ELSEVIER

Journal of Alloys and Compounds 223 (1995) 18–21

Journal of ALLOYS AND COMPOUNDS

Identification of a faulted carbide phase in an annealed $Nd_{10}Dy_6Fe_{76}B_1C_7$ cast magnet

Y.J. Zhang *, A.S. Murthy, Y.Z. Wang **, G.C. Hadjipanayis

Department of Physics and Astronomy, University of Delaware, Newark, DE 19716, USA

Received 27 June 1992; in final form 17 October 1994

Abstract

A rare-earth-rich iron carbide phase commonly observed in annealed cast magnets of $Nd_{10}Dy_6Fe_{76}B_1C_7$ composition has been characterized using electron diffraction. The crystal structure is identified as hexagonal with unit cell parameters $a = 0.462$ nm, $c = 0.787$ nm and $c/a \approx 1.7$. The ratio of R to Fe is estimated to be about 2/3.

Keywords: Faulted carbide phase; Annealing; Cast magnets

1. Introduction

Recently high coercivities have been obtained in as-cast R–Fe–C alloys [1–5]. However, the expected $R_2Fe_{14}C$ phase, which crystallizes directly from the melt, is obtained through a transformation from the 2:17 phase by annealing in a narrow range of temperatures [7]. Because the carbides are less stable than the borides, multiple phases are normally formed after annealing and a rare-earth-rich carbide phase is commonly found besides the major 2:14:1 carbide phase as reported by de Boer et al. [4] and Fidler et al. [6]. It was suggested that the latter phase could be indexed with the cubic R_2C_3-structure but this is not unambiguously confirmed. In this work, we studied the structures of the various phases present in the Nd–Dy–Fe–B–C system and in the present paper we report the crystal structure of a new phase.

2. Experimental

As-cast samples with a nominal composition $Nd_{10}Dy_6Fe_{76}B_1C_7$ were prepared by arc-melting. The samples were annealed at 850 °C for 24 h in vacuum. The magnetic properties were measured with a vibrating sample magnetometer. The microstructural studies were carried out by transmission electron microscopy using a Jeol JEM 100C microscope.

3. Results and discussion

A characteristic phase has been commonly observed in all samples studied. We believe that this is a carbide phase because excess carbon was used in the cast to form the 2:14:1 phase and because this phase has never been reported before in the R–Fe–B system. The ratio of rare earth to iron content in this carbide is determined to be 2:3 using energy dispersive X-ray spectroscopy (EDS). The ratio of the two rare-earth elements Dy to Nd is about 0.72 (as compared with the ratio of 2:1 in the major 2:14:1 carbide phase).

The microstructure (Fig. 1(a) and (b)) of the carbide phase in the annealed cast $Nd_{10}Dy_6Fe_{76}B_1C_7$ magnet is highly faulted, a feature similar to that reported in Ref. [6]. In order to identify its structure, three-dimensional information on its reciprocal lattices is required. For this purpose three systematic electron diffraction patterns were obtained which share a common crystal axis. The patterns are shown in Fig. 2(a)–(c). These diffraction patterns are of respective zone axes [010], [$\bar{1}$10] and [$\bar{2}$10], all sharing a common crystallographic axis with reciprocal lattice vector along (001). Their related reciprocal planes are schematically drawn in Fig. 3(a)–(c) and the related intersections of Edwald spheres with the three-dimensional reciprocal lattices

* Present address: International Research and Development Corporation, Eagan, MN 55122, USA.

** Present address: San Huan, Inc., Chinese Academy of Sciences, Box 603, Beijing 100080, People's Republic of China.

SSDI 0925-8388(94)01474-4

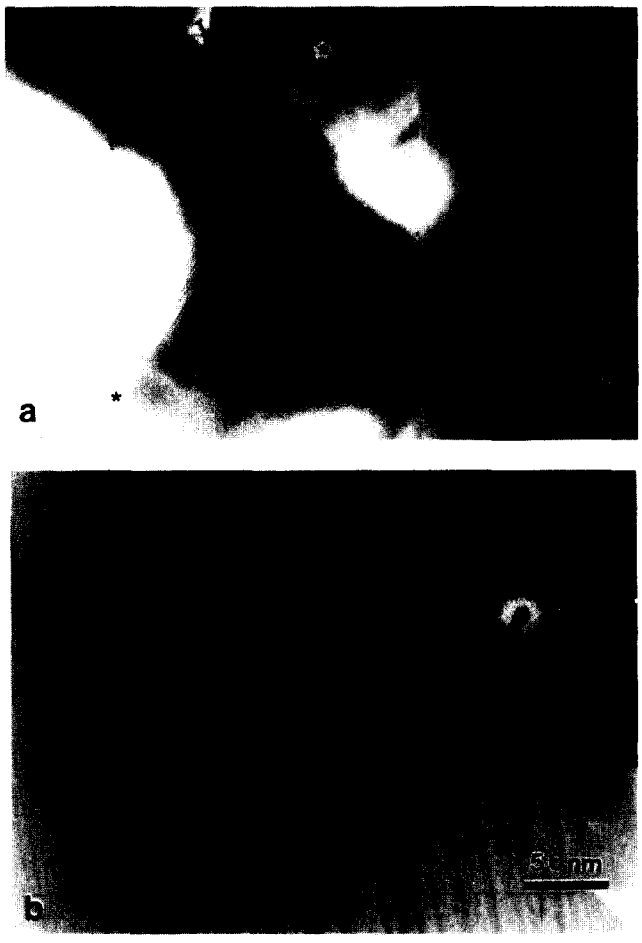

Fig. 1. (a) Highly faulted features can be seen in the carbide phase (A). The grains marked with stars are 2:14:1 carbides. (b) More details of the faults with the diffraction pattern are shown on the top right.

are shown in Fig. 3(d). From this depiction another cross-section of the reciprocal lattice CBA perpendicular to the tilting axis is obtained as shown in Fig. 4(a). R_1, R_2, R_3 can be determined from the diffraction patterns shown in Fig. 2(a)–(c). To decide the basic reciprocal plane perpendicular to the common axis, angle A and length $l/2$ must be known. These can be found from the following relations:

$$\sin(\pi - A) = \frac{1}{l}(R_3 \sin C) \quad (1)$$

$$\cos C = \frac{1}{2R_1R_3}\{4R_2^2 - (R_1^2 + R_3^2)\} \quad (2)$$

The value of cos C can be obtained from the three known lengths R_1, R_2, R_3, using Eq. (2). After measuring R_1, R_2 and R_3 with high accuracy, the angle A was calculated and found to be 60°. Since $R_2/R_1 = \sqrt{3}$ and $A = 60°$, it is easily concluded that $l/2 = R_1$. The reciprocal plane perpendicular to the common axis is thus determined and its corresponding real lattices are simply decided by the principles of reciprocity as shown in Fig. 4(c). The structure of this carbide is analyzed to be hexagonal. In order to obtain reliable lattice parameters for a and c, the ratio c/a was first determined from the same diffraction pattern to avoid any errors introduced by the uncertainty in the camera length of different diffraction patterns. Then the lattice parameter a was found from one pattern for which the camera length was determined from a known 2:14:1 diffraction pattern. The final result indicates that $a = 0.462$ nm, $c = 0.787$ nm with $c/a \approx 1.7$. For convenience in indexing the diffraction patterns, the calculated d-spacings are listed in Table 1.

It is worth noting the two unique features of the electron diffraction patterns obtained for this carbide phase. First, the streaks, which normally flank the major diffracted spots and are related to stacking faults along planes perpendicular to the reciprocal lattice vector, now fall on the superlattice spots of the diffraction pattern (see Fig. 2(a)–(c)). This may be due to the anti-phase boundaries formed along the superlattices

Fig. 2. (a)–(c) Three diffraction patterns of the faulted crystals sharing a common axis. Note the unique features of the diffractions. The streaks do not flank the major spots; satellite spots do not appear at the center of the basic reciprocal rectangles.

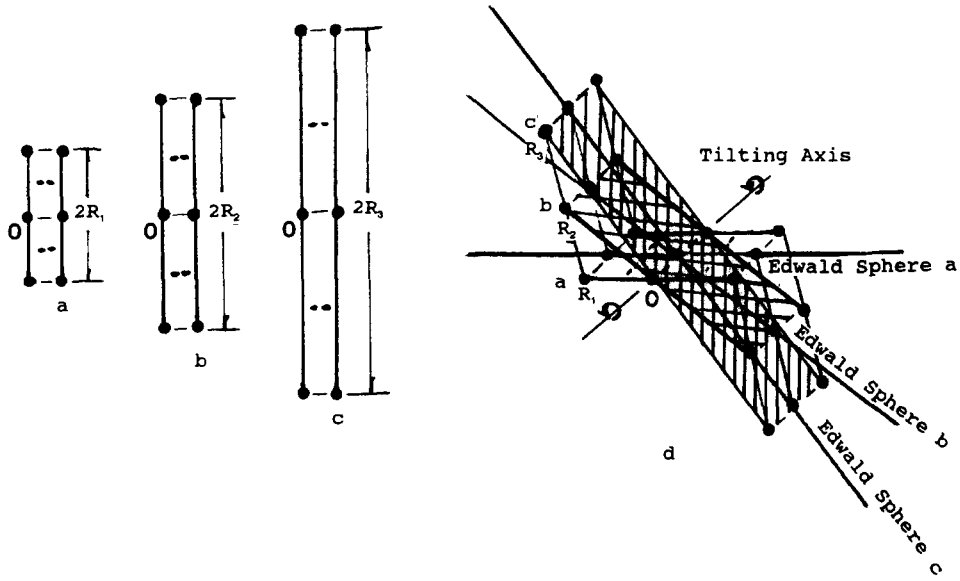

Fig. 3. (a)–(c) Schematic drawings of the diffraction patterns are shown. These patterns are the intersections of related Edwald spheres with different reciprocal lattice planes as depicted in (d).

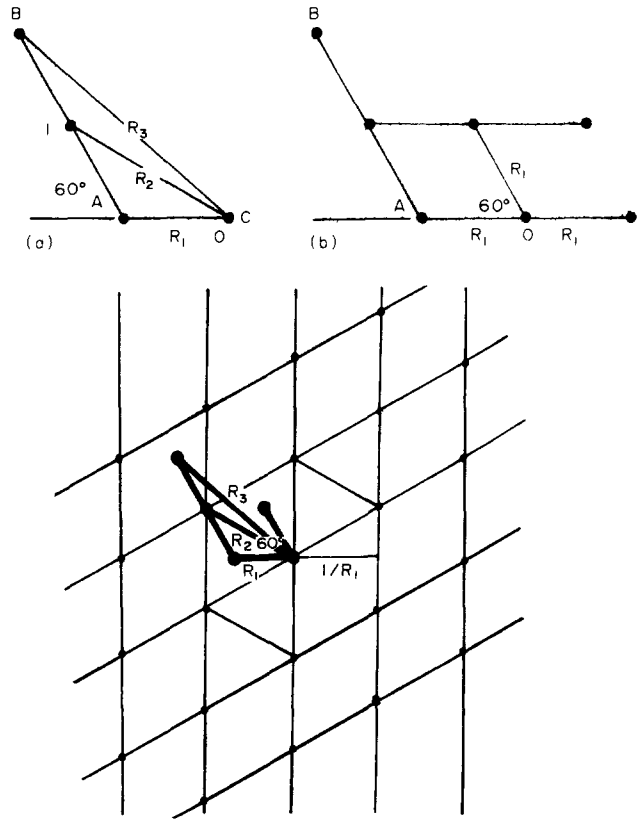

Fig. 4. The reciprocal plane perpendicular to the tilting axis is drawn ((a) and (b)). Angle A and length l can be determined from the measurements of R_1, R_2 and R_3. (c) The reciprocal lattices and real lattices can be decided by simple reciprocity.

with twice the spacing of the c-planes. The second unique feature is that the superlattice diffractions do not appear at the center of the basic reciprocal rectangles. This might be related to a short-range order

Table 1
Interplanar spacings of hexagonal $(Nd_{0.58}Dy_{0.42})_2Fe_3C_x$; lattice parameters: $a=0.462$ nm, $b=0.7869$ nm

$h\ k\ l$	Predicted d-spacings (Å)
0 0 1	7.869
0 0 2	3.934
0 1 1	3.565
0 1 2	2.805
0 0 3	2.623
1 1 0	2.309
0 1 3	2.193
0 2 0	2.000
1 1 2	1.991
0 0 4	1.967
0 2 1	1.938
0 2 2	1.783
0 1 4	1.765
1 1 3	1.733
0 2 3	1.590
0 0 5	1.574
1 2 0	1.511
1 1 4	1.497
1 2 1	1.484
0 1 5	1.464
1 2 2	1.411
0 3 0	1.333
0 3 1	1.314
1 2 3	1.310
0 0 6	1.311
1 1 5	1.300
0 3 2	1.263
0 1 6	1.246
• • •	•
• • •	•
• • •	•

or vacancy phenomenon [8–10]. Further research is under way to clarify the origin of the extra diffraction spots.

Acknowledgements

This work was supported by U.S. Army Research.

References

[1] H.H. Stadelmaier and H.K. Parl, *Z. Metallkd., 72* (1981) 417.
[2] N.C. Liu and H.H. Stadelmaier, *Mater. Lett., 4* (1986) 377.
[3] A.T. Pedziwiatr, W.E. Wallace and E. Burzo, *J. Magn. Magn. Mater., 59* (1986) L179.
[4] F.R. de Boer, Huang Ying-Kai, Zhang Xhi-Dong, D.B. de Mooij and K.H.J. Buschow, *J. Magn. Magn. Mater., 72* (1988) 167.
[5] M. Gueramian, A. Bezinge, K. Yven and J. Muller, *Solid State Commun., 64* (1987) 639.
[6] J. Fidler, C. Groiss, J. Eisses and K.H.J. Buschow, in *Proc. 11th Int. Symp. on Magnetic Anisotropy and Coercivity in Rare-Earth–Transition Metal Alloys, Pittsburgh, PA, 1990,* pp. 549.
[7] M.C. Zhang, Y.Z. Wang, Y.J. Zhang and G.C. Hadjipanayis, to be published.
[8] P.R. OlLiomoto and G. Thomas, *Mat. Res. Bull., 6* (1971) 45; P.R. OlLiomoto and G. Thomas, *Acta Met., 19* (1971) 825.
[9] L.E. Tanner, *Phys. Status Solidi, 30* (1968) 685; L.E. Tanner, *Philos. Mag., 14* (1966) 111.
[10] L.E. Tanner, P.C. Clapp and R.S. Toth, *Mat. Res. Bull., 3* (1968) 855.