedema and Van der Woude [1]. In this model the strain-free dilute limit δIS_{max} of the isomer shift IS relative to α -Fe in an alloy $A_{1-x}Fe_x$ is given by the expression [2]

$$\delta IS_{max} = P'(\phi_A^* - \phi_{Fe}^*) + Q'(n_{ws}^A - n_{ws}^{Fe})/n_{ws}^{Fe}, \qquad (1)$$

where ϕ^* and n_{ws} represent values for the electronegativities and electron densities at the Wigner-Seitz cell boundaries, respectively. For amorphous Fe alloys the constants P' and Q' were determined empirically, and since the values for ϕ^* and n_{ws} have been tabulated for all metals [10] it is possible to predict the strain-free dilute limit for any alloy by means of the relation

$$\delta IS_{max} = 0.75 \Delta \phi^* - 1.65 \Delta n_{ws} / n_{ws}^{Fe} .$$
⁽²⁾

In amorphous alloys $A_{1-x}Fe_x$ of finite Fe concentration x the IS shift is expected to vary linearly with the relative area of contact of a given Fe atom with A neighbours. This fractional area of contact can be estimated by weighting the actual atomic fraction with the cross-sectional areas of Fe and A. The corresponding effective concentrations are given by

$$\bar{x} = x V_{\rm Fe}^{2/3} \left[x V_{\rm Fe}^{2/3} + (1-x) V_{\rm A}^{2/3} \right]^{-1}.$$
 (3)

In this expression the cross-sectional areas of the A and Fe atoms were taken to be proportional to $V^{2/3}$, where V is the atomic volume. The concentration dependence of δIS is then given as $\delta IS_x = (1 - \bar{x}) \delta IS_{max}$.

Intermetallic compounds differ from amorphous alloys in so far as the coordination of the Fe atoms can no longer be described as a more or less statistical distribution of A and Fe atoms in the near-neighbour shell. The A atoms show a strong preference to be nearest neighbours to the Fe atoms. For this reason the fractional area of contact of the Fe atoms with A atoms in intermetallic compounds of nominal Fe concentration x is larger than \bar{x} in eq. (3). It can be estimated by means of the expression [2, 10]

$$f_{\rm Fe}^{\rm A} = (1 - \bar{x}) [1 + 8\bar{x}^2(1 - \bar{x})^2], \qquad (4)$$

where \bar{x} is given by eq. (3). The concentration dependence of the isomer shift in intermetallic compounds is now expressed as

$$\delta \mathrm{IS}_{x} = f_{\mathrm{A}}^{\mathrm{Fe}} \delta \mathrm{IS}_{\mathrm{max}} \,. \tag{5}$$

In dilute crystalline alloys $A_{1-x}Fe_x$ ($x \le 0.01$) the Fe atoms occupy atomic positions in the lattice of the host metal. This results in a size mismatch when the A atoms are much smaller or larger than the Fe atoms, and consequently leads to an additional isomer shift contribution. The size mismatch contribution is absent in amorphous alloys and intermetallic compounds where Fe and A atoms are free to choose their own position. For this reason both classes of materials are expected to behave in an analogous way. In other words, also in intermetallic compounds it is reasonable to obtain the strain-free dilute limit δIS_{max} by plotting the isomer shift observed in the various compounds versus the fractional area of contact and extrapolate to infinite solution $(f_{A}^{Fe} = 1 \text{ in eq. (5)}).$

Experimental results for intermetallic compounds in which Fe is combined with other transition metals are given in figs. 2 and 3. In the cases Zr-Fe and Th-Fe in particular, the straight line (according to eq. (5)) can be drawn rather unambiguously. The situation is less favourable in the case of Hf-Fe. This is due in part to the fact that there are only two compounds in the Hf-Fe system. Of these two compounds, Hf₂Fe and HfFe₂, the latter is not suitable for the determination of IS by means of ⁵⁷Fe Mössbauer spectroscopy since it gives rise to a polymorphic transformation from cubic to hexagonal, both phases being in addition strongly ferromagnetic and having more than one inequivalent Fe site. Inspection of fig. 3 shows that some ambiguity as to fixing the straight line is present also in the cases of Nb-Fe and Ta-Fe.

Experimental results for compounds of Fe with polyvalent s,p metals are shown in figs. 4–6. It can be seen in fig. 4 that the situation is satisfactory in compounds of Fe with elements of the third column of the Periodic Table, while figs. 5 and 6 show that the scatter around the straight lines is somewhat worse in compounds



Fig. 2. ⁵⁷Fe Mössbauer isomer shift in various intermetallic compounds of Fe with transition metals, plotted versus the fractional area of contact between Fe atoms and nonmagnetic A atoms (f_A^{Fe}).



Fig. 3. ⁵⁷Fe Mössbauer isomer shift in various intermetallic compounds of Fe with transition metals, plotted versus the fractional area of contact between Fe atoms and nonmagnetic A atoms (f_{A}^{Fe}) .



Fig. 4. ⁵⁷Fe Mössbauer isomer shift in various intermetallic compounds of Fe with s,p metals, plotted versus the fractional area of contact between Fe atoms and nonmagnetic A atoms (f_A^{Fe}) . The data of B-Fe are taken from Chien et al. [11].



Fig. 5. ⁵⁷Fe Mössbauer isomer shift in various intermetallic compounds of Fe with metalloids, plotted versus the fractional area of contact between Fe atoms and nonmagnetic A atoms (f_A^{Fe}) .



Fig. 6. ⁵⁷Fe Mössbauer isomer shift in various intermetallic compounds of Fe with As and Sb, plotted versus the fractional area of contact between Fe atoms and non-magnetic A atoms (f_A^{Fe}) .

Table V

Strain-free dilute limit (δIS_{max}) of the ⁵⁷Fe isomer shift in (mm/s) at room temperature in intermetallic compounds of Fe with various other metals or metalloids (A). The ratio $(\Delta n/n)/\Delta \phi^*$ was calculated by means of the values listed by Niessen et al. [10] for the electronegativities (ϕ^*) and electron densities at the atomic cell boundaries (n_{ws}) of Fe and A elements

А	δIS _{max}	$\left(\frac{\Delta n}{n}\right) / \Delta \phi$	$\delta \mathrm{IS}_{\mathrm{max}}/\Delta \phi$	
w	- 0.32	- 0.54	2.46	
Mo	- 0.32	0.0	1.14	
V	- 0.27	0.29	0.40	
Nb	-0.32	0.25	0.36	
Та	- 0.30	0.25	0.34	
Ti	- 0.28	0.33	0.25	
Zr	- 0.35	0.33	0.23	
Hf	- 0.15	0.34	0.11	
Th	-0.21	0.38	0.13	
В	0.37	- 0.09	+ 1.00	
A1	0.29	0.71	- 0.40	
Ga	0.28	0.72	- 0.34	
С	0.80	0.19	0.63	
Si	0.30	1.70	- 1.30	
Ge	0.32	1.42	-0.84	
Sn	0.48	0.83	- 0.62	
As	0.42	3.55	- 3.23	
Sb	0.49	1.21	- 0.92	

with elements of the fourth and fifth columns of the Periodic Table. In the system As-Fe in particular, it seems impossible to draw a straight line which could represent eq. (5). From the three δ IS values listed, those of the paramagnetic compounds FeAs and FeAs₂ are the more reliable ones. Since $T_N = 350$ K for Fe₂As the Mössbauer spectrum of this compound shows an incomplete Zeeman splitting and δ IS is less accurately determined. For this reason we have drawn the straight line to fit the data of only the two former compounds.

The experimental values of δIS_{max} obtained by extrapolation in figs. 2–6 have been listed in table V. These values of δIS_{max} have been used for determining P' and Q'. For this purpose we have plotted $\delta IS_{max}/\Delta \phi^*$ versus the corresponding values of $(\Delta n/n)/\Delta \phi^*$. According to eq. (1) a straight line is expected and the results shown in fig. 7 for compounds of Fe with other transition metals are quite convincing. From the intercept on the vertical axis and from the slope of the straight line in fig. 7 one may derive the values of constants



Fig. 7. Plot of experimental values of δIS_{max} normalized with the corresponding electronegativity difference $\Delta \phi^* = \phi_A^* - \phi_{Fe}^*$ versus the ratio $\Delta n/n(\Delta \phi^*)^{-1}$. The electronegativity differences ($\Delta \phi^*$) and difference in electron density at the atomic cell boundaries (Δn) pertain to combinations of Fe with other transition metals.

P' and Q' which are equal to P' = 1.08 and Q' = -2.51.

Arguments were presented by Miedema and Van der Woude [2] to show that eq. (1) is insufficient to describe the isomer shift in alloys or compounds where a transition metal is combined with an s,p metal. Owing to the hybridization between the d electrons of the hybridization between the d electrons of the former component with the s,p electrons of the latter an additional parameter (R') has to be included in eq. (1). Inspection of the plot made for combinations of Fe with s,p metals in fig. 8 show that the agreement with the relation

$$\delta \text{IS}_{\text{max}} = 1.08 \Delta \phi^* - 2.51 \Delta n/n \tag{6}$$

obtained from the previous plot and reproduced here as the straight line is less satisfactory. The differences between the experimental data for $\delta IS_{max}/\Delta \phi^*$ and the corresponding values of the straight line represented by eq. (6) have been listed as $\Delta (\delta IS_{max}/\Delta \phi^*)$ in table VI. From these values one may obtain the excess isomer shift due to the R' term after multiplying with $\Delta \phi^*$.



Fig. 8. Plot of experimental values of δIS_{max} normalized with the corresponding electronegativity difference $\Delta \phi^* = \phi_A^* - \phi_{Fe}^*$ versus the ratio $\Delta n/n(\Delta \phi^*)^{-1}$. The electronegativity differences ($\Delta \phi^*$) and difference in electron density at the atomic cell boundaries (Δn) pertain to combinations of Fe with s,p-metals. In order to make contact with the data given in fig. 7 we included the data points pertaining to W and Mo.

Table VI

Excess values of $\delta IS_{max}/\Delta \phi^*$ (in mm/s) derived from the plot shown in fig. 8. The values of $\Delta(\delta IS_{max})$ represent the excess isomer shift in (mm/s) originating from the additional R' term which has to be included in eq. (1) when Fe is combined with s, p elements

A	$\Delta\left(\frac{\delta \mathrm{IS}_{\mathrm{max}}}{\Delta \phi^*}\right)$	$\Delta(\delta IS_{max})$
В	- 0.30	- 0.11
Al	+ 0.26	- 0.19
Ga	+ 0.37	- 0.31
С	0.00	0.00
Si	+ 1.90	-0.44
Ge	+ 1.60	-0.61
Sn	+ 0.40	-0.31
As	+ 4.70	- 0.61
Sb	+ 1.00	- 0.53

The values of the excess isomer shift $\Delta(\delta IS_{max})$ have been listed in the last column of table VI. It will be clear that any experimental inaccuracies associated with difficulties in the determination of δIS_{max} in systems like Fe-As (fig. 6) will eventually lead to a non-zero value of R' even in cases where such a term is absent or is small. Nevertheless, it is significant that no such term is present in systems of Fe with other transition metals (see fig. 7) while there is a fairly large R' term in some systems of Fe with s,p elements. Inspection of the values listed in the last column of table VI furthermore shows that there is a tendency of R' to increase as the total number of electrons of the s,p element becomes larger.

4. Concluding remarks

The analysis of ⁵⁷Fe isomer shifts in terms of the Miedema–Van der Woude model, made previously for amorphous $A_{1-x}Fe_x$ alloys, has been extended to intermetallic compounds of Fe. The results of this analysis have shown that the parameters P' and Q' determined empirically for amorphous Fe alloys (P' = 0.75; Q' = -1.65) are somewhat different for Fe-base intermetallics (P' = 1.08; Q' = -2.51). From this result and from the fact that slightly different values for P' and Q' were also found for Fe-rich crystalline alloys (P' between 0.71 and 0.90; Q' between -1.50 and -1.85) by Dubiel and Zinn [13], one may conclude that the values of P' and Q' depend somewhat on the nature of the environment of the Fe atoms. This affects primarily the Q' term which is a measure of the intra-atomic redistribution of s and d electrons of the Fe atoms. Apparently this quantity is somewhat sensitive to the local symmetry of the environment of the Fe atoms. This symmetry is low in amorphous and crystalline *alloys* but is high in intermetallic *compounds*.

We found that the accuracy of the model predictions is somewhat less in intermetallics than in amorphous alloys. This is true for intermetallic compounds of Fe with metalloids like As in particular. However unsatisfactory in some cases in a quantitative sense, the model predicts correctly that the isomer shift (relative to α -Fe) in intermetallic compounds is negative when Fe is combined with other transition metals (Ti, Zr, Hf, Th, V, Nb, Ta, Mo, W) but is positive when Fe is combined with s,p metals or metalloids (B, Al, Ga, C, Si, Ge, Sn, As, Sb).

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