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1993 J. Phys.: Condens. Matter 5 L457

(http://iopscience.iop.org/0953-8984/5/38/001)

J. Phys.: Condens. Matter 5 (1993) L457–L460. Printed in the UK

LETTER TO THE EDITOR

A new quaternary phase in the Nd–Co–B–Si system

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Received 28 July 1993

Abstract. A new compound of overall composition $NdCo_8B_3Si$ with a tetragonal structure has been synthesized by arc-melting, and identified by x-ray powder diffraction and analytical electron microscopy.

There are several phases in the Nd–Co–B system including $NdCo_4B$, $NdCo_2B_2$, $NdCo_4B_4$, $NdCo_{12}B_6$ etc [1]. In the Nd–Co–Si system a phase of $NdCo_9Si_2$ has been reported [2]. However, no quaternary phases have been reported in an Nd–Co–B–Si system. A new phase with an overall composition of $NdCo_8B_3Si$ has been discovered. This is the first compound found in the Nd–Co–B–Si system, which fills a gap between the Nd–Co–B system and the Nd–Co–Si system, and provides scope for the synthesis of a range of related compounds. The results of x-ray powder diffraction and analytical electron microscopy of the new phase are presented in this paper.

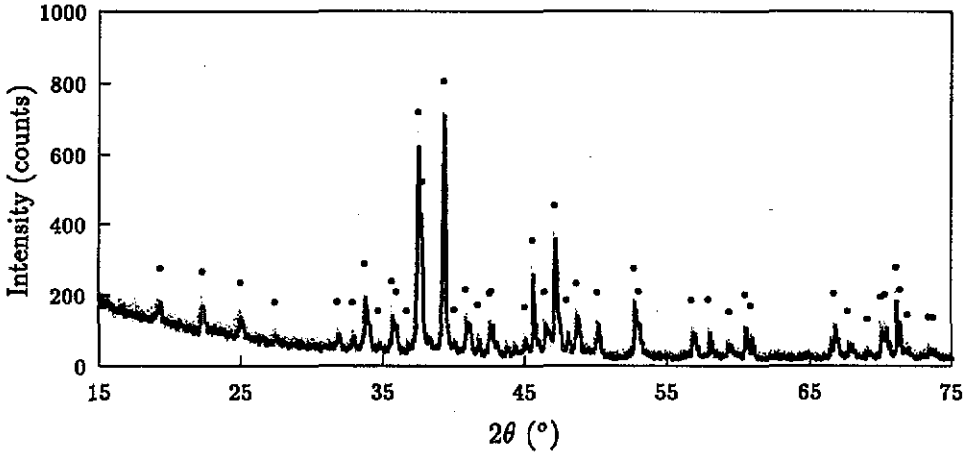

Figure 1. X-ray (Cu Kα) powder diffraction pattern of $NdCo_8B_3Si$; the • on the peaks indicates that they have been indexed.

The sample of $NdCo_8B_3Si$ was prepared from lumps of 99.99% Nd, 99.998% Co, 99.5% B and 99.7% Si by arc-melting and annealing at 900 °C for three weeks. The weight loss during the sample preparation was $\leq 1\%$. The density of the sample was determined as 7.7 ± 0.3 g cm^{-3}. Further details of the preparation procedure can be found in our previous paper [3]. The x-ray powder diffraction studies were made using Cu Kα radiation in a

Philips diffractometer. The powder pattern was indexed by a PowdMult indexing program [4]. A standard transmission electron microscope thin-film specimen was prepared using an electropolisher. The electron microscopy was performed on a Hitachi H800 analytical electron microscope.

Figure 2. SEM image of the $NdCo_8B_3Si$ film (×100).

The x-ray powder diffraction pattern of $NdCo_8B_3Si$ is shown in figure 1. The corresponding observed and indexed d-spacings and their intensities are listed in table 1. The x-ray diffraction pattern was indexed on the basis of a tetragonal unit cell. As shown in figure 1 and table 1, nearly all the measured reflections are well indexed, and there is very good agreement between the observed and calculated interplanar spacings. The tetragonal lattice parameters for $NdCo_8B_3Si$ calculated from the indexing are $a = 11.184(\pm 0.005)$ Å, and $c = 7.953(\pm 0.005)$ Å. The relation between the a axis and c axis ($a/c \simeq \sqrt{2}$) suggests that the tetragonal unit-cell could be derived from two cubic cells with their two [110] axes alternated into the a axis. The appropriate lattice parameter ($a = 7.2986$ Å) and the similarity in the positions of the strongest peaks (2.201 Å (311) and 2.580 Å (220)) for the cubic $NdCo_2$ (space group, $Fd3m$) indicates that the arrangement of the dominant component Co in a derived tetragonal $NdCo_8B_3Si$ unit cell could in general follow that of the Co in the corresponding two cubic $NdCo_2$ unit cells. As no systematic absences have

Table 1. The x-ray powder diffraction data for $NdCo_8B_3Si$.

Measured		Calculated	
I/I_0	d (Å)	d (Å)	$h\,k\,l$
8	4.585	4.574	2 0 1
12	3.978	3.976	0 0 2, 2 2 0
9	3.556	3.553	1 1 2, 2 2 1
4	3.246	3.241	2 0 2, 3 1 1
7	2.809	2.804	2 2 2, 4 0 0
7	2.719	2.720	3 0 2, 4 1 0
23	2.651	2.651	0 0 3
3	2.581	2.580	1 0 3
16	2.514	2.514	1 1 3
11	2.500	2.501	3 3 1, 4 2 0
4	2.445	2.446	3 2 2
87	2.396	2.395	2 0 3
58	2.384	2.386	4 2 1
100	2.290	2.287	4 0 2
4	2.248	2.241	4 1 2
13	2.200	2.202	2 2 3, 3 3 2
6	2.162	2.160	3 0 3
12	2.123	2.121	3 1 3
13	2.117	2.117	4 2 2, 5 1 1
7	2.010	2.010	3 2 3, 5 2 1
35	1.989	1.988	0 0 4
13	1.955	1.958	1 0 4, 5 0 2, 4 3 2
49	1.927	1.928	1 1 4, 4 0 3
9	1.894	1.896	4 1 3
17	1.869	1.869	3 3 3
13	1.818	1.819	4 2 3
24	1.735	1.733	3 1 4
14	1.726	1.726	5 3 2, 6 2 1
11	1.621	1.620	4 0 4, 6 2 2
11	1.591	1.591	0 0 5, 3 3 4
6	1.555	1.554	4 2 4, 5 3 3, 7 1 1
13	1.530	1.530	2 0 5
8	1.521	1.522	6 4 1
14	1.401	1.401	4 4 4, 8 0 0
7	1.383	1.382	4 0 5, 5 3 4
3	1.358	1.359	6 0 4, 5 5 3, 7 1 3
13	1.343	1.342	4 2 5
14	1.338	1.339	6 4 3
25	1.326	1.326	0 0 6
16	1.322	1.321	6 2 4
5	1.312	1.312	5 4 4
4	1.289	1.290	8 3 1, 2 0 6, 5 1 5
4	1.284	1.284	8 2 2

been observed in the indexing, this kind of arrangement will lead to the possible space group of $P\bar{4}m2$ or $P\bar{4}$ for $NdCo_8B_3Si$. Further analysis of the structure is in progress.

The morphology (figure 2) of the $NdCo_8B_3Si$ film observed in the SEM mode shows a typical single-phase feature of homogeneous grains, with grain boundaries readily observed due to preferential etching within the grains. The energy dispersive spectrum (EDS) analysis of the film shows no detectable difference in composition of Nd and Co between different

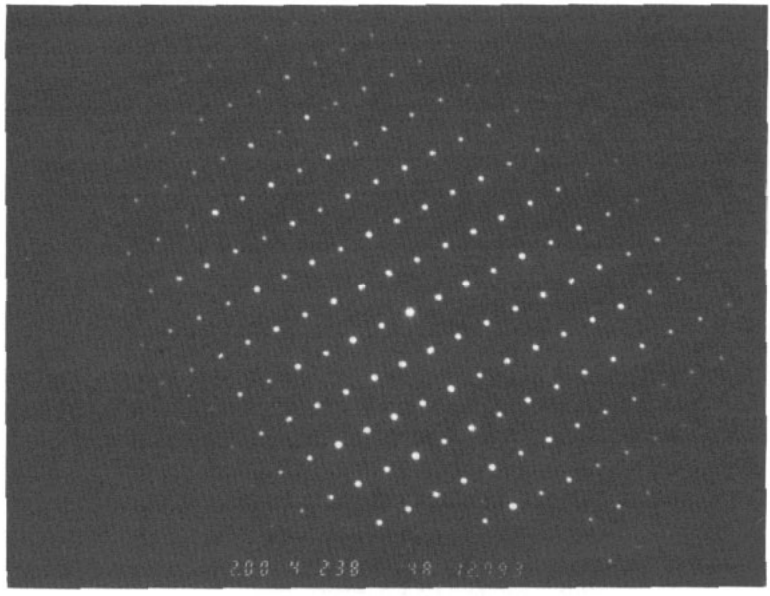

Figure 3. Electron diffraction pattern of $NdCo_8B_3Si$ with the beam directed along the zone axis [010].

grains and between grain and boundaries. Analysis of EDS data provides qualitative agreement with the overall compositional ratio 1:8 between Nd and Co respectively, as determined by stoichiometry during sample preparation. Si is also detected but its quantity can not be defined.

Selected area electron diffraction (SAD) analyses confirm that the grains are single crystals of identical phase. The diffraction patterns obtained from SAD of the single crystals are indexed very well with the tetragonal unit cell. Figure 3 shows an electron diffraction pattern taken with the beam directed along the zone axis [010], revealing diffraction from both (100) and (001) planes. Using superposed Cr film as a standard, the lattice parameters of the unit cell were calculated as $a = 11.15(\pm 0.06)$ Å and $c = 7.95(\pm 0.03)$ Å. The shortest reciprocal axis with spacing of 11.15 Å (100) observed in SAD is in good agreement with the corresponding a axis (11.184 Å), the widest d-spacing calculated from XRD, and indicates the consistency between x-ray and electron diffraction analyses.

The authors thank Mr X L Zhao and Dr A V J Edge for assistance in sample preparation. This work is supported by a grant from the Australian Research Council.

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