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A study of the high temperature spin reorientation in YCoFe₃B

Fernande Grandjean¹, Moulay T Sougrati¹, Hervé Mayot², Olivier Isnard² and Gary J Long³

 ¹ Department of Physics, B5, University of Liège, B-4000 Sart-Tilman, Belgium
 ² Institut Néel, CNRS and Université de Grenoble Joseph Fourier, Avenue des Martyrs, BP166, F-38000 Grenoble, France
 ³ Department of Chemistry, Missouri University of Science and Technology, University of

Missouri, Rolla, MO 65409-0010, USA

E-mail: fgrandjean@ulg.ac.be, olivier.isnard@grenoble.cnrs.fr and glong@mst.edu

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Abstract

The iron-57 Mössbauer spectra of YCoFe₃B have been measured between 4.2 and 480 K and reveal that YCoFe₃B exhibits an axial orientation of the iron magnetic moments below 450 K and a basal orientation above 450 K. This spin reorientation, also observed in the thermomagnetic curves, results from the different signs of the contributions to the magnetic anisotropy of the 2c and 6i sites that are occupied by iron. The neutron diffraction patterns of YCoFe₃B have been measured at 2 K and between 290 and 770 K and have been successfully analyzed with a model compatible with the magnetic orientation obtained from the Mössbauer spectra. The hybridization between the cobalt or iron 3d orbitals and the boron 2p orbitals leads to a larger magnetic moment and hyperfine field on the 2c site as compared to the 6i site.

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

1. Introduction

The $RCo_{4-x}Fe_xB$ compounds, where R is yttrium or a rare earth, have been extensively studied both because their crystalline structure derives from the technologically important RCo_5 structures and because their magnetic properties and, more specifically, their magnetic anisotropy, strongly depend upon the iron content, x [1–13].

Previous studies [8, 9] of the YCo_{4-x}Fe_xB compounds, where x is 1, 2, and 3, reported that they crystallize in the hexagonal *P6/mmn* space group, exhibit an anisotropic expansion of the unit cell, and show an increase in both their Curie temperatures and their 5 K saturation magnetizations with increasing x. The neutron diffraction [9, 11] and Mössbauer spectral [12] studies indicate that the iron substitutes preferentially on the 2c site. Finally, studies by powder neutron diffraction and x-ray diffraction on oriented powders indicate that the transition metal magnetic moments are oriented in the basal plane of the unit cell and do not exhibit [8, 11] any spin reorientation between 4.2 and 295 K for YCo_{4-x}Fe_xB when x is 1, 2, and 3. In contrast, YCo₄B exhibits a spin reorientation [9, 11, 14] from basal below 100 K to axial above 150 K, a spin reorientation that has been extensively studied as a function of temperature, applied field, and pressure on both polycrystalline and single crystal samples [15]. Finally, both an earlier Mössbauer spectral study [12] and neutron and x-ray diffraction measurements [8, 11] on an oriented powder of YCoFe₃B gave somewhat conflicting results for the orientation of the easy magnetization axis below 295 K. In addition, an anomaly [9] in the temperature dependence of the magnetization of YCoFe₃B has been observed at about 450 K.

In this paper, additional Mössbauer spectra between 4.2 and 480 K and neutron diffraction data between 2 and 770 K have been obtained for YCoFe₃B and analyzed in detail together with the magnetization data with the goal of clarifying the magnetic anisotropy of the iron magnetic moments.

2. Experimental details

 $YCoFe_3B$ has been prepared as described earlier [8, 9, 11]. In order to reduce the absorption of thermal neutrons by the sample, isotopic boron-11 has been used rather than natural boron.

The Mössbauer spectra have been measured between 4.2 and 480 K on a constant-acceleration spectrometer which utilized a rhodium matrix cobalt-57 source and was calibrated at room temperature with α -iron powder. The low temperature Mössbauer spectra were obtained in a Janis Supervaritemp cryostat and the absorber contained 25 mg cm⁻² of powdered sample which had been sieved to a 0.045 mm or smaller diameter particle size. The high temperature Mössbauer spectra were obtained in a laboratory built vacuum oven and the absorber was a pressed pellet of a mixture of boron nitride and 40 mg cm⁻² of YCoFe₃B.

In order to determine the easy magnetization direction, xray diffraction patterns have been obtained in reflection mode on powder samples oriented in an external magnetic field. Field-oriented samples were prepared at room temperature by solidifying a mixture of epoxy resin and YCoFe₃B powder with grain size smaller than 50 μ m in a magnetic field of ca 0.5 T; the field was perpendicular to the plane of the sample.

The Curie temperature was measured using a Faradaytype balance operating between 300 and 1000 K with a heating and cooling rate of 5 K min⁻¹. The sample was sealed in an evacuated silica tube to avoid oxidation upon heating.

The neutron diffraction investigations were performed at the Institut Laue Langevin in Grenoble, France. The thermal dependence of the neutron diffraction patterns has been recorded at medium resolution on D1B. The diffraction patterns were obtained over a 2θ angular range of 80° by using a ³He multidetector with a step of 0.2° between each of the 400 detection cells. In its high flux neutron configuration mode used herein, D1B operates at a wavelength, λ , of 2.52 Å. A detailed description of this instrument may be found elsewhere [16].

A sample of YCoFe₃B powder was placed in a 6 mm diameter cylindrical vanadium sample holder. In order to detect and characterize any magnetic spin reorientation, several diffraction patterns have been obtained between 2 and 300 K in a cryostat and between 300 and 770 K in a furnace. The neutron powder diffraction data were analyzed by the Rietveld method with the fullprof suite of programs [17]. Because the sample was prepared with boron-11, the neutron absorption originates mostly from the cobalt atoms and has been taken into account as previously described [18, 19]. These calculated neutron diffraction absorption corrections were used in the Rietveld refinements under the assumption that the density of the sample in the sample holder was only 50% of the bulk density.

3. X-ray diffraction on oriented powder

The room temperature x-ray powder diffraction pattern obtained, in reflection geometry, on a powder of YCoFe₃B oriented under a magnetic field perpendicular to the plate of the sample is compared in figure 1 with the calculated pattern for a random powder. The magnetic field reduces the intensity of many of the Bragg reflections including the most intense (103) and next most intense (102) reflections and enhances the intensity of the (003) and (004) reflections. These changes in intensity indicate that the magnetic moments in YCoFe₃B



Figure 1. The calculated x-ray diffraction pattern for a random powder of YCoFe₃B, bottom, and the experimental x-ray diffraction pattern obtained on an oriented powder of YCoFe₃B, top, with the magnetic easy-axis perpendicular to the plane of the sample. The * indicates peaks assigned to a trace of oxide impurity.

are oriented along the *c*-axis of its hexagonal unit cell at room temperature.

4. Thermomagnetization curves

The temperature dependence of the magnetization of YCoFe₃B is shown in figure 2 together, for comparison, with that of YCo₃FeB. A rather broad anomaly in the magnetization is observed between 430 and 570 K in YCoFe₃B, an anomaly that is not observed in either YCo3FeB, see figure 2, nor in YCo₂Fe₂B. However, a similar broad anomaly has been observed at spin reorientations in other intermetallic compounds that exhibit a competition between the magnetocrystalline anisotropy of different transition metals on their respective sublattices [20-22]. We have also attempted to determine the magnetic structure of YCoFe₃B on each side of the anomaly at about 410 and 590 K. However, both because of the small magnetic contribution to the neutron scattering in YCoFe₃B and because of the relative proximity in temperature of the spin reorientation and Curie temperatures, no definitive conclusion concerning the existence of a spin reorientation or the changes in the magnetic structure could be obtained. Hence, a Mössbauer spectral study between 295 and 480 K has been undertaken because the Mössbauer spectra should be very sensitive to the orientation of the iron magnetic moments [13], see the following discussion. The analysis of the neutron diffraction patterns obtained above 295 K will be discussed below in terms of the results obtained from the Mössbauer spectral study.



Figure 2. Thermomagnetization curves obtained for the $YCo_{4-x}Fe_xB$ compounds with x = 1 and 3. The curve for x = 3 has been increased by 3.58 arbitrary units.



Figure 3. The Mössbauer spectra of $YCoFe_3B$ obtained at and below 85 K. The black lines through the data points are the result of the fits discussed in the text. The very weak black sextets correspond to magnetic impurities and the blue, outer component, and red, inner component, lines correspond to the sum of the 2c and 6i components.

5. Mössbauer spectral results

The Mössbauer spectra of YCoFe₃B obtained from 4.2 to 85 K are shown in figure 3 and the spectra obtained from 300 to 480 K are shown in figure 4. First, it is apparent that the low temperature spectra are similar to the 78 and 295 K spectra previously measured [12] on a different absorber and on a



Figure 4. The Mössbauer spectra of $YCoFe_3B$ obtained at 300 K and above. The black lines are the result of the fits discussed in the text and the blue, outer component, and red, inner component, lines correspond to the sum of the 2c and 6i components.

different spectrometer, spectra that are characteristic [12, 13] of an axial orientation of the iron magnetic moments. Second, it is apparent that above 400 K, in addition to the expected decrease in hyperfine field with increasing temperature, the Mössbauer spectral profile changes significantly, a change [13] that indicates the presence of a spin reorientation.

Because of the simultaneous presence of both small hyperfine fields and large quadrupole interactions, a simulation of the iron-57 Mössbauer spectra of YCoFe₃B is not possible within the first-order perturbation approximation and requires an exact solution [12]. The resulting fits are shown as the black lines in figures 3 and 4 where the blue lines correspond to the 2c sites and the red lines correspond to the 6i sites; the corresponding site averaged isomer shifts, δ , quadrupole interactions, $eQV_{zz}/2$, hyperfine fields, H, and relative areas, and their statistical errors, are given in table 1. The absolute errors associated with these parameters are approximately twice as large as the statistical errors. In addition to the dominant YCoFe3B phase, iron-containing impurities are present and are most apparent at about 5 and 6 mm s^{-1} in the low temperature spectra. These impurities have been fit with two sextets whose total relative area amounts to about 7% of

Table 1. The site averaged Mössbauer spectral parameters for $YCoFe_3B$. (Note: the errors on the average are the statistical errors. More realistic errors may be twice as large. No error is given for the parameters that have been constrained to the value given.)

	2c					6i				
T (K)	δ^a (mm s ⁻¹)	$\frac{eQV_{zz}/2}{(\mathrm{mm~s^{-1}})}$	H(T)	Area (%)	ΔΓ	δ^a (mm s ⁻¹)	$\frac{eQV_{zz}/2}{(\mathrm{mm~s^{-1}})}$	H(T)	Area (%)	ΔΓ
480	-0.30(2)	-0.7	16.3(1)	38(2)	0	-0.16(5)	0.9	11.5(2)	62(2)	0
450	-0.25(2)	-0.7	16.7(1)	31(2)	0	-0.22(1)	0.9	13.2(2)	69(2)	0
400	-0.26(2)	-0.87(4)	18.8(1)	31(2)	0.05	-0.148(7)	0.83(4)	15.8(2)	69(2)	0.05
350	-0.19(1)	-1.10(4)	20.3(1)	31(2)	0.05	-0.094(5)	0.91(4)	16.5(2)	69(2)	0.05
300	-0.13(1)	-1.08(2)	21.31(5)	31(2)	0.05	-0.062(2)	0.96(2)	17.2(2)	69(2)	0.05
85	0.047(5)	-0.94(1)	24.71(5)	22(1)	0	0.040(7)	0.99(1)	18.6(1)	78(1)	0.03
60	0.059(5)	-0.95(1)	24.79(5)	22(1)	0	0.043(1)	0.98(1)	18.7(1)	78(1)	0.03
40	0.054(5)	-0.52(1)	24.83(5)	22(1)	0	0.046(1)	0.99(1)	18.7(1)	78(1)	0.03
20	0.036(5)	-0.89(1)	24.82(5)	22(1)	0	0.051(2)	0.98(1)	18.8(1)	78(1)	0.03
4.2	0.029(5)	-0.87(1)	24.88(5)	22(1)	0	0.051(1)	0.97(1)	18.8(1)	78(1)	0.03

^a The isomer shifts are given relative to room temperature α -iron powder.

the observed spectral area and whose hyperfine parameters are compatible with the presence of an FeCo alloy. No further reference to these impurities will be made in the following discussion.

The specific details concerning the fits and the constraints imposed upon the hyperfine parameters are the same as those used earlier in the analysis of the spectra of the $YCo_{4-x}Fe_xB$ and $GdCo_{4-x}Fe_xB$ compounds [12, 13]. The asymmetry parameter, η , of the 2c and 6i sites has been constrained to 0 and 1, respectively, in agreement with the point symmetry of these sites [6]. Further, any variation of η from 0 and 1 led to substantially poorer spectral fits. For an axial orientation of the iron magnetic moment and, hence, the hyperfine field, the Euler angles, θ and ϕ , of the hyperfine field in the electric field gradient axes are 0° and 0° for the 2c site and 90° and 0° for the 6i site. The Euler angles have been constrained to these values for all the fits of the spectra obtained at and below 400 K. In contrast, for a basal orientation of the iron magnetic moments, the Euler angles, θ and ϕ , for the 2c site are 90° and 0°, respectively. Further, because the principal axis of the electric field gradient, V_{zz} , of the 6i site is along [100], the 6i site must be subdivided into two magnetically inequivalent sites, herein designated as the 6i1 and 6i2 subsites, with relative populations of one and two, respectively. In this case, the angles, θ and ϕ , of the hyperfine field are 0° and 90° for the $6i_1$ subsite and 120° and 90° for the 6i₂ subsite, respectively, and the Euler angles have been constrained to these values for the fits of the spectra obtained at 450 and 480 K. The line widths, isomer shifts, and quadrupole interactions of the 6i1 and 6i2 subsites have been constrained to be the same at all temperatures.

In addition to adjusting the isomer shift, quadrupole splitting, and hyperfine field, for both the 2c and 6i sites in YCoFe₃B, and their line width, an incremental line width has also been fit for the 6i site in order to account for the distribution of cobalt and iron on its near-neighbors. This unitless 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 spectral component. The relative areas of the 2c and 6i components have been adjusted starting from the initial values given by the iron occupancies obtained from the neutron powder diffraction results, but as noted

previously [12], the 2c contribution to the Mössbauer spectral area was underestimated. Hence, the relative areas have been fit and are given in table 1.

In order to account for the spectral profile and, more specifically, for the line profile observed at -5 mm s^{-1} in the spectra obtained at 400 K and below, it was necessary to include four subcomponents for the 2c site and two subcomponents for the 6i site. These components reflect the change in hyperfine parameters with the distribution of cobalt and iron on the 2c and 6i sites. At 450 and 480 K, the decrease in spectral resolution, a decrease that is a normal consequence of the decrease in the hyperfine field and in recoilfree fraction, only two broadened 6i1 and 6i2 components and one 2c component are required to fit these spectra. The relative area of ca 33% observed for the 2c contribution to the Mössbauer spectra above 300 K corresponds to a nearly full occupation of the 2c site by iron in good agreement with the neutron diffraction results [9, 11]. Below 300 K the Mössbauer spectra show a 2c relative area of 22%. This smaller value may result either from the different temperature dependencies of the recoil-free fraction for the 2c and 6i sites or may be an artifact of the complex analysis involving two different models above and below 300 K, or a combination of both.

The temperature dependence of the 2c and 6i site weighted average isomer shift in YCoFe₃B is shown in figure 5. The red solid line results from a fit with the second-order Doppler shift with a Mössbauer temperature of 447 (75) K for the 6i site. Above 300 K, the 2c isomer shift is more negative than the 6i isomer shift. There is no systematic trend [6, 12, 13, 23] in the relative values of the isomer shift of the 2c and 6i sites in the RCo_{4-x}Fe_xB, where R is Sm, Gd, Dy, and Tb and x is 1–3. The quadrupole interactions are virtually independent of temperature.

The temperature dependence of the 2c and 6i site weighted average hyperfine field in YCoFe₃B is shown in figure 6(a). The 2c hyperfine field is larger than the 6i hyperfine field for both the axial and basal orientations of the iron magnetic moments. The same relative values of the hyperfine fields have been observed [12, 13, 23] in the RCo_{4-x}Fe_xB, where R is Gd, Dy, and Tb and x is 1–3, and agree with the larger magnetic moment measured [9, 11] on the 2c site by neutron diffraction.



Figure 5. The temperature dependence of the site weighted average isomer shift obtained for the 2c site, blue squares, and the 6i site, red points, in YCoFe₃B. The solid line corresponds to a fit with the second-order Doppler shift model for the 6i site.

This general trend reflects the difference in local environment of the two inequivalent iron sites, a trend that agrees with an earlier study [24]. The volume, the local symmetry of the iron Wigner–Seitz cell as well as the iron–metalloid bonding are important factors in determining the magnitude of the iron magnetic moments in intermetallic compounds.

A plot of the reduced hyperfine field versus reduced temperature for the 2c and 6i sites is shown in figure 6(b). In this plot the Curie temperature is taken equal to 673 K and the saturation hyperfine field for the 2c and 6i sites are taken equal to 24 and 19 T, respectively. The black solid line is a Brillouin curve for a spin 5/2. It is clear that the reduced 6i hyperfine field follows well the Brillouin curve up to the spin reorientation, a reorientation that occurs at a reduced temperature of ca 0.63. The 2c reduced hyperfine field shows a somewhat different dependence.

6. Neutron diffraction results

A Rietveld refinement of the 2 and 300 K neutron diffraction patterns of $YCoFe_3B$ has been carried out on the basis of

Table 2. The 2 K Rietveld refinement and reliability parameters obtained from the powder neutron diffraction pattern of YCoFe₃B.

z 6i	0.281(2)
2c iron occupancy (%)	81.9(2)
6i iron occupancy (%)	72.8(2)
$\mu_{2c} \left(\mu_{B} \right)$	2.1(2)
μ_{6i} (μ_{B})	1.0(1)
$R_{\rm wp}$	12.1
R _{exp}	1.4
$R_{\rm Bragg}$	5.0
$R_{\rm mag}$	9.7
e	

the orientation of the iron magnetic moments obtained from the above Mössbauer spectral study. The 2 K refinement is shown in figure 7 and the resulting parameters are given in table 2. Hence, the axial orientation of the iron magnetic moment revealed by the Mössbauer spectra is also compatible with the weak magnetic scattering in the neutron diffraction patterns obtained at 2 and 300 K. Indeed, the total magnetic contribution to the diffracted intensity corresponds to only 6% of the total scattering.

The mean Fe–Co magnetic moment obtained from the 2 K refinement of the two inequivalent transition metal sites in YCoFe₃B are 2.1(2) and 1.0(1) μ_B for the 2c and 6i sites, respectively. The smaller moment observed for the 6i site confirms the large influence of the hybridization of the 6i Co(Fe) 3d orbitals with the boron 2p orbitals. Refinement of the 300 K pattern led to similar values of the mean Fe–Co magnetic moment as would be expected at a temperature that is well below the Curie temperature of 673 K.

A comparison of the diffraction patterns of YCoFe₃B, obtained below and above the spin reorientation, at 373 and 573 K is shown in figure 8. It is obvious that these two very similar patterns cannot provide a definitive indication of the presence of a spin reorientation between 373 and 573 K. The main difference between these two patterns is a small intensity reduction of the low angle (100) Bragg reflection upon increasing temperature, a reduction that is expected as the temperature approaches the Curie temperature. Other patterns have been obtained between 300 and 770 K and further support



Figure 6. (a) The temperature dependence of the site weighted average hyperfine fields of the 2c site, blue squares, and the 6i site, red points, in YCoFe₃B. (b) A reduced plot of the 2c and 6i hyperfine fields. The black solid line is a Brillouin curve for a spin 5/2. The spin reorientation occurs at a reduced temperature of 0.63 in this plot.



Figure 7. The neutron powder diffraction pattern of YCoFe₃B obtained at 2 K. The red points are the observed intensity and the solid line is the result of the fit described in the text, the vertical bars indicate the positions of the nuclear and magnetic diffraction reflections, and the bottom line is the difference between the experimental points and the calculated diffraction pattern.



Figure 8. The neutron powder diffraction pattern of YCoFe₃B obtained below the spin reorientation at 373 K, bottom, and above the spin reorientation at 573 K, top.

the conclusion that, because of the weak magnetic scattering contribution to the neutron diffraction pattern, no definitive conclusion can be reached on the direction of the magnetic moments based solely on neutron diffraction studies. However, these patterns can be satisfactorily analyzed on the basis of the direction of the iron magnetic moments obtained from the Mössbauer spectra obtained above and below the spin reorientation.

7. Discussion and conclusions

The Mössbauer spectra obtained at 400 and 450 K conclusively prove that a spin reorientation occurs in YCoFe₃B between these temperatures, a spin reorientation that is also indicated by the broad anomaly in the temperature dependence of the magnetization observed between 430 and 570 K, see figure 2. The possible existence of a spin reorientation has been postulated [12] from an individual site magnetic anisotropy model [25, 26], in which the first-order magnetic anisotropy constant, K_1 , is given by

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

where $K_1(0) = 5 \times 10^5$ J m⁻³ is the magnetic anisotropy constant for x = 0, f_{2c} and f_{6i} are the iron occupancies, 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 are the differences in the iron and cobalt contributions to the magnetic anisotropy for the 2c and 6i sites, respectively. The spin reorientation occurs for x = 3 in the YCo_{4-x}Fe_xB compounds, because, as is indicated in table 2, the 6i sites are occupied at about 75% by iron and the positive contribution of the 6i site to the magnetic anisotropy dominates the negative contribution of the 2c site.

YCo₄B is known to exhibit a spin reorientation transition as a result of the competition between the anisotropy of the two inequivalent cobalt crystallographic sites. Further, iron and cobalt are known to exhibit an opposite sign for the magnetocrystalline anisotropy in isotypic compounds. Upon the substitution of iron for cobalt in the YCo₄B structure, the iron preferentially occupies the 2c site thus reducing the uniaxial anisotropic magnetic character of the compound and, consequently YCo₃FeB and YCo₂Fe₂B exhibit planar anisotropy over the entire range of ordered temperatures. In contrast, in YCoFe₃B the larger iron content leads to a significant occupation of the 6i site by iron and to a competition between the Fe_{2c} and Fe_{6i} contributions to the magnetocrystalline anisotropy, a competition that leads to the appearance of a spin reorientation between 400 and 450 K. Again, in contrast to YCo₄B, whose easy magnetization direction lies in the basal hexagonal plane at low temperature and along the *c*-axis above the spin reorientation [9, 11, 14], YCoFe₃B exhibits uniaxial alignment of the magnetic moment below its spin reorientation and a basal anisotropy at temperatures above its spin reorientation. This observation confirms the opposite character of the anisotropy of iron and cobalt in the $YCo_{4-x}Fe_xB$ compounds.

Both the hyperfine fields obtained from the Mössbauer spectra and the magnetic moments obtained from the neutron diffraction patterns demonstrate the sensitivity of the magnitude of the iron magnetic moment to the presence of boron in its environment.

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