

Effect of substitutions on 3d magnetism in $Gd_2Fe_{14-x}M_xC$ compounds, with $M = Ni, Si, Cu$ or V

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Abstract

Results of magnetic measurements and ^{57}Fe Mössbauer spectroscopy performed on $Gd_2Fe_{14-x}M_xC$ compounds, with $M = Ni, Si, Cu$ or V are presented. As M is substituted for Fe , the six crystallographically inequivalent iron sites split into seven inequivalent sites for $M = Si, Cu, V$, or eight for $M = Ni$. The analysis of the hyperfine fields and relative intensities supports a preferential distribution of the substitutional elements on the Fe lattice sites. The effects of the substitutional elements on Curie temperatures and anisotropy fields as well as on Fe hyperfine parameters are discussed on the grounds of the preferential Fe site occupancy.

Keywords: Intermetallic compounds; Magnetic interactions; Magnetocrystalline anisotropy; ^{57}Fe Mössbauer spectroscopy; Preferential substitutions

1. Introduction

Extensive research work has been devoted to the study of the intrinsic magnetic properties of $R_2Fe_{14}X$ phases, where X stands for B or C , owing to the large interest they present as hard magnetic materials [1–4]. While the boron compounds have higher Curie temperatures and saturation magnetizations, the corresponding carbides are attractive owing to the higher magnetocrystalline anisotropy and simpler technological route for the manufacture of permanent magnets. Both series of compounds crystallize in a tetragonal lattice having the symmetry of the $P4_2/mnm$ space group. In the unit cell there are two crystallographically inequivalent R sites ($4f$ and $4g$), six inequivalent iron sites ($16k_1, 16k_2, 8j_1, 8j_2, 4e$ and $4c$) and one type of site ($4g$) for B (or C) atoms. The differences between the magnetic properties of carbon and those of boron compounds are mainly attributed to the effects of different interatomic distances and crystalline lattice constants in the two series. However, the extra charge contribution of C relative to B also affects the crystalline electrostatic field and the electronic charge density. Whereas the a constants have approximately the same value in the boron and corresponding carbon

compounds, the values of the c constants are smaller by 1–2% in carbides, e.g. c/a ratio has the value 1.375 in the case of $Gd_2Fe_{14}B$ but only 1.350 in case of $Gd_2Fe_{14}C$ [1]. It was also suggested that the smaller Fe – Fe interatomic distances in the carbides compared with the borides may lead to different substitutional properties of the compounds [5].

In this paper we present results of magnetic measurements and ^{57}Fe Mössbauer spectroscopy performed on $Gd_2Fe_{14-x}M_xC$ systems, with $M = Ni, Si, Cu$ or V . The site distributions of the substitutional elements are derived and effects of the substitutions on the local hyperfine parameters, as well as on the Curie temperatures and magnetocrystalline anisotropy, are discussed. Some results of structural analysis and magnetic measurements performed on the same samples have already been reported [6,7], as well as preliminary data obtained by ^{57}Fe Mössbauer spectroscopy [8]. Previously, comprehensive analyses of the Fe hyperfine parameters in $R_2Fe_{14}C$ compounds were reported in Refs. [9,10].

2. Experimental

The samples were prepared by induction melting of high purity constituent elements, in purified argon

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atmosphere, in a two-step process. Firstly, an Fe–C (8 at.%) solid solution was prepared. Then, amounts of this prealloy, in addition with iron, gadolinium and substitutional elements were melted to yield the compounds with the desired stoichiometries. The buttons were sealed in quartz tubes in high vacuum and annealed at 1040°C for 2 weeks. This treatment was followed by rapid cooling.

The spontaneous magnetizations $M_s(T)$ were derived from the magnetic isotherms $M(H)$ measured in magnetic fields up to 7 T in the temperature range 5 K up to Curie points. In order to determine the anisotropy fields, cube-shaped samples of field-aligned powders in wax were prepared. The anisotropy fields were taken as the intersection points of the magnetization curves measured with the direction of alignment parallel and respectively perpendicular to the direction of an external magnetic field, up to 1.8 T and between 77 and 300 K.

The Mössbauer measurements were performed at room temperature by using a constant-acceleration Elron spectrometer, with $\text{Rh-}^{57}\text{Co}$ as a source and calibrated relative to $\alpha\text{-Fe}$ foil. Mössbauer absorbers of ca. 35 mg cm^{-1} effective thickness were prepared from fine ground powders with an average grain size of about 30 μm . The analysis of the experimental spectra was carried out by considering five parameters per sextet: hyperfine field H_{hf} , quadrupole splitting QS, isomer shift IS, linewidth Γ , and relative intensity I . The line intensities within a sextet were constrained to the ratio 3:2:1:1, thus accounting for a slight texture of the samples. The hyperfine parameters were determined within the estimated errors of ± 4.0 kOe for the hyperfine field and ± 0.05 mm s^{-1} for the QS and IS respectively. In our fits, the linewidth proved to be constant, $\Gamma = 0.35$ mm s^{-1} . The IS values are given relative to $\alpha\text{-Fe}$ foil.

X-ray diffraction and thermomagnetic analysis showed the formation of the main 2:14:C phase for $x < 1.5$ in the case of $M = \text{Ni, Si}$ and for $x < 1.0$ in case

of $M = \text{Cu, V}$. These limits of solubility of the substitutional elements are lower than those formerly established in the case of the borides and render some inconveniences in the determination of the preferential occupancies of iron sites. Larger amounts of substitutional elements would be preferable for this purpose. Also, the phase analysis showed that some of the samples contained small quantities of $\text{Gd}_2\text{Fe}_{17}\text{C}_y$ phases, with $y < 0.5$, besides the main phase. The lattice parameters are listed in Table 1.

3. Magnetic results

$\text{Gd}_2\text{Fe}_{14-x}\text{M}_x\text{C}$ compounds are ferrimagnetically ordered [6]. Using the molecular field approximation, the $M_s(T)$ curves were fitted according to the relation $M_s(T) = M_{\text{Fe}}(T) - M_{\text{Gd}}(T)$, where sublattice magnetizations are expressed in terms of the zero-temperature magnetizations and Brillouin function-type dependences on temperature [11]. As a result, the molecular field coefficients and temperature dependences of sublattice magnetizations were calculated. The values of the mean iron moments M_{Fe} at 5 K and 293 K and the Curie temperatures of the compounds are listed in Table 2. The mean iron moments decrease with increasing concentration of the substitutional element in the sequence Cu, Ni, Si, V. The Curie temperature variations can be correlated with the well-known dependence on interatomic distances of Fe–Fe exchange interactions, as a result of the lattice constants variations, in the case of “ $M = \text{Ni}$ ”, Cu and V. However, there is an inconsistency concerning this correlation in the case of Si, as already determined in the $\text{R}_2(\text{Fe, Si})_{14}\text{B}$ [12] and $\text{R}_2(\text{Fe, Si})_{17}$ [13,14] systems.

Also, the values of the molecular fields $H_{m,i}(T)$ acting on iron and gadolinium ions respectively, were derived by using the molecular field coefficients. Their

Table 1
Lattice constants of $\text{Gd}_2\text{Fe}_{14-x}\text{M}_x\text{C}$ compounds

Compound	a (Å)	c (Å)	c/a
$\text{Gd}_2\text{Fe}_{14}\text{C}$	8.807	11.887	1.350
$\text{Gd}_2\text{Fe}_{13.5}\text{Ni}_{0.5}\text{C}$	8.808	11.916	1.353
$\text{Gd}_2\text{GFe}_{13}\text{NiC}$	8.810	11.930	1.354
$\text{Gd}_2\text{Fe}_{12.5}\text{Ni}_{1.5}\text{C}$	8.812	11.942	1.355
$\text{Gd}_2\text{Fe}_{13.5}\text{Si}_{0.5}\text{C}$	8.790	11.887	1.352
$\text{Gd}_2\text{Fe}_{13}\text{SiC}$	8.776	11.874	1.353
$\text{Gd}_2\text{Fe}_{12.5}\text{Si}_{1.5}\text{C}$	8.762	11.868	1.354
$\text{Gd}_2\text{Fe}_{13.5}\text{Cu}_{0.5}\text{C}$	8.817	11.952	1.355
$\text{Gd}_2\text{Fe}_{13}\text{CuC}$	8.825	12.000	1.360
$\text{Gd}_2\text{Fe}_{13.5}\text{V}_{0.5}\text{C}$	8.787	11.874	1.351
$\text{Gd}_2\text{Fe}_{13}\text{VC}$	8.772	11.866	1.353

Table 2
Magnetic data of $\text{Gd}_2\text{Fe}_{14-x}\text{M}_x\text{C}$ compounds

Compound	M_{Fe} (μ_{B} /atom)		T_c (K)
	5 K	300 K	
$\text{Gd}_2\text{Fe}_{14}\text{C}$	2.29	2.17	608
$\text{Gd}_2\text{Fe}_{13.5}\text{Ni}_{0.5}\text{C}$	2.18	2.05	641
$\text{Gd}_2\text{GFe}_{13}\text{NiC}$	2.10	2.03	660
$\text{Gd}_2\text{Fe}_{12.5}\text{Ni}_{1.5}\text{C}$	2.07	1.96	672
$\text{Gd}_2\text{Fe}_{13.5}\text{Si}_{0.5}\text{C}$	2.15	1.99	613
$\text{Gd}_2\text{Fe}_{13}\text{SiC}$	1.96	1.88	616
$\text{Gd}_2\text{Fe}_{12.5}\text{Si}_{1.5}\text{C}$	1.82	1.75	620
$\text{Gd}_2\text{Fe}_{13.5}\text{Cu}_{0.5}\text{C}$	2.25	2.09	617
$\text{Gd}_2\text{Fe}_{13}\text{CuC}$	2.16	2.08	623
$\text{Gd}_2\text{Fe}_{13.5}\text{V}_{0.5}\text{C}$	2.08	1.86	601
$\text{Gd}_2\text{Fe}_{13}\text{VC}$	1.92	1.57	596

composition dependences are plotted in Fig. 1. While in the case of $H_{m,Fe}$ values a dependence on the type and concentration of the M atoms is evidenced, the $H_{m,Gd}$ values are relatively insensitive to substitutions.

The temperature dependences of the anisotropy fields, $H_A(T)$ of $Gd_2Fe_{14-x}M_xC$ compounds, with $x = 1.5$ for $M = Ni, Si$ and $x = 1.0$ for $M = Cu, V$ are shown in Fig. 2. Previously, an anomalous temperature dependence of the anisotropy field of the $Gd_2Fe_{14}C$

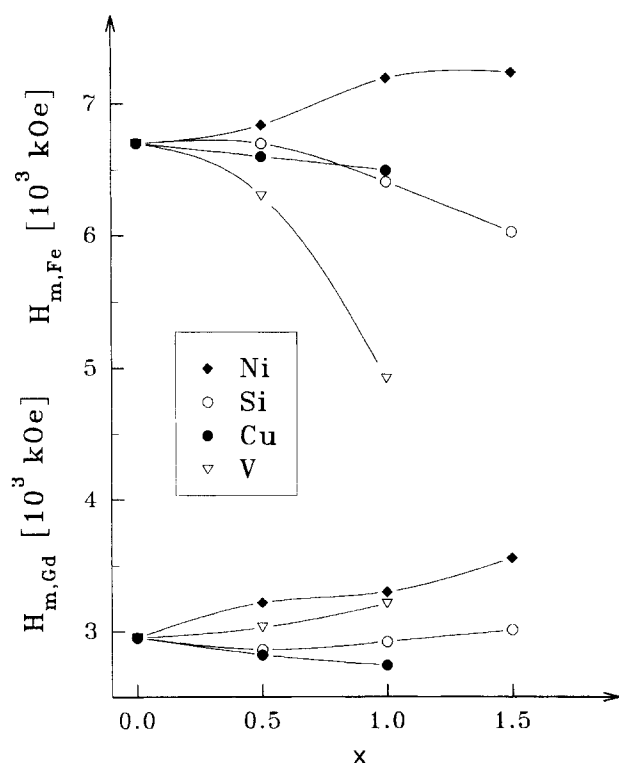


Fig. 1. Composition dependences of the molecular fields acting on iron ions $H_{m,Fe}$, and gadolinium ions $H_{m,Gd}$.

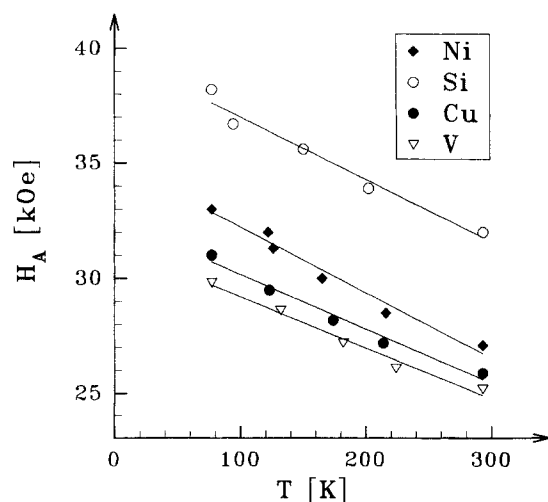


Fig. 2. Temperature dependence of the anisotropy field of the $Gd_2Fe_{14-x}M_xC$ compounds, with $x = 1.5$ for $M = Ni, Si$ and $x = 1.0$ for $M = Cu, V$.

compound was reported [15]. This was attributed mainly to an Invar-like thermal expansion in $R_2Fe_{14}X$ compounds, which leads to the observed magneto-volume anomaly of the 3d-anisotropy. According to our results, in the case of the base compound, $H_A = 28.5$ kOe at 77 K and 34.7 kOe at 293 K, which is consistent with the values given in Ref. [15]. As a result of iron substitution, a qualitative change of the $H_A(T)$ curves occurs, namely a continuous decrease of the anisotropy fields as temperature increases may be observed. The determined behaviour cannot be correlated with the lattice constant variations due to the M substitutions, as they show an increase for $M = Cu$ and Ni and a decrease for $M = Si$ and V , (see Table 1). It is likely that a preferential iron site occupancy mechanism is effective in this case. The substituted compounds have higher magnetocrystalline anisotropies than the base compound at 77 K, but the room temperature values are lower. The largest values of the anisotropy field are obtained for $M = Si$.

4. Mössbauer results

The experimental and calculated Mössbauer spectra of $Gd_2Fe_{14-x}M_xC$ compounds are presented in Figs.

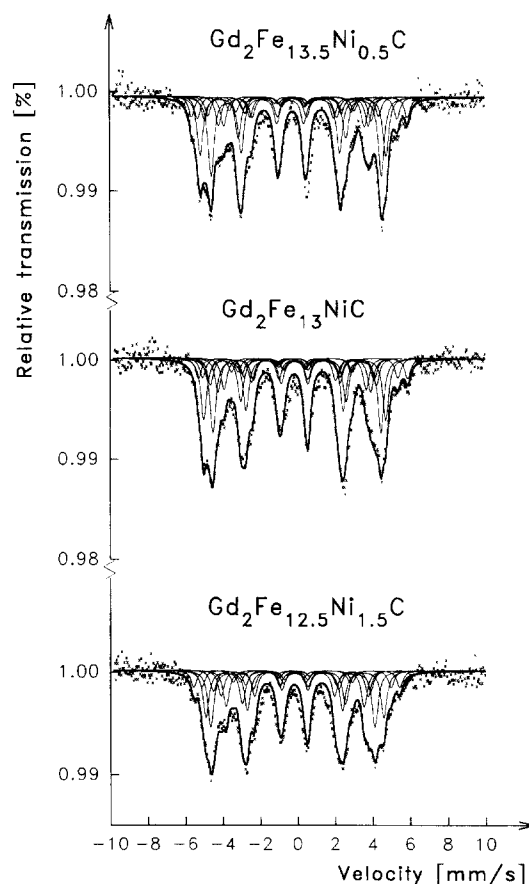


Fig. 3. Experimental and calculated Mössbauer spectra of $Gd_2Fe_{14-x}Ni_xC$ compounds.

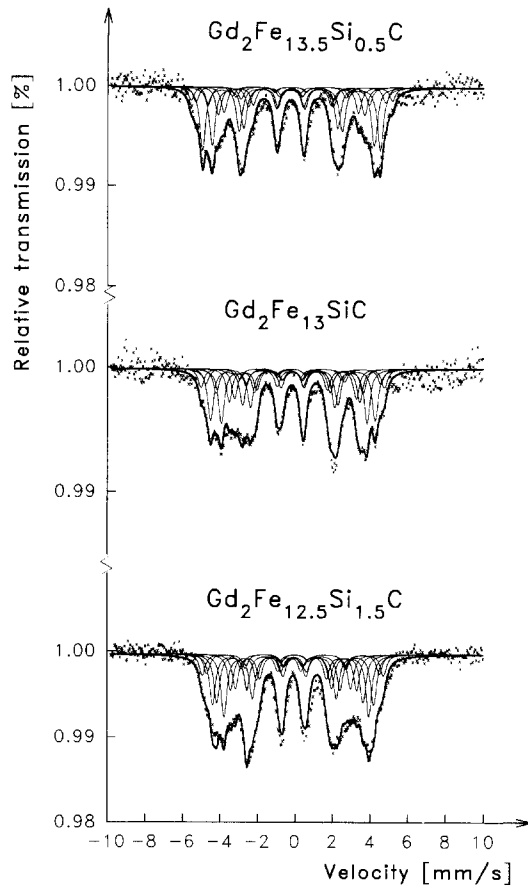


Fig. 4. Experimental and calculated Mössbauer spectra of $Gd_2Fe_{14-x}Si_xC$ compounds.

3–6. A broadening of the absorption lines of the experimental spectra compared with the spectrum of the base compound is observed [9]. This may be attributed to a distribution of the hyperfine fields as the local environment of the iron atoms is modified by alloying with a substitutional element. The spectra of the compounds with $M = Cu, V$ and Si were decomposed satisfactorily into seven sextets, while in the case $M = Ni$ eight sextets were necessary.

The assignment of the magnetic sextets to the six crystallographically inequivalent iron sites was made on the basis of their (i) hyperfine fields and (ii) relative intensities, following the considerations stated in Ref. [9]. The present data show that for all compounds the $Fe(8j_2)$ site splits into two sextets, in the ratio 1:1, with close values of the hyperfine fields. In the nickel compounds there is also a splitting of $Fe(8j_1)$ site into two sextets, in the ratio 1:1. Under the assumption that the iron nearest neighbours (NNs) are the main contributors to the H_{hf} values, the sextets with the largest H_{hf} were assigned to the $(8j_2)$ site (12 Fe NNs). Between the $(16k_2)$ and $(16k_1)$ sites, both with 10 Fe NNs, the sextet with the largest H_{hf} value was assigned

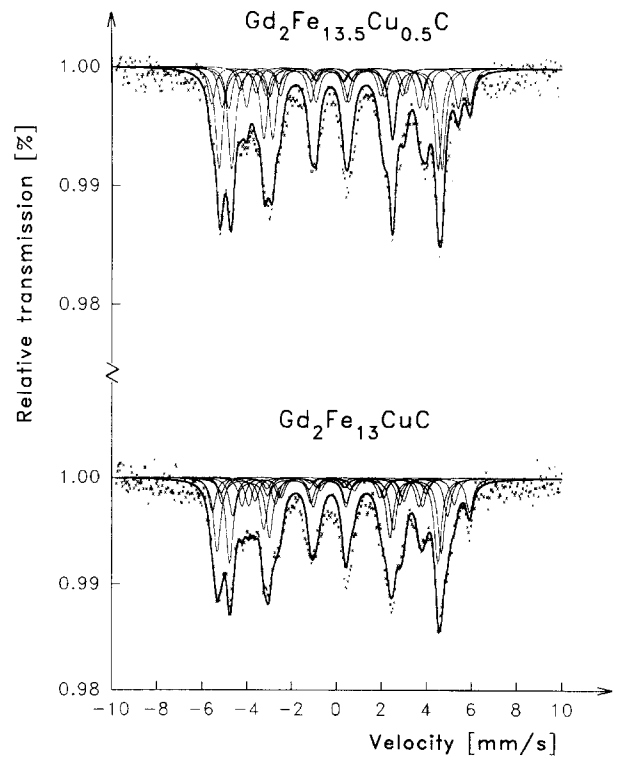


Fig. 5. Experimental and calculated Mössbauer spectra of $Gd_2Fe_{14-x}Cu_xC$ compounds.

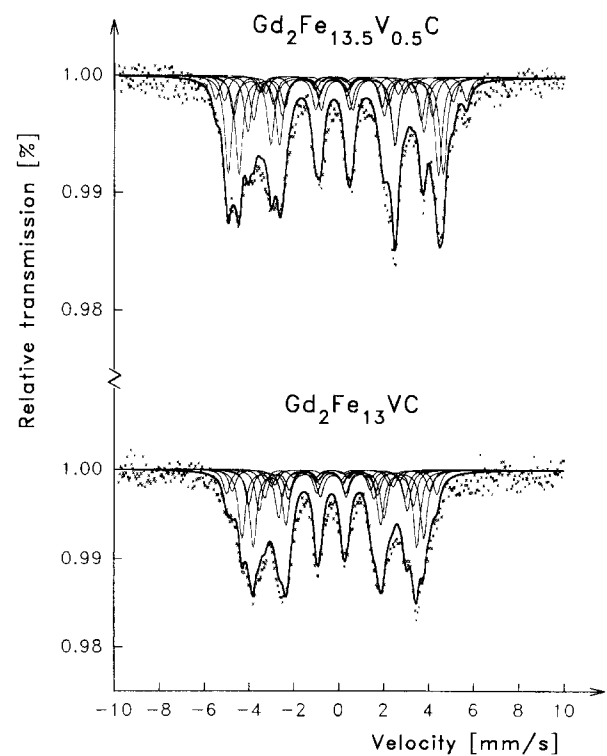


Fig. 6. Experimental and calculated Mössbauer spectra of $Gd_2Fe_{14-x}V_xC$ compounds.

to the Fe(16k₂) site, because the Fe(16k₁) site has one carbon atom NN which reduces its magnetic moment and, by correlation, the hyperfine field. It follows that the sextet with the third highest value of H_{hf} corresponds to the Fe(16k₁) site. The next two sextets are assigned to Fe(8j₁) and Fe(4e) sites, both with 9 Fe NNs but different crystallographic degeneracies. Thus, the sextet with the smallest hyperfine field belongs to the Fe(4c) site. Consequently, in our assignment, the H_{hf} values, listed in Table 3, follow the sequence $8j_2 > 16k_2 > 16k_1 > 8j_1 > 4e > 4c$.

The calculated spectra of the samples containing Gd₂Fe₁₇C_y as a secondary phase, showed an overestimation of the relative intensities of the 4e and 4c sublattices. The 2:17:C phase is most prominent by its 18f and 18h sublattices. Therefore, we assumed an average content of the interstitial carbon $y \sim 0.25$ and subtracted the spectrum of Gd₂Fe₁₇C_{0.25} from the experimental spectra of the samples containing this type of impurity phase [16]. Then, we re-analysed the corrected data. By applying this method, the number of sextets into which the experimental spectra were initially decomposed did not change. We could estimate the total amounts of 2:17:C phase present in the samples as being 6–12%. However, a drawback of the method is that one cannot rely on the accuracy of the determined relative intensities of 4e and 4c sublattices, and hence cannot derive any preferential substitution of these particular iron sites.

Table 3
Local hyperfine fields in Gd₂Fe_{14-x}M_xC compounds

Compound	H_{hf} (kOe)					
	16k ₁	16k ₂	8j ₁	8j ₂	4e	4c
Gd ₂ Fe ₁₄ C ^a	283.0	309.3	283.0	345.7	273.8	246.6
Gd ₂ Fe _{13.5} Ni _{0.5} C	284	310	278	360	250	235
			285	333		
Gd ₂ GFe ₁₃ NiC	280	304	278	349	255	237
			277	326		
Gd ₂ Fe _{12.5} Ni _{1.5} C	274	297	263	343	245	230
			274	315		
Gd ₂ Fe _{13.5} Si _{0.5} C	270	296	268	335	243	224
			316			
Gd ₂ Fe ₁₃ SiC	242	274	244	305	217	202
			291			
Gd ₂ Fe _{12.5} Si _{1.5} C	241	268	243	301	210	192
			287			
Gd ₂ Fe _{13.5} Cu _{0.5} C	287	310	275	359	254	241
			328			
Gd ₂ Fe ₁₃ C ₁₄ C	289	312	299	358	251	237
			285	323		
Gd ₂ Fe _{13.5} V _{0.5} C	277	299	276	349	245	233
			318			
Gd ₂ Fe ₁₃ VC	227	253	229	292	206	195
			275			

^a Data from Ref. [10].

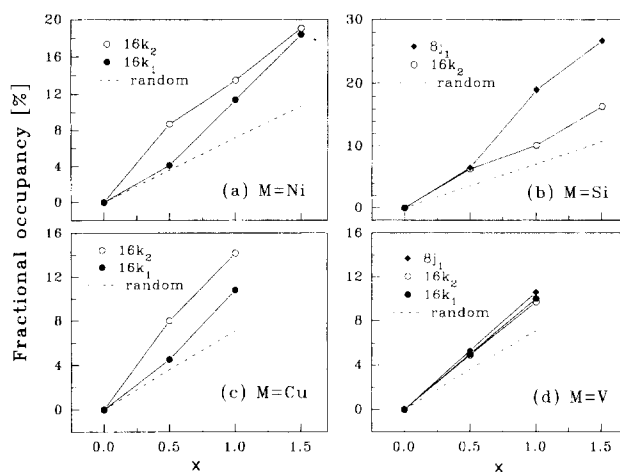


Fig. 7. Fractional occupancy of the (16k₁), (16k₂) and (8j₁) iron sites in Gd₂Fe_{14-x}M_xC compounds.

The differences between the values of the calculated relative intensities of the sextets and the values characterising a uniform distribution of M atoms, $(16-x):(8-y):(4-z) = 4:2:1$, evidence preferentially-substituted Fe sites. These results, presented in Fig. 7, show that Ni and Cu atoms are located mostly in (16k₂) and (16k₁) sites, Si replaces iron preferentially in (8j₁) and (16k₂) sites and V atoms are distributed quite uniformly over the (8j₁), (16k₂) and (16k₁) sites. A previous study performed by neutron diffraction and Mössbauer effect on the Y₂Fe_{14-x}Si_xB system [17], revealed that Si strongly prefers Fe(4c) and Fe(8j₁) sites, with respectively four and three yttrium atoms as NNs. Such a preference of silicon for Fe sites with the largest coordination in rare earth elements or yttrium was also evidenced by the ⁵⁷Fe Mössbauer effect in Y₂(Fe,Si)₁₇ [18] and R(Fe,Si)₁₂ [19] and by neutron diffraction in Nd₂(Fe,Si)₁₇ [20]. Although a similar preference of the silicon can be expected in Gd₂Fe_{14-x}Si_xC compounds, we could not observe the occupancy of Fe(4c) sites owing to the presence of Gd₂Fe₁₇C_y secondary phase in the samples.

The composition dependences of the average IS are plotted in Fig. 8(a). For M = Ni and Cu the IS values are small, negative and approximately constant, in the limit of the experimental errors, suggesting reduced charge transfers as a result of the substitution. In the case of vanadium compounds, the IS increases toward negative values as the concentration increases, while in case of the silicon compounds the IS values show the opposite trend.

The QS values are small and they do not vary with composition except within the limit of errors. This suggests that, in the studied composition ranges, the substitutional atoms do not change significantly the symmetry of charge distributions in the local environment of Fe sites.

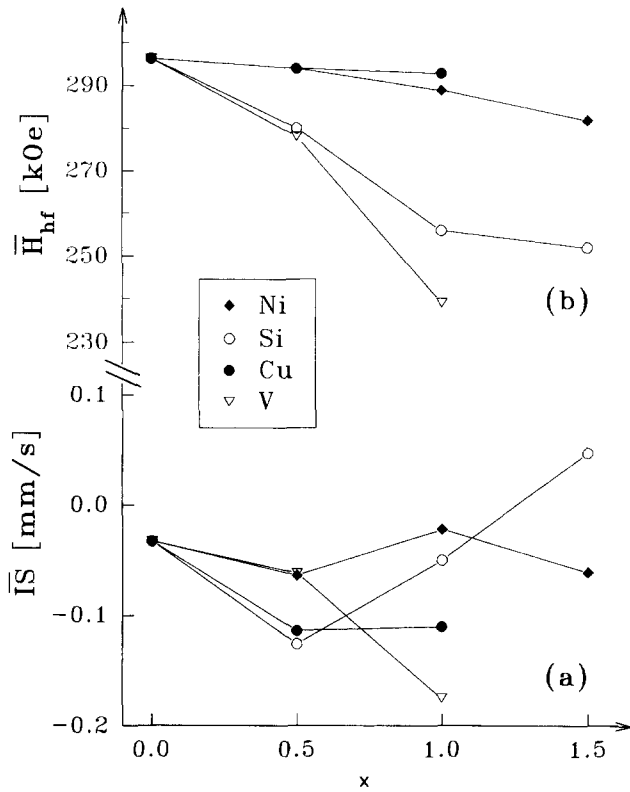


Fig. 8. Composition dependences of the average hyperfine fields (a), and IS values (b), of the $Gd_2Fe_{14-x}M_xC$ compounds.

5. Discussion

In the $Gd_2Fe_{14}C$ compound, the distances between $Fe(k_2)-Fe(k_2)$, $Fe(k_2)-Fe(c)$, $Fe(k_1)-Fe(k_2)$ and $Fe(j_1)-Fe(j_1)$ are less than the critical value, $d_0 = 2.45$ Å, below which Fe–Fe negative exchange interactions are expected. The preferential occupancy of these iron sites by the substitutional elements diminish the negative exchange within the Fe sublattice and hence lead to an increase in Curie temperatures, as observed for $M = Ni, Cu$ and Si . In vanadium compounds, the effect on T_c owing to the strong decrease of iron magnetic moments dominates the effect of negative exchange reduction as a result of the preferential substitution. Therefore, a decrease in Curie temperatures is induced as vanadium concentration increases.

In the mean field approximation, the Curie temperatures of iron compounds are proportional to the Fe–Fe exchange interaction coefficient, n_{FeFe} , and the square of the mean iron moment M_{Fe} , $T_c \sim n_{FeFe} M_{Fe}^2$. The dependence of T_c/M_{Fe}^2 on the n_{FeFe} values for the $Gd_2Fe_{14-x}M_xC$ compounds, presented in Fig. 9, shows a linear relationship, as predicted in the molecular field model. The points corresponding to the compounds with $M = V$, $x = 1.0$, and $M = Si$, $x = 1.0$ and 1.5 had to be withdrawn from the graph owing to their large deviations from the predicted

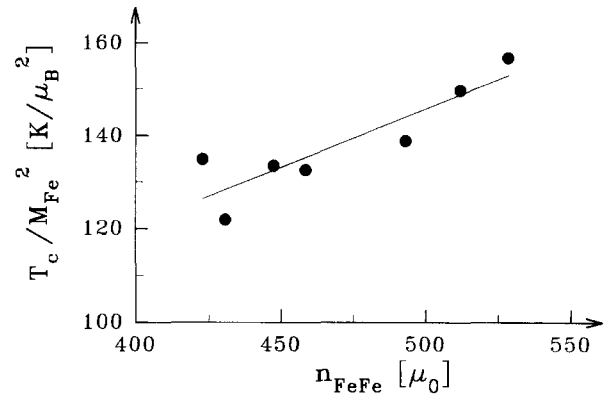


Fig. 9. Dependence of T_c/M_{Fe}^2 values on the n_{FeFe} values for $Gd_2Fe_{14-x}M_xC$ compounds.

behaviour, suggesting that effects due to change of electronic density of states are also significant in these compounds. These may be analysed in correlation with the composition dependences of the lattice constants and average IS values (Table 1 and Fig. 8(a)). For the V and Si substitutions, the lattice constants decrease. In the case of the vanadium compounds there are two contributions to the decrease of the IS values, one due to the shrinkage of the Wigner–Seitz cells of the iron atoms, leading to an increase of the s electron density at the nucleus, and the other due to valence electron transfer from vanadium to iron atoms. In silicon compounds, although the contribution arising from the contraction of the crystalline lattice is also present, it is obvious that a second contribution, related perhaps to the nature of the chemical bonding formed between silicon and iron, is dominant. As a result, the IS values increase to positive values as the Si content increases.

Analysing the effect of the substitutional elements on the anisotropy fields, Fig. 2, compared with the $H_A(T)$ behaviour determined in the base compound, it may be derived that the preferential occupancy of $Fe(16k_2)$, $Fe(16k_1)$ and $Fe(8j_1)$ sites leads to a relaxation of the crystalline lattice, by lowering the value of the magnetoelastic energy responsible for the magnetovolume anomaly observed in $Gd_2Fe_{14}C$. Also, it is apparent that these iron sites have favourable individual contributions to the 3d anisotropy of the base compound, as M substitutions into these sites lower the total magnetocrystalline anisotropy at room temperature.

The preferential substitution of Fe atoms located at the $(16k_2)$, $(16k_1)$ and $(8j_1)$ sites determines the observed splitting of the $Fe(8j_2)$ sublattice into two magnetic sextets. This particular iron site has the largest number of substituted next NN sites and, consequently, its local environment is the most affected one.

The composition dependences of the average hyperfine fields are given in Fig. 8(b). Assuming a

proportionality relation between the average H_{hf} values and the average iron moments in $\text{Gd}_2\text{Fe}_{14-x}\text{M}_x\text{C}$ compounds, $H_{\text{hf}} = AM_{\text{Fc}}$ and using the room temperature data from Table 2, one obtains for the hyperfine constant $A = (144 \pm 4) \text{ kOe } \mu_{\text{B}}^{-1}$. This value is essentially the same as that previously used in calculating the mean iron moments from hyperfine field data in the $\text{R}_2\text{Fe}_{14}\text{X}$ compounds [21]. Using the local H_{hf} values and the hyperfine constant determined in this paper, the decreasing sequence of the Fe local magnetic moments differs from those obtained by neutron scattering [22] or by LMTO calculations [23] for $\text{Tb}_2\text{Fe}_{14}\text{C}$. However, any alternative assignment of the magnetic sextets to the crystallographically inequivalent Fe sites would lead to inconsistencies concerning the values of the relative intensities of the sextets in the Mössbauer spectra, as was also pointed out in Ref. [24].

The replacement of iron by copper has the least decreasing effect on H_{hf} values in $\text{Gd}_2\text{Fe}_{14-x}\text{M}_x\text{C}$ compounds, Fig. 9, as was also observed in $\text{R}_2\text{Fe}_{14-x}\text{Cu}_x\text{B}$ [25]. The Ni substitution has a more pronounced effect than Cu, but moderate, while both Si and V substitutions lead to a severe reduction of the hyperfine field values. The same effects are also evidenced in the composition dependences of the mean iron moments determined by magnetic measurements.

6. Conclusions

Analysis of the ^{57}Fe Mössbauer parameters, namely hyperfine fields and relative intensities, in correlation with the local environment of iron lattice sites indicates a preferential distribution of the substitutional elements on the $\text{Fe}(16k_2)$, $\text{Fe}(16k_1)$ and $\text{Fe}(8j_1)$ sites. As a result of the preferential substitution of these iron atoms also involved in negative exchange interactions, an increase in Curie temperatures is observed for $\text{M} = \text{Ni}, \text{Cu}$ and Si . In the case of vanadium compounds, this effect is counteracted by the strong decrease of iron moments due to a conduction electron transfer from V to Fe 3d band. This is also supported by the large, negative IS values determined for the $\text{Gd}_2\text{Fe}_{14-x}\text{V}_x\text{C}$ system.

Iron substitution by Ni, Si, Cu or V determines the decrease of the mean hyperfine fields and of the mean iron moments, the proportionality constant being approximately $145 \text{ kOe } \mu_{\text{B}}^{-1}$.

Experimental data suggest that iron atoms located at $(16k_2)$, $(16k_1)$ and $(8j_1)$ sites have significant contributions to the 3d sublattice anisotropy. Their substitu-

tion leads to a decrease of both magnetoelastic energy and magnetocrystalline anisotropy of the compounds.

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