

Neutron diffraction determination of the magnetic structure of DyFe₆Ge₆

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Abstract. We have determined the magnetic structures of the Dy and Fe sublattices in DyFe₆Ge₆ by high-resolution neutron powder diffraction. The crystal space group is orthorhombic *Cmcm*. The Fe sublattice is antiferromagnetic with a Néel temperature of 489(1) K and it orders along the [100] direction with a magnetic space group *C₂pm'c'm'* and a propagation vector [010]. The Dy sublattice orders independently of the Fe sublattice at 7.5(1) K and comprises a ferromagnetic ordering mode along [001] and an antiferromagnetic mode along [010]. At 2 K the ferromagnetic and antiferromagnetic components of the Dy magnetic moment are 6.64(14) μ_B and 2.20(22) μ_B , respectively, yielding a net Dy moment of 6.99(15) μ_B . The respective magnetic space groups of the Dy sublattice are *F-Cm'c'm* and *AF-C₂pm'c'm'*.

1. Introduction

RFe₆Ge₆ intermetallic compounds form for R = Y and Gd–Lu and it is well established that in those RFe₆Ge₆ compounds where the R ion has a magnetic moment, the magnetic ordering processes of the R and Fe sublattices take place quite independently of one another [1–3].

The Fe sublattice orders antiferromagnetically and its Néel temperature (T_N) remains essentially constant across the series at ~ 485 K with no evidence of a net magnetization in any of the alloys. Furthermore, the hyperfine field B_{hf} at the ⁵⁷Fe nuclei, as measured by Mössbauer spectroscopy, is virtually independent of the rare earth present [3]. Neutron powder diffraction has been used to show that the easy direction of magnetic order for the Fe sublattice is along the [100] direction in the orthorhombic setting [4–8].

For R = Gd–Er, the rare-earth sublattice orders ferromagnetically with Curie temperatures (T_C) ranging from a high of 29 K at Gd to 3 K at Er [3]. In TbFe₆Ge₆ the Tb magnetic order comprises a ferromagnetic mode along [100] and an antiferromagnetic mode along [001] [6, 9]. ErFe₆Ge₆ orders ferromagnetically along [100] [4]. The case of HoFe₆Ge₆ is more complex and the magnetic ordering was described [8] in terms of microdomains, 75% of which are ordered in the (100) plane with the remaining 25% aligned along [100], i.e. perpendicular to the former microdomains.

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The magnetic independence of the R and Fe sublattices is related to the layered structure of these compounds. The RFe_6Ge_6 structures are formed by placing rare-earth atoms between the hexagonal Fe planes of the parent FeGe (B35) structure. Binary FeGe consists of ferromagnetic Fe planes coupled antiferromagnetically to each other [10] and the local environment of the R atoms in the RFe_6Ge_6 structures leads to a net cancellation of the Fe–R exchange at the rare-earth sites, effectively isolating them from the ordering of the iron moments. The magnetic ordering of the R sublattice, two orders of magnitude lower in temperature than the Fe ordering, is most likely the result of an RKKY-type coupling between R moments, which seems to take place without upsetting the magnetic order of the intervening Fe layers. This is supported by the fact that the ordering temperatures of the R sublattice scale linearly with the de Gennes factor of the R^{3+} ion and this situation is reminiscent of the R magnetic ordering in the tetragonal RFe_2Ge_2 compounds, where the R sublattice is the only magnetically ordered species [11].

In this paper we determine the magnetic modes and the magnetic space groups of the Dy and Fe sublattices in $DyFe_6Ge_6$ using high-resolution neutron powder diffraction.

2. Experimental methods

The $DyFe_6Ge_6$ samples were prepared by arc melting stoichiometric amounts of the pure elements under Ti-gettered argon. The samples were subsequently annealed at 900 °C for two weeks, sealed under vacuum in quartz tubes. Powder x-ray diffraction patterns were obtained using Cu $K\alpha$ radiation on an automated Nicolet-Stoe diffractometer. Thermogravimetric analysis was carried out on a Perkin-Elmer TGA-7 in a small magnetic field gradient to look for evidence of ferromagnetic or ferrimagnetic ordering in either the $DyFe_6Ge_6$ compound or in any impurity phases which might be present. The Néel temperature of the Fe sublattice in $DyFe_6Ge_6$ was measured by differential scanning calorimetry (DSC) on a Perkin-Elmer DSC-7, using the heat capacity peak at T_N as the signature of magnetic ordering. Magnetometry and ac-susceptometry measurements were carried out on a Quantum Design PPMS system. ^{57}Fe Mössbauer spectroscopy was carried out in constant-acceleration, transmission mode with a $^{57}CoRh$ source to determine the local Fe magnetic moment and also to check the sample purity. The results of our Mössbauer study of the entire RFe_6Ge_6 series have been reported previously [3].

Neutron powder diffraction experiments were carried out on ~4 g samples on the DUALSPEC C2 high-resolution powder diffractometer located at the NRU reactor, Chalk River Laboratories, operated by Atomic Energy Canada Limited. The neutron wavelength was 1.5049(1) Å. A detailed review of the neutron scattering facilities at Chalk River, including a description of C2, can be found in [12]. All diffraction patterns were analysed using the Rietveld method with the FULLPROF program [13].

3. Results and discussion

3.1. Crystal structure

The annealed sample of $DyFe_6Ge_6$ was virtually single phase with a trace of α -Fe present in the amount of 2 wt%, as determined from the fits to the neutron diffraction patterns. In figure 1 we show the neutron diffraction pattern of $DyFe_6Ge_6$ collected at 520 K, which is above the Néel temperature of the Fe sublattice, determined by DSC to be 489(1) K.

The crystal structure of $DyFe_6Ge_6$ is orthorhombic $TbFe_6Sn_6$ type with the space group $Cmcm$ (No 63) [14]. There is one Dy site, three Fe sites and five Ge sites. The lattice parameters determined from the neutron diffraction pattern (at 295 K) are $a = 8.1223(6)$ Å,

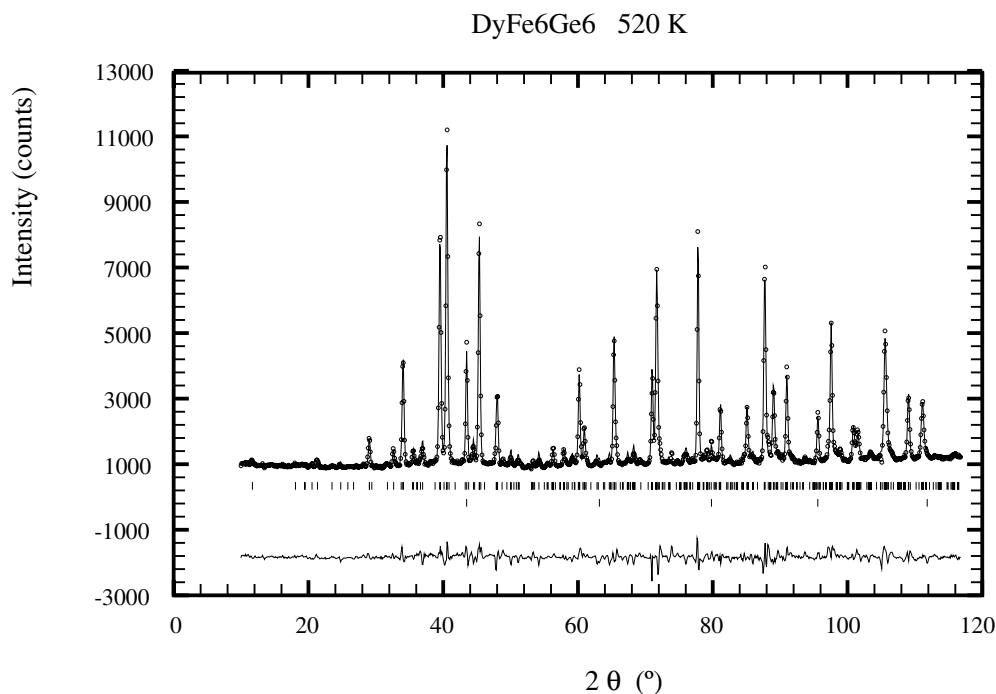


Figure 1. The neutron powder diffraction pattern of DyFe₆Ge₆ obtained at 520 K with $\lambda = 1.5049(1)$ Å. The Bragg position markers are for DyFe₆Ge₆ (top) and α -Fe (bottom).

$b = 17.7111(18)$ Å and $c = 5.1209(4)$ Å. The refinement R -factors (%) are: $R(\text{Bragg}) = 8.6$, $R(\text{F-struct.}) = 7.7$, $R(\text{wp}) = 6.9$, $R(\text{exp}) = 2.1$ and $R(\text{mag}) = 11.9$. The refined atomic position parameters are given in table 1. The lattice parameters at 520 K are $a = 8.1252(6)$ Å, $b = 17.7877(22)$ Å and $c = 5.1430(6)$ Å and at 2 K are $a = 8.1107(3)$ Å, $b = 17.6324(12)$ Å and $c = 5.1008(4)$ Å. We have employed a site disorder in fitting these patterns, as discussed in the recent reports by Oleksyn *et al* [4] and Schobinger-Papamantellos *et al* [6]. Basically, one admits the possibility of some site disorder with partial occupancy of sites displaced from the original site by $a/2$ along the [100] direction. This corresponds to the underlying FeGe channels which make up the RFe₆Ge₆ crystal structures. The site disorder affects the R (4c) and Ge (8g) sites and the relative occupancy of the $a/2$ displaced sites (which are denoted by asterisks in table 1) amounts to 11% in TbFe₆Ge₆ [6] and 13% in DyFe₆Ge₆ (this work).

3.2. Fe-sublattice ordering

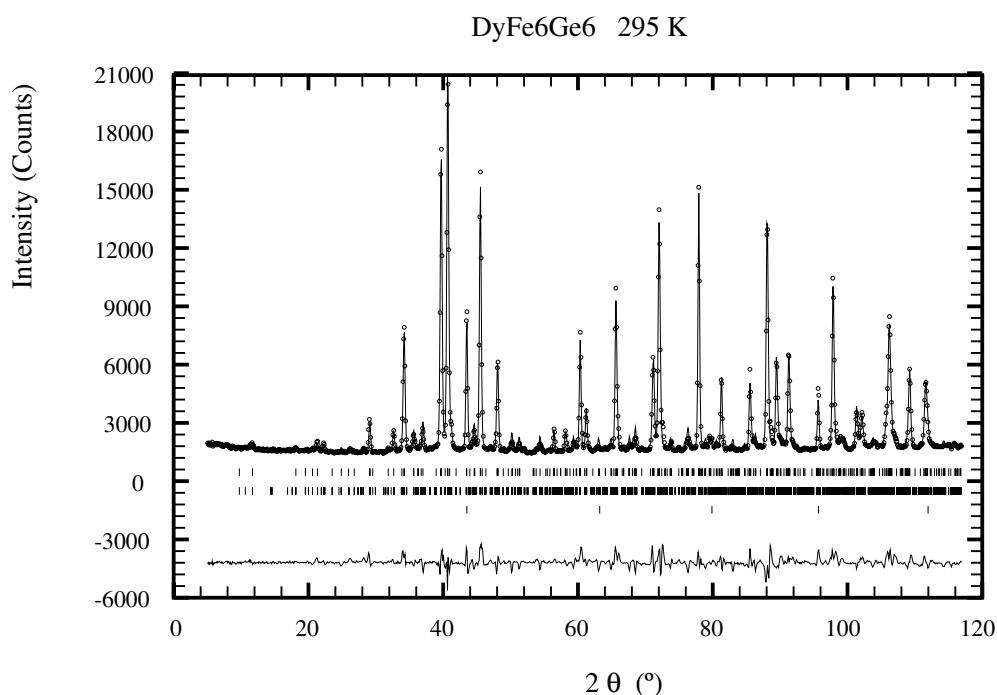
In figure 2 we show the neutron diffraction pattern of DyFe₆Ge₆ obtained at 295 K, at which temperature only the Fe sublattice is magnetically ordered.

Comparison of the neutron diffraction patterns taken above and below T_N indicated that the magnetic ordering of the Fe results in the appearance of extra peaks which may be indexed as $h+k = \text{odd}$ (nuclear scattering peaks obey $h+k = \text{even}$ for the $Cmcm$ space group). Thus, the Fe order may be described as *anti-C*; i.e. Fe moments related by the C -translation $+(\frac{1}{2}\frac{1}{2}0)$ are antiparallel.

As outlined in our previous paper on YFe₆Ge₆ [5], there are sixteen possible magnetic space groups associated with the $Cmcm$ crystal space group [15] and the observed *anti-C* order

Table 1. Atomic positions and isotropic thermal parameters for DyFe₆Ge₆

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{iso} (Å ²)
Dy	4c	0	0.1253(7)	$\frac{1}{4}$	0.57(7)
Dy*	4c	$\frac{1}{2}$	0.1253(7)	$\frac{1}{4}$	0.57(7)
Fe	8d	$\frac{1}{4}$	$\frac{1}{4}$	0	0.10(5)
Fe	8e	0.2484(11)	0	0	0.10(5)
Fe	8g	0.2531(8)	0.1201(12)	$\frac{3}{4}$	0.10(5)
Ge	4c	0	0.0484(24)	$\frac{3}{4}$	0.26(5)
Ge	4c	$\frac{1}{2}$	0.0493(23)	$\frac{3}{4}$	0.26(5)
Ge	4c	0	0.2152(22)	$\frac{3}{4}$	0.26(5)
Ge	4c	$\frac{1}{2}$	0.2124(20)	$\frac{3}{4}$	0.26(5)
Ge	8g	0.3502(6)	0.1275(6)	$\frac{1}{4}$	0.26(5)
Ge*	8g	0.8502(6)	0.1275(6)	$\frac{1}{4}$	0.26(5)

**Figure 2.** The neutron powder diffraction pattern of DyFe₆Ge₆ obtained at 295 K with $\lambda = 1.5049(1)$ Å. The Bragg position markers (top to bottom) are for DyFe₆Ge₆ nuclear scattering, DyFe₆Ge₆ Fe magnetic scattering and α -Fe, respectively.

indicates a C_p -type magnetic group, of which four members may be excluded by considering the special position of the Fe 8d site which has the crystal point group $\bar{1}$: a magnetic point group of $\bar{1}'$ is inadmissible [16]. The *anti-C* magnetic space groups, magnetic point symmetries and admissible ordering directions at the three Fe sites in RFe₆Ge₆ are shown in table 2, reproduced from our previous paper on YFe₆Ge₆. An excellent summary of the various magnetic modes supported by the $Cmcm$ space group can be found in the article by Prandl [16].

Table 2. Magnetic groups and allowed ordering directions of the Fe sublattice in DyFe₆Ge₆. A dash indicates no restriction on the allowed ordering direction.

Magnetic group	Fe(8d)	Fe(8e)	Fe(8g)	Ordering direction
C_{Pmcm}	$\bar{1}'$, none	2x	mz	None
$C_{Pm'cm}$	$\bar{1}$, none	2x	mz	None
$C_{Pmc'm}$	$\bar{1}$, —	2'yz	mz	z
$C_{Pmcm'}$	$\bar{1}$, —	2'yz	m'xy	y
$C_{Pm'c'm}$	$\bar{1}'$, none	2'yz	mz	None
$C_{Pmc'm'}$	$\bar{1}'$, none	2x	m'xy	None
$C_{Pm'cm'}$	$\bar{1}'$, none	2'yz	m'xy	None
$C_{Pm'c'm'}$	$\bar{1}$, —	2x	m'xy	x

We obtained the best fit to the 295 K neutron diffraction pattern of DyFe₆Ge₆ with the Fe moments placed along the [100] direction with a propagation vector [010], precisely as found previously with other RFe₆Ge₆ compounds (R = Y [5, 7], Er [4], Tb [6], Ho [8] and Lu [7]). The refined Fe magnetic moment in DyFe₆Ge₆ at 295 K is 1.40(9) μ_B which is in very good agreement with the value determined by Mössbauer spectroscopy (*vide infra*). The magnetic space group of the Fe sublattice in DyFe₆Ge₆ is antiferromagnetic $C_{Pm'c'm'}$.

The ⁵⁷Fe B_{hf} -value of 14.7(1) T at 295 K [3] can be translated into an Fe atomic magnetic moment if one knows the conversion factor. As discussed in our previous paper [5], Häggström *et al* [17] compiled field–moment conversion factors for a number of Fe–Ge binary compounds, taken from the literature, and found a conversion factor of 11.2(25) T/ μ_B . Adopting this value, our ⁵⁷Fe Mössbauer B_{hf} -value for DyFe₆Ge₆ corresponds to an Fe moment of 1.3(3) μ_B at 295 K, in agreement with the neutron diffraction result.

The ordering of the Fe moments along the orthorhombic *a*-axis is consistent with the magnetic order found in the parent FeGe compound. Above about 10 K, the Fe moments in FeGe order along the *c*-axis of this hexagonal B35 cell. The orthorhombic $Cmcm$ structure of DyFe₆Ge₆ is formed by stacking FeGe units such that the *a*-direction of DyFe₆Ge₆ corresponds to the *c*-axis of FeGe. The orthorhombic planar ordering of the Fe moments is also consistent with our previous arguments based on consideration of the ⁵⁷Fe quadrupole splitting measured by Mössbauer spectroscopy [2].

The neutron diffraction pattern of DyFe₆Ge₆ obtained at 15 K, which is just above the ordering temperature of the Dy sublattice, is similar to that obtained at 295 K (figure 2) and is not shown here. We observed no change in the Fe magnetic ordering mode between 295 K and 15 K. The refined Fe magnetic moment at 15 K is 1.66(8) μ_B .

3.3. Dy-sublattice ordering

In our previous Mössbauer and magnetometry study of the RFe₆Ge₆ series [3] we showed that the Dy sublattice in DyFe₆Ge₆ orders magnetically at 7.5(1) K, which we deduced by SQUID magnetometry. In figure 3 we show the neutron diffraction pattern of DyFe₆Ge₆ obtained at 2 K, which is below the Dy-sublattice ordering temperature. The magnetic *R*-factor for the Dy-sublattice contribution is 12.9%. Besides a magnetic contribution to the nuclear peaks, which indicates a ferromagnetic component to the Dy magnetic order, we also observed extra peaks in this pattern corresponding to $h + k = \text{odd}$, i.e. an antiferromagnetic component. Of the sixteen possible magnetic space groups derived from the $Cmcm$ crystal group, only three allow ferromagnetic order at the Dy site. These groups and their ordering directions are

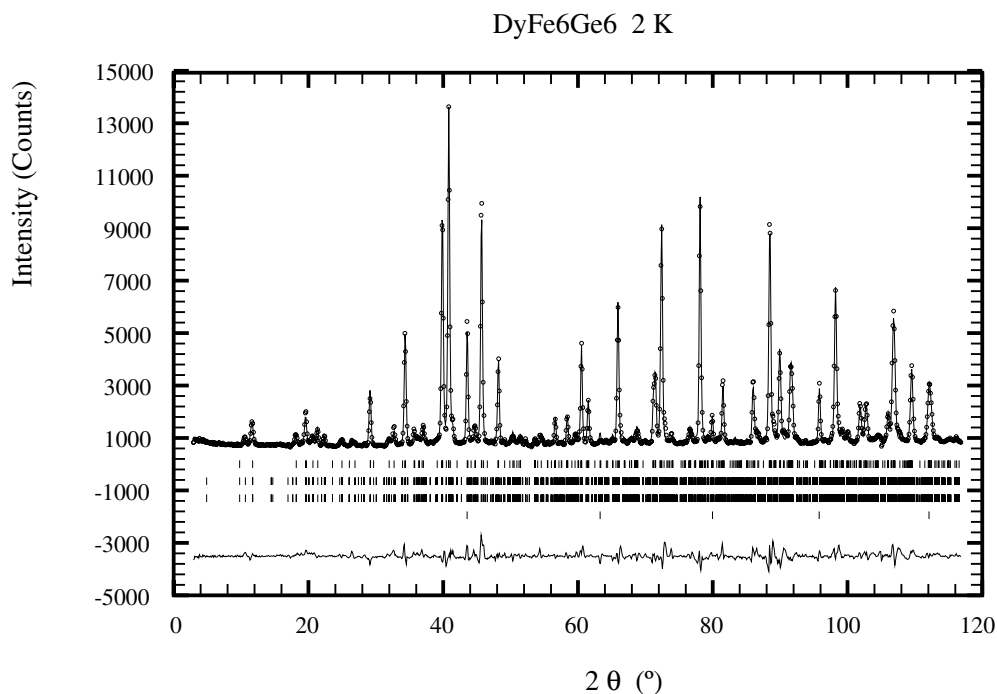


Figure 3. The neutron powder diffraction pattern of DyFe₆Ge₆ obtained at 2 K with $\lambda = 1.5049(1)$ Å. The Bragg position markers (top to bottom) are for the DyFe₆Ge₆ nuclear scattering, DyFe₆Ge₆ Fe magnetic scattering, DyFe₆Ge₆ Dy magnetic scattering and α -Fe, respectively.

$Cmc'm'$ (along [100]), $Cm'cm'$ (along [010]) and $Cm'c'm$ (along [001]). Now, for a given ferromagnetic group there are only four options for the corresponding *anti-C* antiferromagnetic group, given that the ferromagnetic and antiferromagnetic modes are orthogonal. We find that the best fit to the 2 K pattern is obtained with the Dy ferromagnetic component along [001] and the antiferromagnetic component along [010], leading to the magnetic space groups $F-Cm'c'm$ and $AF-C_{pm'cm'}$, respectively. The ferromagnetic Dy moment at 2 K is $6.64(14) \mu_B$ and the antiferromagnetic component is $2.20(22) \mu_B$, yielding a net moment of $6.99(15) \mu_B$, which is 70% of the free-ion value, appropriate to $T/T_C = 0.27$. The magnetic ordering of the Dy sublattice has no discernible effect on the Fe order and we note here that the ferromagnetic Dy magnetic ordering mode is perpendicular to the antiferromagnetic order of the Fe sublattice.

3.4. Magnetocrystalline anisotropy

As mentioned earlier, Schobinger-Papamantellos and co-workers [6] showed that TbFe₆Ge₆ has a ferromagnetic ordering mode along [100] and an antiferromagnetic mode along [001]. We have shown that isostructural DyFe₆Ge₆ has its Dy ferromagnetic order along [001], i.e. perpendicular to that of the Tb sublattice in TbFe₆Ge₆. This difference illustrates the effect of the crystal field acting on the R³⁺ ions in RFe₆Ge₆. As shown in table 3, both ions have negative second-order crystal-field terms but their fourth- and sixth-order terms have opposite signs.

Clearly, the magnetic ordering direction of the R sublattice in RFe₆Ge₆ cannot be determined solely by the second-order crystal-field terms.

Table 3. Signs of the Stevens coefficients (second-order α_J , fourth-order β_J and sixth-order γ_J) for the Tb³⁺ and Dy³⁺ ions.

R	α_J	β_J	γ_J
Tb	-	+	-
Dy	-	-	+

The magnetocrystalline anisotropy of the R³⁺ sublattice in TbFe₆Ge₆ and DyFe₆Ge₆ is determined by the crystal-field Hamiltonian appropriate to the $m2m$ point symmetry of the R³⁺ site:

$$\mathcal{H}_{cf} = B_{20}O_{20} + B_{22}^cO_{22}^c + B_{40}O_{40} + B_{42}^cO_{42}^c + B_{44}^cO_{44}^c + B_{60}O_{60} + B_{62}^cO_{62}^c + B_{64}^cO_{64}^c + B_{66}^cO_{66}^c \quad (1)$$

where the B_{nm} are the crystal-field parameters [18] and the O_{nm} are the standard Stevens spin operators [19].

The rotational transformation properties of the spin operators O_{nm} (tabulated by Rudowicz [20]) may be used to show that the anisotropy of the R³⁺ sublattice in an orthorhombic cell can be written in a phenomenological form involving anisotropy constants (K_i) [21]:

$$E_a = K_0 + (K_1 + K'_1 \cos 2\phi) \sin^2 \theta + (K_2 + K'_2 \cos 2\phi + K''_2 \cos 4\phi) \sin^4 \theta + (K_3 + K'_3 \cos 2\phi + K'''_3 \cos 4\phi + K'''_3 \cos 6\phi) \sin^6 \theta \quad (2)$$

where θ and ϕ are the polar angles of the R³⁺ magnetization relative to the crystal axes with $(\theta, \phi) = (90, 0)^\circ$, $(90, 90)^\circ$ and $(0, -)^\circ$ being the a -, b - and c -axes, respectively. The anisotropy constants are related to the crystal-field parameters by:

$$K_0 = B_{20}\langle O_{20} \rangle + B_{40}\langle O_{40} \rangle + B_{60}\langle O_{60} \rangle \quad (3)$$

$$K_1 = -\frac{3}{2}B_{20}\langle O_{20} \rangle - 5B_{40}\langle O_{40} \rangle - \frac{21}{2}B_{60}\langle O_{60} \rangle \quad (4)$$

$$K_2 = \frac{35}{8}B_{40}\langle O_{40} \rangle + \frac{189}{8}B_{60}\langle O_{60} \rangle \quad (5)$$

$$K_3 = -\frac{231}{16}B_{60}\langle O_{60} \rangle \quad (6)$$

$$K'_1 = \frac{1}{2}B_{22}^c\langle O_{20} \rangle + \frac{3}{4}[B_{42}^c\langle O_{40} \rangle + B_{62}^c\langle O_{60} \rangle] \quad (7)$$

$$K'_2 = -\frac{7}{8}[B_{42}^c\langle O_{40} \rangle - 3B_{62}^c\langle O_{60} \rangle] \quad (8)$$

$$K'_3 = \frac{33}{16}B_{62}^c\langle O_{60} \rangle \quad (9)$$

$$K''_2 = \frac{1}{8}[B_{44}^c\langle O_{40} \rangle + 5B_{64}^c\langle O_{60} \rangle] \quad (10)$$

$$K''_3 = -\frac{11}{16}B_{64}^c\langle O_{60} \rangle \quad (11)$$

$$K'''_3 = \frac{1}{16}B_{66}^c\langle O_{60} \rangle \quad (12)$$

and the corresponding anisotropy energies for the three principal directions in the orthorhombic cell are

$$E_a[100] = K_0 + K_1 + K'_1 + K_2 + K'_2 + K''_2 + K_3 + K'_3 + K''_3 + K'''_3 \quad (13)$$

$$E_a[010] = K_0 + K_1 - K'_1 + K_2 - K'_2 + K''_2 + K_3 - K'_3 + K''_3 - K'''_3 \quad (14)$$

$$E_a[001] = K_0. \quad (15)$$

Of the nine anisotropy constants needed to describe the orthorhombic symmetry (ignoring the angle-independent K_0 -term), seven depend only on the fourth- and sixth-order crystal-field terms ($K_2, K'_2, K''_2, K_3, K'_3, K''_3$ and K'''_3). Thus, the differences in the magnetic ordering modes of the Dy and Tb sublattices in the respective RFe₆Ge₆ compounds are the result of the fourth- and sixth-order crystal-field terms. Unfortunately, there are not enough data at present to deduce the nine crystal-field parameters appropriate to the RFe₆Ge₆ series.

4. Conclusions

The Fe sublattice in DyFe₆Ge₆ is antiferromagnetic with a Néel temperature of 489(1) K. We have used high-resolution neutron powder diffraction to show that the direction of Fe magnetic order is [100] and the Fe magnetic moment (at 295 K) is 1.40(9) μ_B . The Dy sublattice orders magnetically at 7.5(1) K and comprises a ferromagnetic component along [001] and an antiferromagnetic component along [010]. These two components at 2 K are 6.64(14) μ_B and 2.20(22) μ_B , respectively, yielding a total Dy moment of 6.99(15) μ_B . The magnetic ordering behaviours of the Dy and Fe sublattices are independent of each other.

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