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# Neutron diffraction determination of the magnetic structure of DyFe<sub>6</sub>Ge<sub>6</sub>

J M Cadogan<sup>†</sup>||, D H Ryan<sup>‡</sup> and I P Swainson<sup>§</sup>

 † School of Physics, The University of New South Wales, Sydney NSW 2052, Australia
 ‡ Department of Physics and Centre for the Physics of Materials, McGill University, Montreal, Canada H3A 2T8

§ Neutron Programme for Materials Research, Steacie Institute for Molecular Sciences, National Research Council, Chalk River Laboratories, Ontario, Canada K0J 1J0

E-mail: j.cadogan@unsw.edu.au

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**Abstract.** We have determined the magnetic structures of the Dy and Fe sublattices in DyFe<sub>6</sub>Ge<sub>6</sub> 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  $C_Pm'c'm'$  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)  $\mu_B$  and 2.20(22)  $\mu_B$ , respectively, yielding a net Dy moment of 6.99(15)  $\mu_B$ . The respective magnetic space groups of the Dy sublattice are F-*Cm'c'm* and AF-*C\_Pm'cm'*.

## 1. Introduction

 $RFe_6Ge_6$  intermetallic compounds form for R = Y and Gd-Lu and it is well established that in those  $RFe_6Ge_6$  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 <sup>57</sup>Fe 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 TbFe<sub>6</sub>Ge<sub>6</sub> the Tb magnetic order comprises a ferromagnetic mode along [100] and an antiferromagnetic mode along [001] [6, 9]. ErFe<sub>6</sub>Ge<sub>6</sub> orders ferromagnetically along [100] [4]. The case of HoFe<sub>6</sub>Ge<sub>6</sub> 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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<sup>||</sup> Author to whom any correspondence should be addressed.

# 8964 J M Cadogan et al

The magnetic independence of the R and Fe sublattices is related to the layered structure of these compounds. The RFe<sub>6</sub>Ge<sub>6</sub> 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<sub>6</sub>Ge<sub>6</sub> structures leads to a net cancellation of the Fe–R exchange at the rareearth 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<sup>3+</sup> ion and this situation is reminiscent of the R magnetic ordering in the tetragonal RFe<sub>2</sub>Ge<sub>2</sub> 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<sub>6</sub>Ge<sub>6</sub> 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<sub>6</sub>Ge<sub>6</sub> compound or in any impurity phases which might be present. The Néel temperature of the Fe sublattice in DyFe<sub>6</sub>Ge<sub>6</sub> 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. <sup>57</sup>Fe Mössbauer spectroscopy was carried out in constant-acceleration, transmission mode with a <sup>57</sup>Co**Rh** 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<sub>6</sub>Ge<sub>6</sub> series have been reported previously [3].

Neutron powder diffraction experiments were carried out on  $\sim 4$  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<sub>6</sub>Ge<sub>6</sub> was virtually single phase with a trace of  $\alpha$ -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<sub>6</sub>Ge<sub>6</sub> 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<sub>6</sub>Ge<sub>6</sub> is orthorhombic TbFe<sub>6</sub>Sn<sub>6</sub> 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 DyFe<sub>6</sub>Ge<sub>6</sub> obtained at 520 K with  $\lambda = 1.5049(1)$  Å. The Bragg position markers are for DyFe<sub>6</sub>Ge<sub>6</sub> (top) and  $\alpha$ -Fe (bottom).

b = 17.7111(18) Å and c = 5.1209(4) Å. The refinement *R*-factors (%) are: R(Bragg) = 8.6, R(F-struct.) = 7.7, R(wp) = 6.9, R(exp) = 2.1 and R(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 RFe<sub>6</sub>Ge<sub>6</sub> 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 TbFe<sub>6</sub>Ge<sub>6</sub> [6] and 13% in DyFe<sub>6</sub>Ge<sub>6</sub> (this work).

### 3.2. Fe-sublattice ordering

In figure 2 we show the neutron diffraction pattern of DyFe<sub>6</sub>Ge<sub>6</sub> 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 = odd (nuclear scattering peaks obey h + k = 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  $YFe_6Ge_6$  [5], there are sixteen possible magnetic space groups associated with the *Cmcm* crystal space group [15] and the observed *anti-C* order

# J M Cadogan et al

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Atom	Site	x	у	z	$B_{iso}$ (Å <sup>2</sup> )	
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)	

Table 1. Atomic positions and isotropic thermal parameters for DyFe<sub>6</sub>Ge<sub>6</sub>



**Figure 2.** The neutron powder diffraction pattern of DyFe<sub>6</sub>Ge<sub>6</sub> obtained at 295 K with  $\lambda = 1.5049(1)$  Å. The Bragg position markers (top to bottom) are for DyFe<sub>6</sub>Ge<sub>6</sub> nuclear scattering, DyFe<sub>6</sub>Ge<sub>6</sub> Fe magnetic scattering and  $\alpha$ -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  $\overline{1}$ : a magnetic point group of  $\overline{1}'$  is inadmissible [16]. The *anti-C* magnetic space groups, magnetic point symmetries and admissible ordering directions at the three Fe sites in RFe<sub>6</sub>Ge<sub>6</sub> are shown in table 2, reproduced from our previous paper on YFe<sub>6</sub>Ge<sub>6</sub>. An excellent summary of the various magnetic modes supported by the *Cmcm* space group can be found in the article by Prandl [16].

8966

Magnetic group	Fe(8d)	Fe(8e)	Fe(8g)	Ordering direction
C <sub>P</sub> mcm	$\overline{1}'$ , none	2 <i>x</i>	mz	None
$C_P m' cm$	$\overline{1}$ , none	2x	mz	None
$C_Pmc'm$	<u>1</u> , —	2'yz	mz	z
$C_Pmcm'$	<u>1</u> , —	2'yz	m'xy	у
$C_P m' c' m$	$\overline{1}'$ , none	2'yz	mz	None
$C_Pmc'm'$	$\overline{1}'$ , none	2x	m'xy	None
$C_Pm'cm'$	$\overline{1}'$ , none	2'yz	m'xy	None
$C_P m' c' m'$	ī, —	2x	m'xy	x

**Table 2.** Magnetic groups and allowed ordering directions of the Fe sublattice in DyFe<sub>6</sub>Ge<sub>6</sub>. A dash indicates no restriction on the allowed ordering direction.

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

The <sup>57</sup>Fe  $B_{hf}$ -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/ $\mu_B$ . Adopting this value, our <sup>57</sup>Fe Mössbauer  $B_{hf}$ -value for DyFe<sub>6</sub>Ge<sub>6</sub> corresponds to an Fe moment of 1.3(3)  $\mu_B$  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 DyFe<sub>6</sub>Ge<sub>6</sub> is formed by stacking FeGe units such that the *a*-direction of DyFe<sub>6</sub>Ge<sub>6</sub> 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 <sup>57</sup>Fe quadrupole splitting measured by Mössbauer spectroscopy [2].

The neutron diffraction pattern of DyFe<sub>6</sub>Ge<sub>6</sub> 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)  $\mu_B$ .

### 3.3. Dy-sublattice ordering

In our previous Mössbauer and magnetometry study of the RFe<sub>6</sub>Ge<sub>6</sub> series [3] we showed that the Dy sublattice in DyFe<sub>6</sub>Ge<sub>6</sub> orders magnetically at 7.5(1) K, which we deduced by SQUID magnetometry. In figure 3 we show the neutron diffraction pattern of DyFe<sub>6</sub>Ge<sub>6</sub> 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 = 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  $DyFe_6Ge_6$  obtained at 2 K with  $\lambda = 1.5049(1)$  Å. The Bragg position markers (top to bottom) are for the  $DyFe_6Ge_6$  nuclear scattering,  $DyFe_6Ge_6$  Fe magnetic scattering,  $DyFe_6Ge_6$  Dy magnetic scattering and  $\alpha$ -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'mand 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  $TbFe_6Ge_6$  has a ferromagnetic ordering mode along [100] and an antiferromagnetic mode along [001]. We have shown that isostructural DyFe<sub>6</sub>Ge<sub>6</sub> has its Dy ferromagnetic order along [001], i.e. perpendicular to that of the Tb sublattice in  $TbFe_6Ge_6$ . This difference illustrates the effect of the crystal field acting on the  $R^{3+}$  ions in  $RFe_6Ge_6$ . 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 RFe<sub>6</sub>Ge<sub>6</sub> cannot be determined solely by the second-order crystal-field terms.

**Table 3.** Signs of the Stevens coefficients (second-order  $\alpha_J$ , fourth-order  $\beta_J$  and sixth-order  $\gamma_J$ ) for the Tb<sup>3+</sup> and Dy<sup>3+</sup> ions.

R	$\alpha_J$	$\beta_J$	γJ
Tb	_	+	_
Dy	-	-	+

The magnetocrystalline anisotropy of the  $R^{3+}$  sublattice in TbFe<sub>6</sub>Ge<sub>6</sub> and DyFe<sub>6</sub>Ge<sub>6</sub> is determined by the crystal-field Hamiltonian appropriate to the *m*2*m* point symmetry of the  $R^{3+}$  site:

$$\mathcal{H}_{cf} = B_{20}O_{20} + B_{22}^cO_{22}^c + B_{40}O_{40} + B_{42}^cO_{42}^c + B_{44}^cO_{44}^c + B_{60}O_{60} + B_{62}^cO_{62}^c + B_{64}^cO_{64}^c + B_{66}^cO_{66}^c$$
(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 R<sup>3+</sup> sublattice in an orthorhombic cell can be written in a phenomenological form involving anisotropy constants ( $K_i$ ) [21]:

$$E_{a} = K_{0} + (K_{1} + K'_{1} \cos 2\phi) \sin^{2}\theta + (K_{2} + K'_{2} \cos 2\phi + K''_{2} \cos 4\phi) \sin^{4}\theta + (K_{3} + K'_{3} \cos 2\phi + K''_{3} \cos 4\phi + K'''_{3} \cos 6\phi) \sin^{6}\theta$$
(2)

where  $\theta$  and  $\phi$  are the polar angles of the R<sup>3+</sup> 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:

$$K_0 = B_{20} \langle O_{20} \rangle + B_{40} \langle O_{40} \rangle + B_{60} \langle O_{60} \rangle \tag{3}$$

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

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

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

$$K_1' = \frac{1}{2} B_{22}^c \langle O_{20} \rangle + \frac{3}{4} \left[ B_{42}^c \langle O_{40} \rangle + B_{62}^c \langle O_{60} \rangle \right]$$
(7)

$$K_{2}^{\prime} = -\frac{7}{8} \left[ B_{42}^{c} \langle O_{40} \rangle - 3B_{62}^{c} \langle O_{60} \rangle \right]$$
(8)

$$K'_{3} = \frac{33}{16} B^{c}_{62} \langle O_{60} \rangle \tag{9}$$

$$K_2'' = \frac{1}{8} \left[ B_{44}^c \langle O_{40} \rangle + 5 B_{64}^c \langle O_{60} \rangle \right]$$
(10)

$$K_3'' = -\frac{11}{16} B_{64}^c \langle O_{60} \rangle \tag{11}$$

$$K_3^{\prime\prime\prime} = \frac{1}{16} B_{66}^c \langle O_{60} \rangle \tag{12}$$

8970 J M Cadogan et al

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

$$E_a[100] = K_0 + K_1 + K_1' + K_2 + K_2' + K_2'' + K_3 + K_3' + K_3'' + K_3'''$$
(13)

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

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

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 RFe<sub>6</sub>Ge<sub>6</sub> 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 RFe<sub>6</sub>Ge<sub>6</sub> series.

# 4. Conclusions

The Fe sublattice in DyFe<sub>6</sub>Ge<sub>6</sub> 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)  $\mu_B$ . 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)  $\mu_B$  and 2.20(22)  $\mu_B$ , respectively, yielding a total Dy moment of 6.99(15)  $\mu_B$ . The magnetic ordering behaviours of the Dy and Fe sublattices are independent of each other.

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