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## Neutron diffraction study of $RFe_{10}V_2C_x$ prepared by arc melting

Chin Lin<sup>†</sup>, Xiao-Feng Xu<sup>†</sup>, Yun-Xi Sun<sup>†</sup>, Zun-Xiao Liu<sup>†</sup>, Ji-Lian Yang<sup>‡</sup>,  
Bai-Sheng Zhang<sup>‡</sup>, Hong-Lin Du<sup>‡</sup> and Chun-Tang Ye<sup>‡</sup>

<sup>†</sup> Department of Physics, Peking University, Beijing, People's Republic of China

<sup>‡</sup> Institute of Atomic Energy, Academia Sinica, Beijing, People's Republic of China

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**Abstract.** Samples of  $RFe_{10}V_2C_x$  with  $R = Y, Gd, Dy$  or  $Er$  were prepared by arc melting. X-ray diffraction and thermomagnetic analyses showed that interstitial carbides with the  $ThMn_{12}$  structure can be formed only for a small amount of C ( $x \leq 0.3$ ). The effects of C addition on lattice expansion and Curie temperature are much smaller in the carbides prepared by arc melting than in those prepared by gas–solid reaction. Neutron diffraction shows that the C atoms occupy the interstitial site in the structure.

### 1. Introduction

Interstitial nitrogen (carbon) atoms improve the magnetic properties of  $R_2Fe_{17}$  and  $R(Fe, M)_{12}$  ( $R = Ti, V, Mo, Cr, Si$  or  $W$ ) compounds markedly [1–6]. The interstitial nitrides  $R_2Fe_{17}N_{3-\delta}$  and  $R(Fe, M)_{12}N_{1-\delta}$ , and carbides  $R_2Fe_{17}C_{3-\delta}$  and  $R(Fe, M)_{12}C_{1-\delta}$  are promising permanent magnet materials. They are usually generated by the gas–solid reaction. An important restriction on any process aimed at producing a high-performance magnet is the disproportionation at high temperatures. Above  $650^\circ C$ , they decompose into a mixture of  $\alpha$ -Fe and RN.  $R_2Fe_{17}C_x$  compounds can be formed by arc melting only for  $x \leq 1.5$  [7]. Stable  $R_2Fe_{17}C_x$  compounds with higher C concentration can be prepared by melt spinning [8]. We try to introduce C atoms into the  $R(Fe, M)_{12}$  compounds by arc melting and study the site preference by neutron diffraction. The magnetic properties of  $RFe_{10}Si_2C_x$  have been reported [9]. In the  $ThMn_{12}$  structure, Si atoms strongly prefer the 8f site, although they also occupy the 8i and 8j sites [10]. V atoms have a strong preference for occupying the 8i site [11]. In this work we present the structural and magnetic properties of  $RFe_{10}V_2C_x$  and study the site preference of C atoms.

### 2. Experiment

Samples of  $RFe_{10}V_2C_x$  with  $R=Y, Gd, Dy$  or  $Er$ ,  $x = 0, 0.1, 0.3$  and  $0.5$ , were prepared by arc melting the appropriate amount of the constituents under a purified argon atmosphere. These samples were annealed in vacuum for 1 month at  $950^\circ C$  and quenched. X-ray diffraction and thermomagnetic analyses showed that the samples are almost single phase with the  $ThMn_{12}$  structure when  $x \leq 0.3$ . Small amounts of  $\alpha$ -Fe and  $R_2Fe_{17}$  are found in some samples. When  $x = 0.5$ ,  $\alpha$ -Fe becomes the main phase.

The magnetic properties were studied at fields up to 70 kOe in the temperature range from 1.5 K to room temperature. Powdered samples were aligned in a field of 10 kOe and fixed in epoxy resin. Magnetization curves were measured with the measuring field applied parallel ( $M_{\parallel}$ ) and perpendicular ( $M_{\perp}$ ) to the alignment direction. Thermomagnetic curves at high temperatures were measured with a vibrating-sample magnetometer operating at a low field. The Curie temperature was determined by plotting  $M^2$  versus  $T$  near  $T_C$  and extrapolating the linear part to  $M = 0$ . The AC susceptibility was measured with a mutual inductance bridge with a two-phase lock-in amplifier.

The C atom occupation preference was determined by neutron diffraction experiments. Neutron diffraction data were collected on a two-axis neutron diffractometer with  $\lambda = 1.184 \text{ \AA}$  at the Institute of Atomic Energy in Beijing. The data were analysed by the Rietveld method.

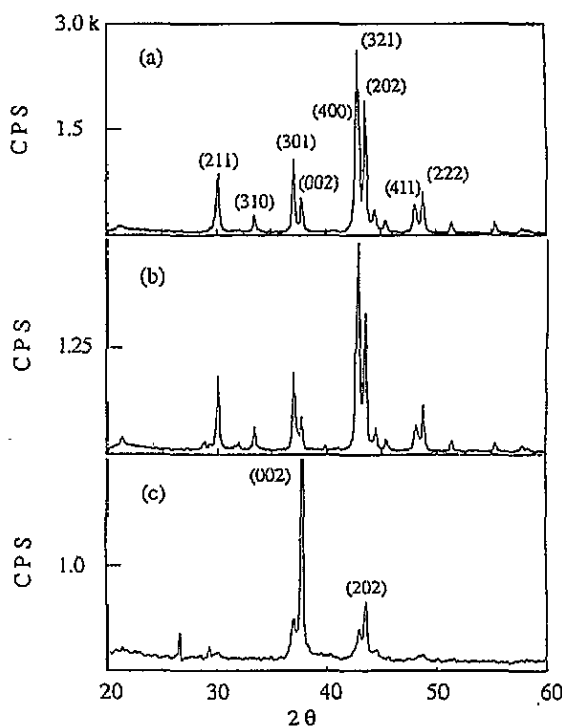


Figure 1. X-ray diffraction patterns of  $\text{ErFe}_{10}\text{V}_2\text{C}_x$ : (a)  $x = 0$ ; (b)  $x = 0.3$ ; (c)  $x = 0.3$ , magnetically aligned.

### 3. Results and discussion

X-ray diffraction patterns are shown in figures 1(a), 1(b) and 1(c) for  $\text{ErFe}_{10}\text{V}_2$ ,  $\text{ErFe}_{10}\text{V}_2\text{C}_{0.3}$  and magnetically aligned  $\text{ErFe}_{10}\text{V}_2\text{C}_{0.3}$ , respectively. The x-ray diffraction patterns of C-containing samples are identical with those of the parent compounds except the small shift towards a small angle. X-ray diffraction analyses on aligned powders indicate that all samples have the easy-magnetization direction parallel to the  $c$  axis at room temperature. The lattice parameters, saturation magnetizations at 1.5 K and Curie temperatures for  $\text{RFe}_{10}\text{V}_2\text{C}_x$  are listed in table 1. The lattice expansion is much smaller

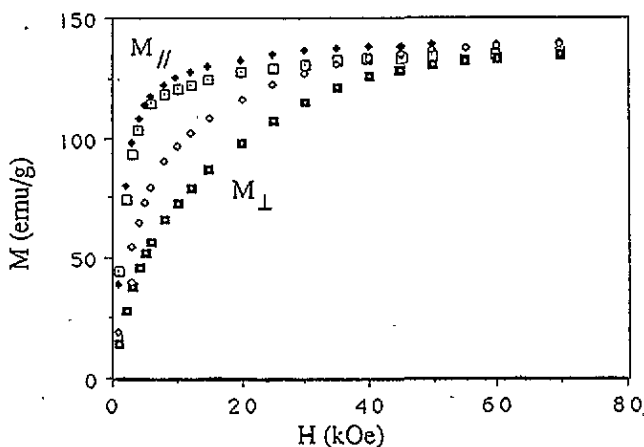


Figure 2. Magnetization curves for  $YFe_{10}V_2C_x$  at 1.5 K with  $x = 0$  ( $\square$ ,  $\blacksquare$ ) and 0.3 ( $\diamond$ ,  $\blacklozenge$ ).  $M_{\parallel}$  and  $M_{\perp}$  represent directions parallel and perpendicular, respectively to the alignment direction.

than that given by Hurley and Coey [5]. It is in agreement with the fact that only a small amount of C can be added to  $RFe_{10}V_2C_x$  by arc melting. The saturation magnetization and the Curie temperature increase slightly with increasing C content.

Table 1. Lattice parameters, saturation magnetizations at 1.5 K, and Curie temperatures of  $RFe_{10}V_2C_x$ .

R	$x$	$a$ ( $\text{\AA}$ )	$c$ ( $\text{\AA}$ )	$V$ ( $\text{\AA}^3$ )	$M$ ( $\mu_B/\text{FU}$ )	$T_C$ (K)
Y	0.0	8.499	4.773	344.8	17.7	593
	0.1	8.498	4.776	344.9	18.0	597
	0.3	8.503	4.780	345.3	18.3	598
Gd	0.0	8.520	4.773	346.5	11.7	660
	0.1	8.508	4.768	345.1	12.7	664
	0.3	8.520	4.785	347.3	13.2	665
Dy	0.0	8.492	4.765	343.6	8.90	600
	0.1	8.489	4.769	343.7	9.49	604
	0.3	8.494	4.771	344.2	10.06	606
Er	0.0	8.467	4.761	341.3	8.29	570
	0.1	8.465	4.762	341.2	8.96	575
	0.3	8.472	4.771	342.4	9.47	579

Figure 2 shows the magnetization curves for  $YFe_{10}V_2C_x$  with  $x = 0$  and 0.3 at 1.5 K. The addition of C results in a decrease in the iron sublattice anisotropy. The anisotropy field  $H_A$  was determined from the  $H/M$  versus  $M^2$  curves in the hard-magnetization direction [12].  $H_A$  for  $YFe_{10}V_2C_x$  decreases from 4.0 T ( $x = 0$ ) to 3.0 T ( $x = 0.3$ ). This result is in agreement with that in  $YFe_{11}TiC_{1-\delta}$  [5] and in  $YFe_{10}Si_2C_x$  [9].

Figure 3 is the temperature dependence of the magnetization and the AC susceptibility for  $DyFe_{10}V_2C_x$ . The spin reorientation temperature increases from 183 K for  $x = 0$  to 212 K for  $x = 0.3$ . The  $Dy^{+3}$  ion has a negative  $\alpha_J$ , so that the second-order anisotropy favours the  $c$  plane. The competition between  $Dy^{+3}$  and iron sublattice anisotropies results

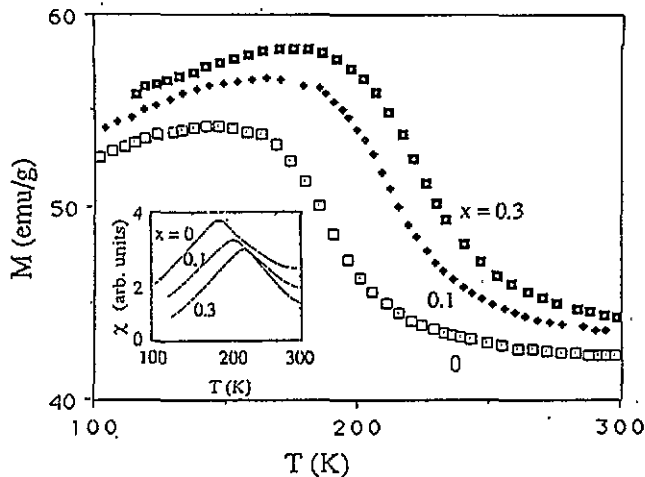


Figure 3. Temperature dependence of the magnetization for  $\text{DyFe}_{10}\text{V}_2\text{C}_x$  with  $x = 0, 0.1$  and  $0.3$ . The inset is the temperature dependence of the AC susceptibility of these samples.

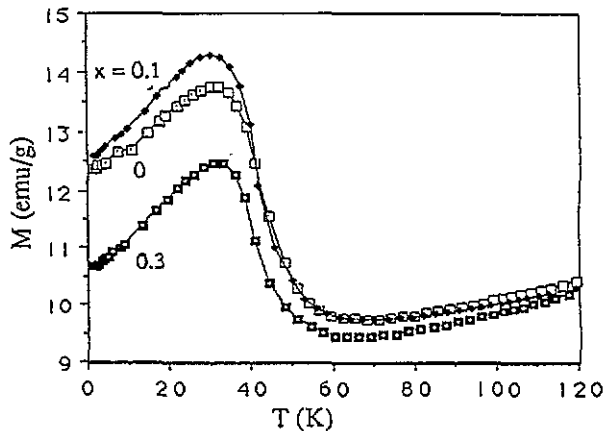


Figure 4. Temperature dependence of the magnetization perpendicular to the alignment direction for  $\text{ErFe}_{10}\text{V}_2\text{C}_x$  with  $x = 0, 0.1$  and  $0.3$ .

in spin reorientation [13, 14]. The addition of C decreases the anisotropy of iron sublattice, leading to an increase in the spin reorientation temperature.

Figure 4 shows the temperature dependence of the  $M_{\perp}$  for  $\text{ErFe}_{10}\text{V}_2\text{C}_x$ . The spin reorientation temperature is almost unchanged.  $\text{Er}^{3+}$  ions have a positive  $\alpha_f$ . The second-order anisotropy favours the  $c$  axis. The spin reorientation in  $\text{ErFe}_{10}\text{V}_2$  is actually driven by the sixth-order term, due to the large and positive value of  $\gamma_f$  of  $\text{Er}^{3+}$  [15].

Figure 5 shows the neutron diffraction pattern for  $\text{YFe}_{10}\text{V}_2\text{C}_{0.1}$ . To fit the experimental data, C atoms were allowed to occupy different sites 8f, 8i, 8j and 2b in the structure. The best fit is given in table 2. The V atoms exhibit a strong preference for occupying the 8i site, in agreement with the results of  $\text{YFe}_{10}\text{V}_2$  [16]. The C atoms occupy the interstitial site although the lattice expansion is small. The magnetic moments of Fe obtained by fitting the neutron diffraction pattern are also listed.

Our experiments show that interstitial C atoms can be introduced into the  $\text{ThMn}_{12}$ -type structure by arc melting, but the amount is small ( $x \leq 0.3$ ). Consequently, the effects of

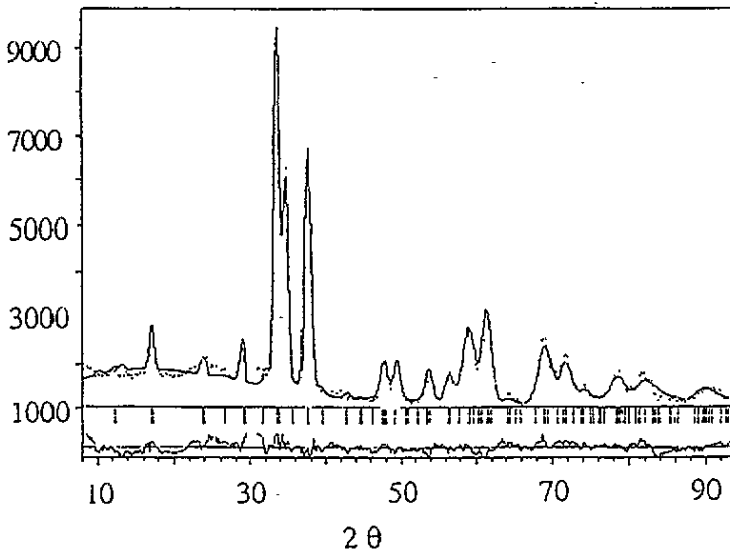


Figure 5. Neutron diffraction results for  $YFe_{10}V_2C_{0.1}$ . Data are indicated by the dots and the calculated pattern by the upper solid curve. The lower curve is the difference between the observed and calculated patterns. The location of nuclear and magnetic reflections are indicated by the upper and lower small vertical bars, respectively, below the diffraction pattern.

Table 2. Position parameters, occupations of atoms, and magnetic moments in the  $ThMn_{12}$  structure for  $YFe_{10}V_2C_{0.1}$ .

Atom	Site	$x$	$y$	$z$	$N$	$M$ ( $\mu_B$ )
Y	2a	0.0000	0.0000	0.0000	2.0000	
Fe	8i	0.3585	0.0000	0.0000	6.0651	1.73
Fe	8j	0.2709	0.5000	0.0000	8.0000	1.63
Fe	8f	0.2500	0.2500	0.2500	8.0000	1.52
V	8i	0.3585	0.0000	0.0000	1.9349	
C	2b	0.0000	0.0000	0.5000	0.2000	

$R = 8.33\%$ ;  $R_N = 8.28\%$ ;  $R_M = 10.6\%$ .

C addition on lattice expansion and Curie temperature are much smaller in the carbides prepared by arc melting than in those prepared by gas-solid reaction.

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