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Studies of Mössbauer spectra for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$

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Abstract. Mössbauer spectra of $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ as well as their parents, $\text{Sm}_2\text{Fe}_{17}$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$, respectively, have been studied. Because of the influence of the C and Si atoms, at least five and seven subspectra are necessary to obtain a good fit to the data for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$, respectively. The effects of the C atoms on the hyperfine parameters are discussed. The hyperfine fields for the carbides increase on the 6c and 9d sites but decrease on the 18f and 18h sites as compared to those for their parents. This suggests an origin for the lower magnetization observed for $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ as compared with that for $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$.

1. Introduction

$\text{R}_2\text{Fe}_{17}\text{N}_y$ and $\text{R}_2\text{Fe}_{17}\text{C}_y$ have some outstanding magnetic properties. However, they decompose into RN or RC and α -Fe at temperatures of 600–700 °C. Because of this drawback, it is difficult for these nitrides and carbides to be made into useful permanent magnets with high remanences, at least by normal techniques. Recently, it was found that Si is able to stabilize the structure of the 2:17-type carbides [1, 2]. These new Si-containing carbides, $\text{R}_2(\text{Fe}, \text{Si})_{17}\text{C}_y$, made by a gas-phase reaction, are stable even at a high temperature of 700 °C. They overcome a major drawback of the carbides $\text{R}_2\text{Fe}_{17}\text{C}_y$ or nitrides $\text{R}_2\text{Fe}_{17}\text{N}_y$, namely chemical (or structural) instability at high temperatures. Meanwhile, these Si-containing carbides have good magnetic properties. For $\text{R}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$, the Curie temperature is 630 K and the anisotropy field is over 70 kOe; however, the saturation magnetization is low, about 88 emu g^{-1} at room temperature [1].

Mössbauer spectroscopy is a powerful technique for studying the magnetic properties of the rare-earth iron compounds. Mössbauer spectra of the nitrides, $\text{R}_2\text{Fe}_{17}\text{N}_y$, have been studied extensively—see, for example, [3–8]. However, only a few papers have appeared on the carbides $\text{R}_2\text{Fe}_{17}\text{C}_y$ [9, 10]. In fact, the Mössbauer spectra for the carbides and nitrides are significantly different; they are also different for $\text{R}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ and $\text{R}_2\text{Fe}_{17}\text{C}_y$. Furthermore, the Mössbauer spectra of $\text{R}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ are more complicated than those of $\text{R}_2\text{Fe}_{17}\text{N}_y$ and $\text{R}_2\text{Fe}_{17}\text{C}_y$. Hence, it is necessary to study the Mössbauer spectra for $\text{R}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ further in order to gain a better understanding of their magnetic properties. In this paper, Mössbauer spectra for $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ and $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ as well as their parents ($y = 0$) have been studied and the effects of C atoms on the hyperfine parameters are discussed. These results will be useful in the analysis of the Mössbauer spectra of similar carbides.

2. Experimental details

$\text{Sm}_2\text{Fe}_{17}$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$ compounds were prepared by arc melting of primary materials that were better than 99.8% pure in a purified argon atmosphere. The ingots were annealed in an argon atmosphere at 900 °C for 4 h and then quenched in water. The carbides $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ were made by heating the powders of their parents in CH_4 for 2 h at 450 °C and 700 °C, respectively. X-ray diffraction patterns showed that all samples contain single-phase $\text{Th}_2\text{Zn}_{17}$ structure; however, small amounts of α -Fe are present in $\text{Sm}_2\text{Fe}_{17}$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ samples and a considerable amount occurs in the $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ sample.

^{57}Fe Mössbauer spectra were collected using a conventional constant-acceleration spectrometer. The γ -ray source was ^{57}Co in a Rh matrix. Mössbauer absorbers were powdered samples with about 8 mg cm^{-2} of natural iron. Calibration was made by using the spectrum of α -Fe at room temperature. Since the quadrupole splitting was much smaller than the magnetic hyperfine splitting for all samples, a perturbation Hamiltonian was used to analyse the Mössbauer spectral data. The parameters obtained for each six-line subspectrum by a least-squares fitting procedure were the hyperfine field, H_{hf} , the quadrupole splitting, ϵ , the isomer shift, δ , the line widths and the intensities. The area ratios of the six absorption lines in each sextet were constrained to be 3:2:1:1:2:3 because the absorbers were powders with randomly oriented particles. For each Fe site, the subspectral areas for $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ and $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ were kept the same as those for their corresponding parents because it is assumed that the distributions of Si and Fe atoms are not changed during the process of C absorption.

3. Results and discussion

3.1. Analysis of Mössbauer spectra

Most of the neutron diffraction experiments indicate that N or C atoms exclusively occupy the 9e sites in the $\text{Th}_2\text{Zn}_{17}$ structure [11–14]. The crystal structure and local environment around each Fe site are shown in figure 1. The interatomic distances between Fe and C atoms are also listed in the figure. Because no exact determination of the parameters of the atomic positions could be found for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$, atomic positions for $\text{Nd}_2\text{Fe}_{17}\text{N}_y$ [12] were used instead to calculate the interatomic distances.

Mössbauer spectra and the computer-fitted curves for $\text{Sm}_2\text{Fe}_{17}$, $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$, $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ are shown in figure 2; the hyperfine parameters are listed in table 1. For the $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ sample, there is a considerable amount of α -Fe; the relative area of the α -Fe subspectrum is about 25–30%. The spectrum for the $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ shown in the figure 1 is the remanent one after stripping off the α -Fe spectrum.

The Mössbauer spectra of $\text{Sm}_2\text{Fe}_{17}$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$ were fitted by using four subspectra, corresponding to the four Fe sites, the 6c, 9d, 18f and 18h sites. However, for the carbides $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{R}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ a good fit with only four subspectra cannot be obtained. The effect of the C or/and Si atoms on the hyperfine parameters has to be considered.

3.1.1. $\text{Sm}_2\text{Fe}_{17}\text{C}_y$. If it is assumed that the recoil-free fractions on the four Fe sites are the same, the relative area ratios of the subspectra should be 2:3:6:6 for the 6c, 9d, 18f and 18h sites, respectively. In order to maintain the relative area of 3/17 for the 9d site, the subspectrum of the carbides has to be split into two components. Their area ratios are $6.8:10.8 = 0.63(16)$ and $9.2:11.2 = 0.82(15)$ for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{R}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$, respectively.

It is known that the 9d sites have four C atoms as their nearest neighbours (1st nn).

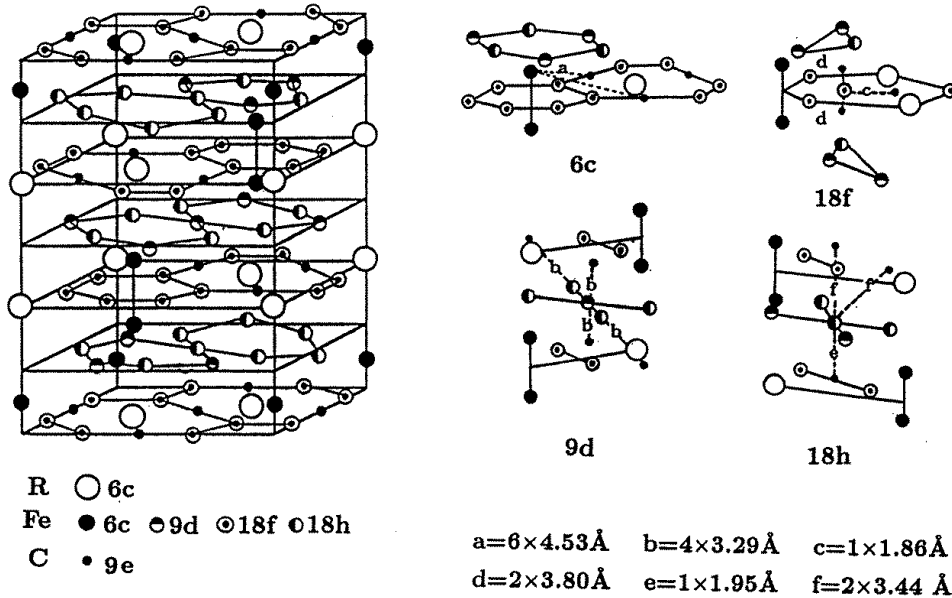


Figure 1. The crystal structure and local environment around Fe sites for $\text{R}_2\text{Fe}_{17}\text{C}_y$ with the $\text{Th}_2\text{Zn}_{17}$ structure.

On the basis of the binomial distribution model, the probabilities of finding m adjacent C atoms for the 9d sites are given by

$$P(m) = \frac{n!}{m!(n-m)!} c^m (1-c)^{n-m} \quad (1)$$

where $n = 4$ is the number of adjacent 9e sites around the 9d site and $c = y/3$ is the relative carbon concentration. The ratio $P(3)/P(4)$, as a function of C concentration, y , is plotted in figure 3. The area ratios of the two components for the 9d sites, 0.63 and 0.82, obtained from the Mössbauer spectra, correspond to C concentrations of 2.6(1) and 2.5(1) for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$, respectively. These values are consistent with C concentrations, 2.4–2.6 and 2.3–2.5, obtained by weighing. Hence, the two components for the 9d sites can be attributed to the various configurations of adjacent C atoms.

For the 6c sites, there is no 1st nn C atom. The shortest distance between the Fe (the 6c site) and C atoms is 4.53 Å. Moreover, the 18f sites are located between the 6c sites and 9e sites. As a result, the C atoms are screened by Fe atoms on the 18f sites and have little influence on the hyperfine parameters of the 6c sites. For the 18f and 18h sites, there is only one 1st nn C atom and the distances between Fe and the 1st nn C atom are 1.86 and 1.95 Å, respectively. The probabilities for finding one C atom are over 0.8 and the probabilities for other C-atom configurations, therefore, are so small that they can be neglected. Hence, for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$, there is only one component for each of the 6c, 18f and 18h sites.

3.1.2. $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$. The Mössbauer spectrum of $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ is complicated. In particular, some fine structures exist in the first spectral lines ($v \sim -2.5$ to -4 mm s^{-1}) for the 18f and 18h sites. In order to obtain a good fit and to maintain the relative area of about 6/17 for the 18f and 18h sites, each subspectrum for the two sites has to be split into two

Table 1. Mössbauer parameters of $\text{Sm}_2\text{Fe}_{17}$, $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$, $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$: hyperfine field, H_{hf} ; quadrupole splitting, ϵ ; isomer shift, δ ; and relative area, S . The numbers in parentheses are fitting errors in the last significant digit.

	Site	H_{hf} (kOe)	ϵ (mm s^{-1})	δ (mm s^{-1})	S (%)
$\text{Sm}_2\text{Fe}_{17}$	6c	264(3)	0.02(3)	0.02(2)	11.9(10)
	9d	229(2)	-0.10(3)	-0.25(2)	17.9(16)
	18f	209(2)	0.10(2)	-0.32(1)	35.7(31)
	18h	206(2)	0.14(2)	0.01(1)	34.5(30)
$\text{Sm}_2\text{Fe}_{15}\text{Si}_2$	6c	268(3)	0.04(3)	0.05(2)	12.0(6)
	9d	238(2)	-0.07(2)	-0.11(2)	20.4(12)
	18f	211(2)	0.06(2)	-0.27(1)	31.3(15)
	18h	208(2)	0.14(2)	0.05(1)	36.3(15)
$\text{Sm}_2\text{Fe}_{17}\text{C}_y$	6c	343(2)	-0.09(3)	0.08(2)	12.0(10)
	9d(I)	330(2)	-0.03(3)	-0.06(2)	10.8(10)
	9d(II)	275(3)	-0.22(5)	-0.04(3)	6.8(11)
	18f	253(2)	-0.50(2)	-0.06(4)	35.8(28)
	18h	244(2)	-0.16(2)	0.07(5)	34.6(27)
$\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$	6c	310(2)	-0.14(4)	0.13(2)	12.0(8)
	9d(I)	289(2)	-0.08(4)	0.03(2)	11.2(8)
	9d(II)	210(2)	-0.43(4)	-0.03(3)	9.2(9)
	18f(I)	196(3)	-0.64(5)	-0.10(3)	9.6(8)
	18f(II)	187(2)	-0.30(3)	0.03(2)	21.7(10)
	18h(I)	194(2)	-0.21(4)	0.09(2)	13.8(8)
	18h(II)	162(2)	-0.10(3)	0.15(2)	22.5(11)

components, i.e. 18f(I), 18f(II), 18h(I) and 18h(II). The area ratios of the two components are $9.6:21.7 = 0.44(6)$ and $14.1:22.5 = 0.63(8)$ for the 18f and 18h sites, respectively.

For the 18f site, the number, n , of the nearest-neighbour Fe plus Si atoms is 10. The probability for zero Si atoms adjacent to the 18f site, based on equation (1), is calculated to be 0.29; the total probability for one or two adjacent Si atoms is 0.61. Their ratio is $0.29:0.61 = 0.48$. For the 18h site, n is equal to 9. The probabilities for zero adjacent Si atoms and for one or two adjacent Si atoms are 0.32 and 0.60, respectively. Their ratio is $0.32:0.60 = 0.53$. The area ratios of the two components obtained from Mössbauer spectra, which are 0.44 and 0.63 for the 18f and 18h sites, respectively, are close to the results calculated by the binomial distribution model of Fe and Si atoms. Hence, the components 18f(I) and 18h(I) correspond to the neighbour configuration with zero Si atoms; the 18f(II) and 18h(II) correspond to the configuration with one or two Si atoms. It is known that the neighbouring Si atoms lead to a decrease in the hyperfine field and an increase in the isomer shift [15, 16]. As a result, the hyperfine fields are larger and the isomer shifts are smaller for the 18f(I) than for the 18f(II), respectively; the same is true for the 18h(I) and 18h(II).

For simplicity, the subspectra for the 6c and 9d sites still consist of one and two components, respectively, as for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$.

3.2. Mössbauer parameters

The isomer shifts, quadrupole splittings and hyperfine fields for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ are significantly different from their corresponding parents, as shown in figure 4.

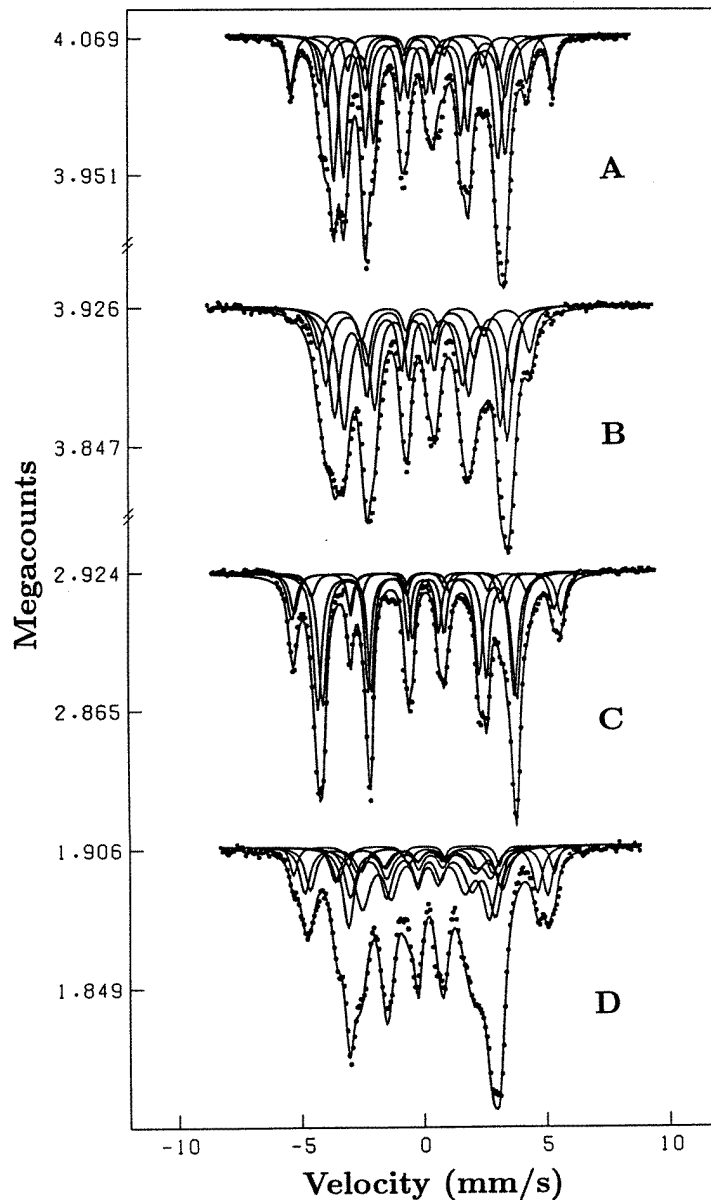


Figure 2. Mössbauer spectra and their fitted curves for (A) $\text{Sm}_2\text{Fe}_{17}$, (B) $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$, (C) $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and (D) $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$.

3.2.1. Isomer shifts. The average isomer shifts are $-0.15(2)$ and $-0.08(2)$ mm s^{-1} for $\text{Sm}_2\text{Fe}_{17}$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$, respectively, and $0.01(4)$ and $0.06(2)$ mm s^{-1} for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$, respectively. As compared to $\text{Sm}_2\text{Fe}_{17}$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$, there is an increase of 0.16 and 0.14 mm s^{-1} in the average isomer shifts for the corresponding carbides. There are two factors which are largely responsible for the increase in the average isomer shifts. One is the expansion of the cell volume because C atoms are introduced. This expansion leads to a decrease in the charge density at the nucleus and thus there is an increase in the

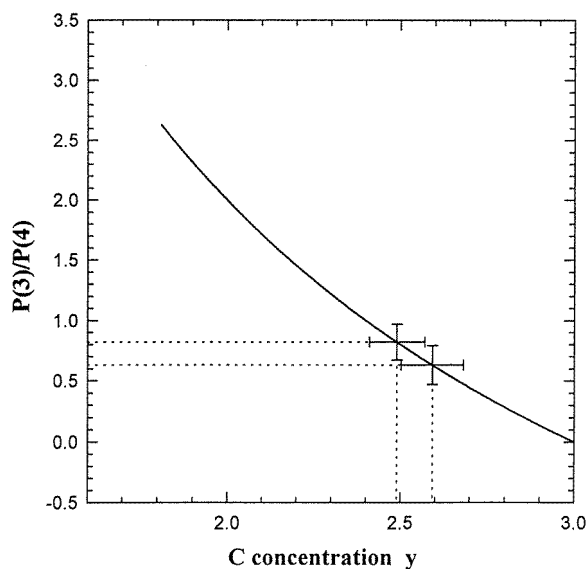


Figure 3. The dependence of $P(3)/P(4)$ on the C concentrations based on the binomial distribution model. Here $P(m)$ is the probability of m nearest carbon neighbours.

isomer shifts. The other factor is the transfer of electrons from Fe to C atoms. Since the electronegativity is much larger for C than for Fe or Si, the C atoms have a tendency to attract the conduction electrons of Fe or Si. A decrease in the Fe conduction electrons will also lead to an increase in the isomer shifts.

3.2.2. Quadrupole splittings. The quadrupole splittings, for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$, are close in magnitude and the same in sign for the corresponding Fe sites. However, they are opposite in sign for the 6c, 18f and 18h sites, as compared to those for $\text{Sm}_2\text{Fe}_{17}$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$, and are significantly different in magnitude for the corresponding sites. Specifically, for the 18f site, the splittings are only $0.10(2)$ and $0.06(2)$ mm s^{-1} for $\text{Sm}_2\text{Fe}_{17}$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$, respectively; they are, however, $-0.50(2)$ and $-0.40(2)$ mm s^{-1} for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$, respectively. Further, the average quadrupole splittings, calculated by weighting their subspectral areas, are about $0.06(2)$ mm s^{-1} for $\text{Sm}_2\text{Fe}_{17}$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$, but about $-0.25(4)$ mm s^{-1} for their carbides. These results indicate that the carbides and their parents have a different type of magnetic anisotropy. It is known that if the magnetic moments are either in the c -plane or along the c -axis, the quadrupole splittings will be different in magnitude and/or in sign because of the different angles between the direction of the moment and the principal axis of the EFG (electric-field gradient). X-ray diffraction studies and magnetic measurements have also shown that $\text{Sm}_2\text{Fe}_{17}$ and $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$ have an easy-plane anisotropy and their carbides have an easy c -axis anisotropy [2].

The finding that the quadrupole splittings are much larger in magnitude for the carbides than for their parents can be attributed to a large asymmetry of the EFG caused by the C atoms. In addition, for the nitride $\text{Sm}_2\text{Fe}_{17}\text{N}_x$, the quadrupole splittings are -0.11 , -0.13 , -0.17 and -0.22 mm s^{-1} for the 6c, 9d, 18f and 18h sites, respectively, and the average quadrupole splitting is $-0.17(3)$ mm s^{-1} [17]. As compared to that for $\text{Sm}_2\text{Fe}_{17}\text{N}_y$, the average quadrupole splitting for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ is the same in sign; however, it increases in

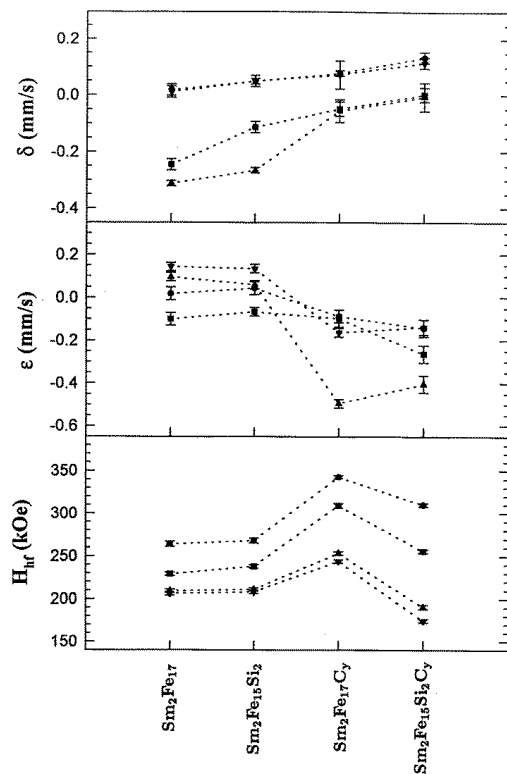


Figure 4. Mössbauer parameters. δ , ϵ and H_{hf} are the isomer shifts, quadrupole splittings and hyperfine fields (6c, ●; 9d, ■; 18f, ▲; and 18h, ▼) of the various intermetallic compounds, respectively.

magnitude by about 50%, which shows that the C atoms produce a larger asymmetry in the EFG than do the N atoms.

3.2.3. Hyperfine fields. The Mössbauer spectral shapes for either $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ or $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ are significantly different from those for their corresponding parents. The first and sixth spectral lines for the 6c and 9d sites are separated from those for the 18f and 18h sites of the carbides.

The hyperfine fields at room temperature for $\text{Sm}_2\text{Fe}_{15}\text{Si}_2\text{C}_y$ are 310, 256, 184 and 179 kOe for the 6c, 9d, 18f and 18h sites, respectively. They, as compared to those for $\text{Sm}_2\text{Fe}_{15}\text{Si}_2$, increase by 32 and 18 kOe for the 6c and 9d sites, respectively, but decrease by 26 and 29 kOe for the 18f and 18h sites, respectively.

Similarly, the hyperfine fields at room temperature for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ are 342, 309, 253 and 244 kOe, respectively. They, as compared to those for $\text{Sm}_2\text{Fe}_{17}$, increase by 78 and 80 kOe for the 6c and 9d sites, respectively, and only by 44 and 38 kOe for the 18f and 18h sites, respectively. Because the Curie temperatures for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ and $\text{Sm}_2\text{Fe}_{17}$ are very different, it may be more reasonable for the hyperfine fields to be compared at the same reduced temperature, T/T_f . If the hyperfine fields at room temperature ($T/T_f = 300 \text{ K}/660 \text{ K} \sim 0.45$) for $\text{Sm}_2\text{Fe}_{17}\text{C}_y$ are compared to those at $T = 176 \text{ K}$ ($T/T_f = 173 \text{ K}/410 \text{ K} \sim 0.42$) for $\text{Sm}_2\text{Fe}_{17}$ [4], the hyperfine fields increase by about 21

kOe for the 6c and 9d sites, but decrease by 13 kOe for the 18f site. The same result is obtained again—that the C atoms lead to an increase in the hyperfine field for the 6c and 9d sites, but a decrease for the 18f and 18h sites at the same reduced temperature T/T_f .

This conclusion is supported by a non-self-consistent LCAO calculation, which showed that the magnetic moments on the 6c and 9d sites are larger for $Y_2Fe_{17}C_3$ than those for Y_2Fe_{17} , but the moments on the 18f and 18h sites are smaller [5]. In addition, there is a similar result for Fe–C martensite; the 1st nn Fe atoms around a C atom have a hyperfine field of 265 kOe and the second-nearest-neighbour Fe atoms have a hyperfine field of 334 kOe. These are smaller and larger than 331 kOe for α -Fe [18], respectively.

This difference may be related to the distances between the Fe and C atoms. It is known that the shortest distances between the C atoms and the 6c and 9d sites are 4.53 and 3.29 Å, respectively; however, the shortest distances of the C atoms from the 18f and 18h sites are only 1.86 and 1.95 Å. These short distances may lead to a hybridization of the 3d electrons of an Fe atom and the 2s and 2p electrons of a neighbouring C atom. From the Alexander and Anderson model [19], the covalent bonds formed in pure Fe or Fe alloys will lead to a decrease of the magnetic moments and, therefore, of the hyperfine fields for the 18f and 18h sites.

4. Conclusions

Because of the effect of C and Si atoms, at least five and seven subspectra are needed to obtain a good fit for $Sm_2Fe_{17}C_y$ and $Sm_2Fe_{15}Si_2C_y$, respectively. These results will also be useful in the analysis of Mössbauer spectra of similar carbides.

The C atoms lead to an increase in the isomer shifts and a large increase with opposite signs in the quadrupole splittings for $Sm_2Fe_{17}C_y$ and $Sm_2Fe_{15}Si_2C_y$, as compared to those for their parents. For the carbides, the hyperfine fields on the 6c and 9d sites increase, whereas the fields on the 18f and 18h sites decrease, as compared to those for their parents. This may be the source of the lower magnetization for $Sm_2Fe_{15}Si_2C_y$ as compared to $Sm_2Fe_{15}Si_2$.

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