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Abstract

The Mössbauer spectra of the YCo$_{4-x}$Fe$_x$B compounds, where x is 1, 2 and 3,
have been measured at 78 and 295 K and reveal relatively small iron hyperfine
fields of about 15 T, relatively large quadrupole interactions of about $+1.0$
and -1.0 mm s^{-1} and very different spectra for YCo$_3$FeB and YCo$_2$Fe$_2$B as
compared with YCoFe$_3$B. As a consequence, the spectra have been fitted with
a full Hamiltonian by adjusting the hyperfine parameters of the iron 2c and 6i
sites found in these compounds. The fits have used an asymmetry parameter,
η, and Euler angles, θ and ϕ, that relate the hyperfine field to the iron electric
field gradient axes of each iron site in an orientation that is consistent with the
structural and magnetic properties of the site. The fits indicate that the relative
areas of the spectral components assigned to the two iron 2c and 6i sites are
in agreement with the iron occupancy of the sites as determined by neutron
diffraction. The results of the fits also indicate both that the full Hamiltonian
approach is required for physically reasonable spectral fits and that the small
observed fields result from the presence of large orbital contributions which
subtract from the Fermi contact contributions to the magnetic hyperfine fields
of the two sites.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Since the initial work of Kuz'ma *et al* in 1974 [1], many RCo$_{4-x}$Fe$_x$B compounds, where R
is Y or a rare earth and with $0 \leqslant x \leqslant 4$, have been synthesized and studied in detail [2–10].

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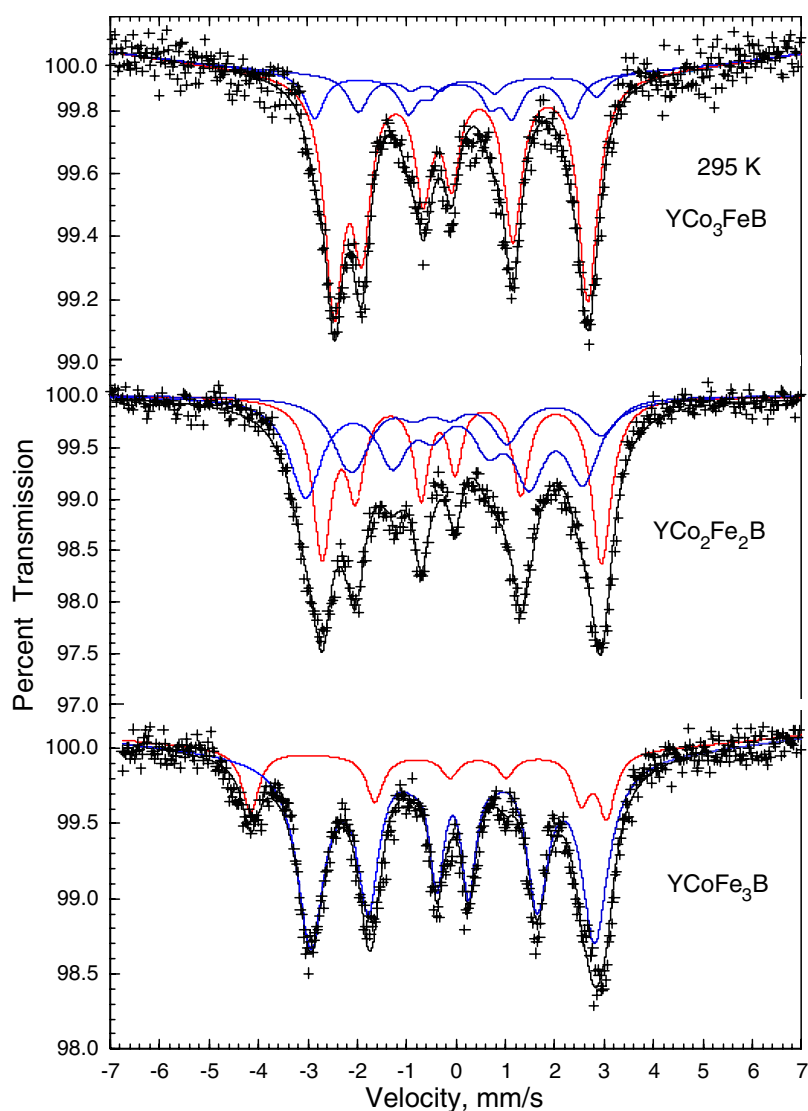
Figure 1. The Mössbauer spectra of the $YCo_{4-x}Fe_xB$ compounds obtained at 295 K. The 2c and 6i spectral components are shown in red and blue, respectively, in the online colour edition. The identity of each spectral component may be determined from the hyperfine fields given in table 1 and are shown in red and blue for the 2c and 6i spectral components, respectively, in the online colour edition.

These compounds crystallize with the hexagonal $P6/mmm$ space group and exhibit either axial or basal magnetic anisotropy, an anisotropy that can change as a function of either temperature or composition, or both. These compounds are interesting both because their structures can be derived from that of RCo_5 by replacing one cobalt with one boron and because they appear in the ternary phase diagrams of the rare earths, iron and boron, phase diagrams that also contain the important permanent magnet $R_2Fe_{14}B$ phase. Further, when R is a magnetic rare earth, these compounds have a compensation temperature at which the magnetization vanishes because the magnetic rare-earth and transition metal moments oppose each other. Hence, it is of value to
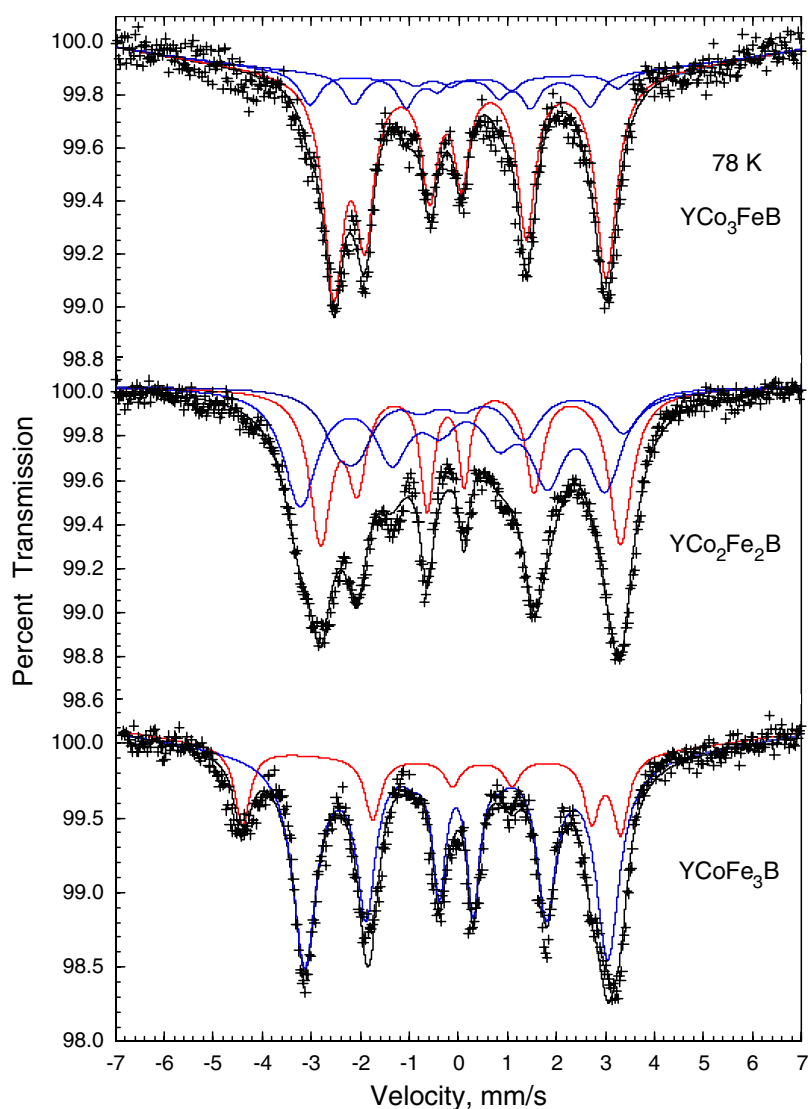
Figure 2. The Mössbauer spectra of the YCo$_{4-x}$Fe$_x$B compounds obtained at 78 K. The identity of each spectral component may be determined from the hyperfine fields given in table 1 and are shown in red and blue for the 2c and 6i spectral components, respectively, in the online colour edition.

understand the macroscopic and microscopic properties of the RCo$_{4-x}$Fe$_x$B compounds. Their macroscopic properties may be investigated by x-ray and neutron diffraction and magnetic measurements, whereas their microscopic properties, such as the magnetic moments carried by both cobalt and iron, may be obtained from neutron diffraction and Mössbauer spectral measurements. Herein, the Mössbauer spectra of the YCo$_{4-x}$Fe$_x$B compounds, with $x = 1, 2$ and 3, are presented and analysed, with the goal of investigating the transition metal sublattice, that is the unique magnetic sublattice.

The YCo$_{4-x}$Fe$_x$B compounds, with $x = 0, 1, 2$ and 3, crystallize [8] with the hexagonal $P6/mmm$ space group. The substitution of cobalt by iron induces an anisotropic expansion of the c-axis of the unit cell. However, the unit-cell volume increases linearly from 150.4 to

155.3 Å^3, when x increases from 0 to 3; concurrently, the Curie temperature increases from 380 to 674 K, and the 5 K saturation magnetization increases from 2.9 to 6.1 μ_B/fu. A 300 K neutron diffraction study [9, 11] has indicated that iron preferentially occupies the 2c site rather than the 6i site. Further, the 300 K iron magnetic moments are about 2.0 and 1.2 μ_B for the 2c and 6i sites and are oriented within the basal plane [9, 11], an orientation which is in agreement with the 295 K x-ray diffraction patterns obtained on oriented powders. Although YCo$_4$B exhibits a spin reorientation from basal [9, 11, 12], below 100 K, to axial, above 150 K, a spin reorientation which results from the competition between the different magnetic anisotropies of the two cobalt sites, the YCo$_{4-x}$Fe$_x$B compounds, with $x = 1$, 2 and 3, do not exhibit [8, 11] any spin reorientation between 4.2 and 295 K.

2. Experimental details

The samples of YCo$_{4-x}$Fe$_x$B, with $1 \leqslant x \leqslant 3$, used for the present Mössbauer spectral studies are the same as those prepared and characterized [8, 9, 11] earlier. The Mössbauer spectra have been measured at 78 and 295 K on a constant-acceleration spectrometer which utilized a rhodium matrix cobalt-57 source and was calibrated at room temperature with α-iron foil. The Mössbauer spectral absorbers contained about 35 mg cm^{-2} of powdered sample which had been sieved to a 0.045 mm or smaller diameter particle size.

3. Analysis of the Mössbauer spectra

The Mössbauer spectra of the YCo$_{4-x}$Fe$_x$B compounds, with $1 \leqslant x \leqslant 3$, obtained at 295 and 78 K are shown in figures 1 and 2, respectively. An initial visual inspection of these spectra reveals, first, that the iron hyperfine fields are relatively small at about 14–24 T and, second, that the spectra of YCoFe$_3$B are very different from those of YCo$_3$FeB and YCo$_2$Fe$_2$B. Similar observations have been reported by Gros *et al* [6] in their study of RCo$_3$FeB, where R is Pr, Nd or Sm. These authors have analysed in detail the influence upon the Mössbauer spectral profile of the orientation of the iron magnetic moment and hyperfine field, in the presence of a large quadrupole interaction, on both the iron 2c and 6i sites. A comparison of their results with the spectra in figures 1 and 2 indicates that YCoFe$_3$B exhibits an axial magnetization at both 78 and 295 K whereas YCo$_3$FeB and YCo$_2$Fe$_2$B exhibit a basal magnetization at both 78 and 295 K. The axial magnetization in YCoFe$_3$B disagrees with the basal magnetization previously reported [8, 11] for this compound, a disagreement that is discussed in detail below.

The presence of small hyperfine fields and large quadrupole interactions in the YCo$_{4-x}$Fe$_x$B compounds does not permit a fit of the Mössbauer spectra with a first-order perturbation of the Hamiltonian. Such incorrect fits have been reported by Maruyama *et al* [10] for the Mössbauer spectra of ErCo$_{4-x}$Fe$_x$B and the hyperfine parameters quoted by these authors should be interpreted with great caution. Hence, a fit of the spectra of the YCo$_{4-x}$Fe$_x$B compounds with a full Hamiltonian has to be carried out and the resulting fits are shown in figures 1 and 2; the resulting spectral parameters are given in table 1. The accuracy in the refined parameters given in parentheses in this table represents the statistical accuracy of the fits. More realistic accuracies are probably twice as large as the given values. In these fits, the asymmetry parameter, η, of the 2c and 6i sites has been constrained to 0 and 1, respectively. The zero asymmetry parameter of the 2c sites is expected [6] from the axial point symmetry of this site. The orthorhombic symmetry at the 6i sites does not impose an asymmetry parameter of 1 but fits carried out with a different asymmetry parameter for this site were significantly poorer. The Euler angles, θ and ϕ, of the hyperfine field in the electric field gradient axes depend on the orientation of the iron magnetic moments in the unit cell. For the 2c site, in the case of an axial orientation of the iron magnetic moments,

Table 1. Mössbauer spectral parameters for the $YCo_{4-x}Fe_xB$ compounds. (Note: The parameters are defined in the text and are given with statistical accuracies if they have been refined.)

Compd.	T (K)	Site	δ [a] (mm s^{-1})	$e^2Qq/2$ (mm s^{-1})	H (T)	η	θ (deg)	ϕ (deg)	Area (%)	Γ (mm s^{-1})	$\Delta\Gamma$	y
YCo_3FeB	295	2c	−0.134(3)	−1.05(1)	15.32(3)	0	90	0	73	0.39(2)	0.02(1)	2
		6i$_1$	−0.08(1)	1.16(3)	14.6(2)	1	0	90	9	0.39(2)	0.02(2)	2
		6i$_2$	−0.08(1)	1.16(3)	14.1(1)	1	120	90	18	0.39(2)	0.02(2)	2
	78	2c	−0.016(2)	−1.08(1)	16.66(2)	0	90	0	73	0.332(4)	0.055(2)	2
		6i$_1$	0.030	1.18(3)	16.1(2)	1	0	90	9	0.61(3)	0.000(1)	2
		6i$_2$	0.030	1.18(3)	15.9(1)	1	120	90	18	0.61(3)	0.000(1)	2
YCo_2Fe_2B	295	2c	−0.119(3)	−1.01(2)	17.05(3)	0	90	0	43	0.35(1)	0.05(1)	3.20
		6i$_1$	−0.08(1)	0.86(2)	15.8(1)	1	0	90	19	0.65(3)	0.05(1)	3.20
		6i$_2$	−0.08(1)	0.86(2)	16.4(1)	1	120	90	38	0.65(3)	0.05(1)	3.20
	78	2c	−0.013(3)	−1.05(1)	18.52(3)	0	90	0	43	0.30(1)	0.097(4)	3.31
		6i$_1$	0.070(4)	0.86(1)	17.7(1)	1	0	90	19	0.70(4)	0.04(1)	3.31
		6i$_2$	0.070(4)	0.86(1)	18.40(5)	1	120	90	38	0.70(4)	0.04(1)	3.31
$YCoFe_3B$	295	2c	−0.055(5)	−1.00(1)	22.37(4)	0	0	0	18.3(4)	0.40(1)	0.000(4)	2.22
		6i	−0.066(2)	0.91(1)	17.21(2)	1	90	0	81.7(4)	0.319(4)	0.115(2)	2.22
	78	2c	−0.029(2)	−1.035(3)	23.94(1)	0	0	0	21.1(1)	0.374(3)	0.001(1)	2.42
		6i	−0.042(1)	0.974(3)	18.48(1)	1	90	0	78.9(1)	0.284(1)	0.095(1)	2.42
$YCoFe_3B$ [b]	295	2c	−0.058(4)	−0.96(1)	22.8(2)	0	0	0	31	0.30(1)	0	2.54
		6i	−0.078(4)	0.91(1)	16.9(1)	1	90	0	69	0.30(1)	0	2.54
	78	2c	−0.052(4)	−0.91(1)	24.1(2)	0	0	0	31	0.28(1)	0	2.39
		6i	−0.038(4)	0.96(1)	18.4(1)	1	90	0	69	0.28(1)	0	2.39

[a] The isomer shifts are given relative to room temperature α-iron foil.
[b] The weighted average parameters obtained from the binomial fits discussed in the text.

θ and ϕ are 0° and 0°, respectively, whereas in the case of a basal orientation of the iron magnetic moments, θ and ϕ are 90° and 0°, respectively [6]. For the 6i site, in the case of axial orientation of the iron magnetic moments, θ and ϕ are 90° and 0°, respectively. In the case of basal orientation of the iron magnetic moments, the 6i sites must be subdivided into two magnetically inequivalent 6i$_1$ and 6i$_2$ subsites, with relative populations of 1 and 2, respectively. In this case, the Euler angles, θ and ϕ, of the hyperfine field are 0° and 90° for the 6i$_1$ subsite and 120° and 90° for the 6i$_2$ subsites, respectively [6]. Both the line widths and isomer shifts of the 6i$_1$ and 6i$_2$ subsites have been constrained to be the same and the relative areas of the 2c and 6i sites have been constrained to be consistent with the 2c occupancies determined [8, 9, 11] from neutron diffraction measurements. In addition to the hyperfine parameters and line width, an incremental line width has been fitted in order to account for the distribution of cobalt and iron on the 2c and 6i sites. This incremental line width, $\Delta\Gamma$, has been defined as $\Gamma(v) = \Gamma + (v - \delta)\Delta\Gamma$, where v is the velocity and δ is the isomer shift of a given subspectrum. Finally, a texture parameter, y, was introduced into the fits, as a multiplying factor of the Clebsch–Gordan coefficient, $2^{1/2}$, of the $\Delta m = 0$ transitions. In the case of a sextet this is essentially equivalent to a component area ratio of $3:y:1:1:y:3$ where y may vary from zero for a hyperfine field parallel to the γ-ray direction, to 4 for a hyperfine field perpendicular to the γ-ray direction. In the absence of this texture parameter, much poorer fits were obtained. Specifically, the line positions were correctly fitted but their relative intensities were very poorly fitted. The presence of texture in the absorbers probably results from the strong magnetic anisotropy of the $YCo_{4-x}Fe_xB$ compounds, an anisotropy that favours a preferential orientation of the crystallites in the absorbers.

The fits obtained with the above model are all very good to excellent as indicated by the solid lines in figures 1 and 2 and by the resulting χ^2 values. However, the fits of the spectra
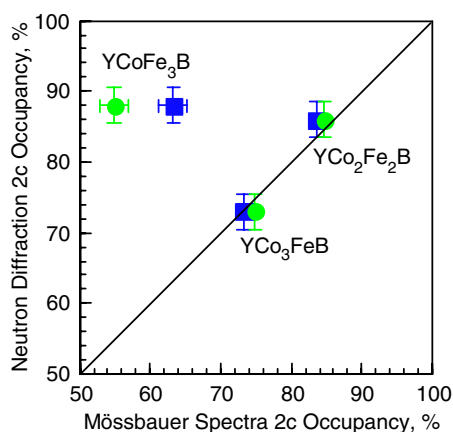
Figure 3. The correlation between the iron occupancy of the 2c site, as determined at 78 K (squares; blue in the online version) and at 295 K (circles; green in the online version) by Mössbauer spectroscopy, without a binomial distribution, and as determined at 295 K by neutron diffraction.

of $YCoFe_3B$ do show some misfit specifically about the absorption at about -4 mm s^{-1}. This misfit clearly indicates the difficulty of modelling the distributions of the cobalt and iron on the 2c and 6i sites through the use of an incremental line width. Thus, as is discussed below, the spectra of $YCoFe_3B$ have also been fitted with a binomial distribution of the cobalt and iron near-neighbours of the 2c and 6i sites.

In the fits shown in figures 1 and 2 the relative areas of the 2c and 6i components found in the YCo_3FeB and YCo_2Fe_2B spectra have been constrained to be consistent with the occupancies obtained from neutron diffraction studies [8, 9, 11], a constraint that assumes that the recoil-free fractions of the two sites are the same. Both to test this assumption and to determine the impact of the constraint on the quality of the fit, fits without this constraint have been carried out. In the case of YCo_3FeB and YCo_2Fe_2B there is only a marginal improvement in the fit and little change in the iron occupancy of the 2c site as may be seen in figure 3. In contrast to the above two compounds, the fits of the Mössbauer spectra of $YCoFe_3B$ with constrained areas are very poor and the neutron diffraction based constraint on the relative areas must be relaxed in order to obtain a reasonable fit of the spectra. Unfortunately, as may be seen in figure 3, the resulting relative amount of iron on the 2c site is dramatically underestimated in the resulting fit. This underestimation occurs because of the poor fit of the 2c component—especially the absorption line near -4 mm s^{-1} (see the bottom of figures 1 and 2). In order to overcome the limitations of the incremental linewidth approach and to determine whether the neutron diffraction occupancies are compatible with the Mössbauer spectra, we have undertaken a binomial distribution fit of the Mössbauer spectra of $YCoFe_3B$. This binomial fit, which used three components for the 6i site and four components for the 2c site, is based on the 88% iron occupancy of the 2c site determined by neutron diffraction and an assumed random distribution of the iron and cobalt on the six 6i near-neighbours of the 2c site and the four 6i near-neighbours of the 6i site. The four sextets for the 2c site correspond to iron atoms with zero to four iron near neighbours, whereas the three sextets for the 6i site correspond to iron atoms with zero to three iron near neighbours. The above-mentioned constraints on the asymmetry parameter and Euler angles have also been maintained and the isomer shifts, hyperfine fields and quadrupole interactions of the seven sextets have been fitted. One line width and a zero incremental line width have been used for all seven sextets to both reduce the number of fitted parameters and to test the validity of the binomial model. The best fits
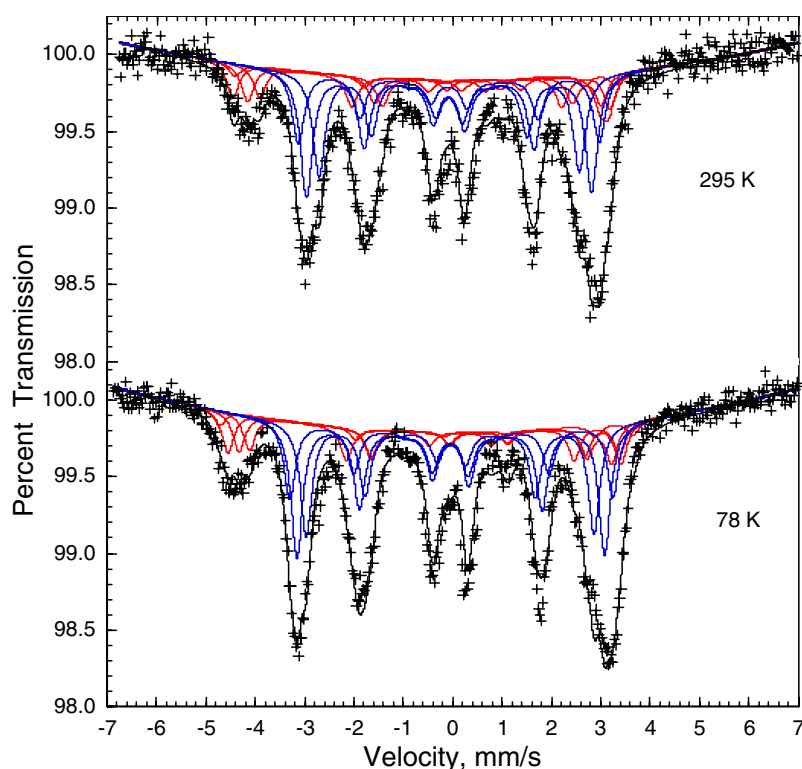
Figure 4. The Mössbauer spectra of YCoFe$_3$B obtained at 78 and 295 K and fit with a binomial distribution of near-neighbour environments for the 2c and 6i sites by using the iron occupancies determined from neutron diffraction. The identity of each component may be determined from the hyperfine fields given in table 1 and are shown in red and blue for the 2c and 6i spectral components, respectively, in the online colour edition.

obtained at 78 and 295 K are shown in figure 4. Although these fits may not be unique, they do indicate that satisfactory fits, specifically at about -4 mm s^{-1}, may be obtained with this approach. Further, the percentage of iron present on the 2c site is in agreement with the neutron diffraction [8, 9, 11] results. The resulting weighted average hyperfine parameters for the binomial fits are given at the bottom of table 1 and are used in figure 5. In the binomial model approach, the hyperfine fields increase with the increasing number of iron near neighbours.

4. Results and discussion

As would be expected, and as is indicated by the values given in table 1 and figure 5, all the 78 K hyperfine fields and isomer shifts are larger than the 295 K hyperfine fields and isomer shifts, respectively. The observed temperature dependences of the hyperfine fields and isomer shifts are expected from the mean-field Brillouin behaviour of the magnetization and the second-order Doppler shift, respectively, and provide confidence in the fits shown in figures 1, 2 and 4. Further, the quadrupole interactions, $e^2Qq/2$, of the 2c and 6i sites are about -1 and $+1$ mm s^{-1}, respectively. These values agree well with those observed [6] in RCo$_3$FeB, with R = Pr, Nd or Sm. The large magnitude of the quadrupole interactions results from the low symmetry experienced by the iron-57 on both of the sites.
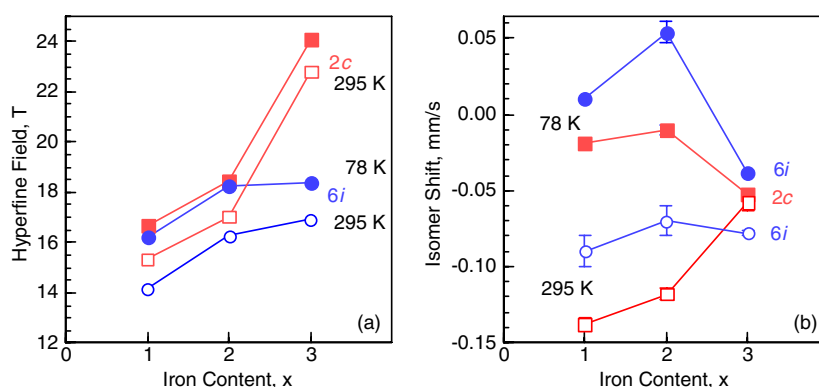
Figure 5. The compositional dependence of the site average hyperfine fields, (a), and the isomer shifts, (b), in the $YCo_{4-x}Fe_xB$ compounds. The error bars on the hyperfine fields are smaller than the size of the data points. This is also the case for some of the error bars on the isomer shifts. The 2c and 6i parameters are shown as the red squares and blue circles, respectively, in the online colour edition.

The compositional dependence of the hyperfine fields is shown in figure 5(a). The increase in hyperfine field with increasing iron content is related to the increase in magnetization observed [8, 9, 11] both at 4 and 300 K. The hyperfine fields of 14–18 T in YCo_3FeB and YCo_2Fe_2B are small for the iron magnetic moments of 2.1 and 1.2 μ_B measured [8, 9, 11] by neutron diffraction for the 2c and 6i sites, respectively. These small hyperfine fields result from the combination of the Fermi contact and orbital terms, terms that have opposite signs. Because the orbital term is roughly proportional to the electric quadrupole interaction, it is expected to be large in the case of the $YCo_{4-x}Fe_xB$ compounds, compounds that exhibit large quadrupole interactions. Hence, the observed hyperfine field is reduced from the Fermi contact term of -22 T, a conclusion that is supported by earlier ^{59}Co NMR measurements on YCo_4B [13, 14] which have revealed a large orbital contribution to the cobalt hyperfine field. The orbital contributions to the hyperfine field may be estimated to be about $+4$ to $+6$ T. The interplay of the Fermi contact and orbital terms in determining the hyperfine field also explains the increase in hyperfine field observed in figure 5(a) for $YCoFe_3B$. As indicated above, the Mössbauer spectra of $YCoFe_3B$ are typical of an axial orientation of the iron magnetic moments. Hence, for the 2c and 6i sites θ is 0° and 90°, respectively, and the two components to the hyperfine field add differently to lead to a large increase in the 2c hyperfine field of about 5 T and a small increase of the 6i hyperfine field of about 0.5 T.

Even though the Mössbauer spectral data are both completely internally consistent and consistent with earlier work [6] on similar compounds, it should be noted that the axial orientation of the iron magnetic moments in $YCoFe_3B$ disagrees with the basal orientation reported earlier [8, 9, 11] based on both x-ray diffraction on oriented powder and neutron diffraction measurements. In order to resolve this discrepancy, a new x-ray diffraction study of a field-oriented powder of the sample used for the above Mössbauer spectral study and the earlier diffraction studies [8, 9, 11] has been carried out and indicates that the sample now exhibits axial magnetization in agreement with the Mössbauer spectral results reported herein. The discrepancy with the earlier diffraction results is currently unresolved.

The compositional dependence of the 2c and 6i isomer shifts at 78 and 295 K is shown in figure 5(b). In both YCo_3FeB and YCo_2Fe_2B, compounds that exhibit a basal magnetization, the 6i isomer shift is more positive than the 2c isomer shift. Further, both isomer shifts increase with the increasing unit-cell volume as x increases from 1 to 2.

An individual site magnetic anisotropy model [15, 16] has successfully described [17] the change in magnetic anisotropy in the GdCo$_{4-x}$Fe$_x$B compounds. In this model, the first-order magnetic anisotropy constant, $K_1(x)$, is given by

$$K_1(x) = K_1(0) + 2f_{2c}(x)\Delta K_1^{2c} + 6f_{6i}(x)\Delta K_1^{6i}, \tag{1}$$

where $K_1(0) = 5 \times 10^5$ J m^{-3} is the magnetic anisotropy constant for GdCo$_4$B, $f_{2c}(x)$ and $f_{6i}(x)$ are the iron 2c and 6i occupancies and ΔK_1^{2c} and ΔK_1^{6i} are the differences in the iron and cobalt contributions to the magnetic anisotropy for the 2c and 6i sites, respectively. The same model applied to the YCo$_{4-x}$Fe$_x$B compounds, with $x = 0, 1, 2$ and 3, using the iron 6i and 2c occupancies determined [11] from neutron diffraction and $\Delta K_1^{2c} = -7.55 \times 10^5$ J m^{-3} and $\Delta K_1^{6i} = +3.55 \times 10^5$ J m^{-3} predicts both a first spin reorientation between $x = 0$ and 1 from axial to basal and a second spin reorientation from basal back to axial for $x > 2.0$. This second spin reorientation in the larger x compositional range results from the filling of the 6i sites by iron and its positive, i.e. axial, contribution to the magnetic anisotropy.

In conclusion, the analysis of the complex Mössbauer spectra observed at 78 and 295 K for the YCo$_{4-x}$Fe$_x$B compounds, where x is 1, 2 and 3, shows that it is mandatory to use the full excited state Hamiltonian with inclusion of a strong quadrupole interaction with a large asymmetry parameter and the two Euler angles of the small hyperfine field within the electric field gradient axes. The relative areas of the components assigned to the 2c and 6i sites are in agreement with the iron occupancy of the 2c site determined from neutron diffraction measurements. The distribution of iron and cobalt on the 2c and 6i sites can be successfully modelled by an incremental line width in YCo$_3$FeB and YCo$_2$Fe$_2$B and by a binomial distribution in YCoFe$_3$B. The small observed hyperfine fields of about 15 T result from the large orbital contribution.

Acknowledgments

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