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Structural and magnetic behaviour of the series $Th_2Fe_{17}C_x$ ($x = 0$ to 1.25)

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Abstract

The crystal properties of the $Th_2Fe_{17}C_x$ series with $x = 0$, 0.3, 0.6, 0.9, 1.2, 1.25, have been determined from neutron powder diffraction. The effects of interstitial carbon on the structure and the magnetization have been studied both at 2 and 300 K. As in the structurally related rare earth intermetallics, carbon is found statistically to occupy a distorted octahedral site shared by two thorium and four iron atoms. The interatomic distances are compared with those previously reported and discussed with reference to some binary carbides.

1. Introduction

Recently, numerous studies have been devoted to R_2Fe_{17}, $R_2Fe_{17}H_x$, $R_2Fe_{17}C_x$ and $R_2Fe_{17}N_x$ compounds (R = rare earth metal) [1–5]. This interest is mostly due to the spectacular effect of the interstitial atoms on the physical properties, in particular on the hard magnet properties. Until now, only a few investigations of the corresponding compounds of Th–Fe have been reported, even though it has been shown [6] that the alloy Th_2Fe_{17} is very sensitive to the insertion of light elements. Th is a non-magnetic element and the various interstitial compounds of Th_2Fe_{17} can be taken as a reference for studying the effect of interstitial atoms on the iron sublattices only.

This paper deals with neutron powder diffraction refinements of both crystal and magnetic structures of the carbide series $Th_2Fe_{17}C_x$.

2. Experimental details

2.1. Synthesis and phase control

The $Th_2Fe_{17}C_x$ compounds were synthesized by the arc-melting technique from elements having at least 99.9% purity. The ingots were wrapped in

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tantalum foil and sealed into evacuated silica tubes. The samples were annealed at 1100 °C for about two weeks and then water quenched. All the samples were checked by X-ray diffractometry using the Cu Kα radiation on a backscattering graphite monochromator set up. The samples were found to be single phase, with the rhombohedral Th_2Zn_{17} structure type, up to a carbon concentration $x = 1.25$. In most of the $Th_2Fe_{17}C_x$ samples no free iron was detected by X-ray analysis [6]. The lattice parameters of all the samples are listed in Table 1.

2.2. *Neutron powder diffraction experiments*

Neutron diffraction experiments were carried out at the Siloe reactor of the Centre d'Etudes Nucléaires de Grenoble (C.E.N.G.). The instrument used, DN5, is equipped with a large ($2\theta = 80°$) one-dimension curved detector, allowing the recording of a pattern on 800 cells. At room temperature, the data were collected using the wavelength $\lambda = 2.478$ Å, whereas at 2 K $\lambda = 1.522$ Å was used for better assessment of magnetic data.

2.3. *Data analysis*

The data were analysed using the Rietveld technique [7] as implemented in the Fullprof program [8]. Both nuclear and magnetic contributions were simultaneously refined for single- as well as for multiphase patterns. The neutron scattering lengths used for the structure refinements are $b_{Th} = 9.84$, $b_{Fe} = 9.54$, $b_C = 6.65$ Fermi as taken from [9]. In order to minimize the number of refined parameters, only one overall Debye–Waller term was used, since the materials are primarily composed of iron atoms.

3. Discussion of the results

3.1. *Crystal structure analysis*

As previously analysed [6], the Th_2Fe_{17}–C phase diagram reveals a threshold carbon uptake, *i.e.* a limit of carbon solubility estimated to $x_l \approx 1.25$ per unit formula. The present work being restricted mostly to carbon compositions below this limit, it is noteworthy that all the carbides studied ($x = 0.3$, 0.6, 0.9, 1.2, 1.25) retain the host metal symmetry (Th_2Fe_{17}, $R\text{-}3m$ space group).

In contrast to the parent $R_2Fe_{17}C_x$ series, synthesis of the Th_2Fe_{17} carbides does not yield significant increases of free α-iron particles.

The refinements show that the carbon atoms are only accommodated at the octahedral 9e site, in agreement with the study performed on the Nd_2Fe_{17} carbide [10]. This also corroborates the extensive structure determination work performed on R_2Fe_{17}, the parent nitrides [4]. However, nitrogen has been found to almost completely fill the octahedral site (the metal–gas reaction proceeds from an equilibrium plateau type reaction [11]), while the carbon content appears as fully variable from $x = 0$–1.25. For some R_2Fe_{17} compounds x_l may take somewhat larger values (1.5), the complete filling

TABLE 1

Lattice parameters, agreement factors of the refinements and carbon content refined for the nominal formula $Th_2Fe_{17}C_x$ ($x=0$ to 1.5) at 300 K and 2 K. The agreement factors are the conventional Rietveld factors, R_{exp}, R_{prof}, R_{Bragg} and R_{mag} reference to the expected, profile, Bragg and magnetic factors respectively; λ is the neutron wavelength

Compound	λ (Å)	T (K)	C per formula unit	a (Å)	c (Å)	V (Å^3)	R_{exp} (%)	R_{prof} (%)	R_{Bragg} (%)	R_{mag} (%)
Th_2Fe_{17}	2.478	300	0	8.578(1)	12.476(1)	795	1.53	7.91	5.38	–
$Th_2Fe_{17}C_{0.3}$	2.478	300	0.28(4)	8.606(1)	12.492(1)	801	1.95	10.5	7.82	7.9
$Th_2Fe_{17}C_{0.6}$	2.478	300	0.62(1)	8.643(1)	12.504(1)	808	2.10	10.7	6.35	6.86
$Th_2Fe_{17}C_{0.9}$	2.478	300	0.91(4)	8.666(1)	12.508(1)	814	1.23	12.6	11.0	14.9
$Th_2Fe_{17}C_{1.2}$	2.478	300	1.10(5)	8.697(1)	12.518(1)	820	1.94	8.63	3.36	5.19
$Th_2Fe_{17}C_{1.5}$	2.478	300	1.25(4)	8.741(1)	12.558(1)	831	3.50	10.6	5.84	9.87
Th_2Fe_{17}	1.522	2	0	8.592(1)	12.508(1)	799	2.04	7.77	2.72	3.01
$Th_2Fe_{17}C_{0.3}$	1.522	2	0.28(4)	8.597(1)	12.489(2)	799	2.18	8.93	3.47	3.94
$Th_2Fe_{17}C_{0.6}$	1.522	2	0.62(1)	8.643(1)	12.516(2)	808	1.92	8.75	3.47	5.13
$Th_2Fe_{17}C_{0.9}$	1.522	2	0.84(4)	8.650(1)	12.494(2)	810	3.00	11.0	3.57	7.13
$Th_2Fe_{17}C_{1.2}$	1.522	2	1.10(5)	8.699(1)	12.533(2)	821	3.96	9.95	3.73	4.08
$Th_2Fe_{17}C_{1.5}$	1.522	2	1.25(4)	8.690(2)	12.510(3)	818	3.00	10.4	2.90	4.36

corresponding to $x=3$. In fact, for the off-limit compound $Th_2Fe_{17}C_{1.5}$ the data do not show a single-phase pattern and both $Th_2Fe_{17}C_{1.25}$ and a $BaCd_{11}$ type of compound were observed. We conclude that the experimental limit of carbon solubility corresponds to $x_1=1.25$. Increase of the carbon content favours the appearance of a $BaCd_{11}$ type of structure [6], whose characteristics are summarized below. More details on this new ternary carbide will appear in a forthcoming paper [12].

In the R_2Fe_{17} carbides, it can be noticed that the carbon atoms are located in the atomically dense basal phase of the hexagonal representation of the lattice. This leads to a significantly larger expansion rate for the in-plane (a, b) cell parameters than for the c parameter. Figures 1(a), 1(b) and 1(c) show the concentration dependence of a, c and the unit cell volume V as measured at 300 K. The cell parameters are reported in Table 1. It can be noticed that both lattice parameters expand linearly with the concentration of inserted carbon up to about 1.1 C per formula unit. For the upper carbon contents, a slight deviation from linearity is observed, the increase of the a and c parameters being more pronounced. This effect probably corresponds to the limit of stability of the $Th_2Fe_{17}C_x$ phase.

The crystal structure parameters deduced from the refinements are reported in Tables 2 and 3. The neighbourhood of the carbon atom is formed of two thorium and two iron atoms, all residing in the basal plane and two iron atoms situated apart from this plane (Fig. 2). The Th–C distance increases from $d_{Th-C}=2.49$ Å for $x=0.3$ to 2.53 Å for $x\approx 1.25$. These values remain smaller by 0.2–0.15 Å than that observed in cubic ThC (NaCl type), the carbon having the same sixfold metal coordination with $d_{Th-C}=2.67$ Å.

Two different Fe–C distances are observed, corresponding to the different iron sites Fe_3(18f) and Fe_4(18h). They are also found to increase with x:

$d_{Fe_3-C}=1.88$ Å for $x=0.3$ to 1.91 Å for $x_1\approx 1.25$

$d_{Fe_4-C}=1.97$ Å for $x=0.3$ to 2.00 Å for $x_1\approx 1.25$

These distances satisfactorily approach those obtained by addition of the atom radii used elsewhere [13].

As far as cementite, Fe_3C, is concerned, carbon is here again sixfold coordinated by metal atoms, but forming trigonal prisms. Two Fe–C distances are also observed: d_{Fe_1-C} in $Fe_3C=1.988$ Å and d_{Fe_2-C} in $Fe_3C=2.035$ Å [14, 15]. These are close to those found in $Th_2Fe_{17}C\approx 1.25$, and more precisely account for the expansion rate measured between $x=0.3$–1.25. An extrapolation to $x=3$ leads to 1.979 and 2.053 Å for Fe_3–C and Fe_4–C respectively, in full agreement with the values observed in Fe_3C. Based on interatomic distances, it can be concluded that carbon is more strongly bonded with iron than with thorium (R).

The short dumbbell distance measured at 2 and 300 K seems first to decrease from $x=0.3$–0.6, then it increases up to $x=1.25$. It is rather difficult to compare this distance with the values obtained on the rare earth based nitrides $R_2Fe_{17}N_x$ where a net increase of such a distance is

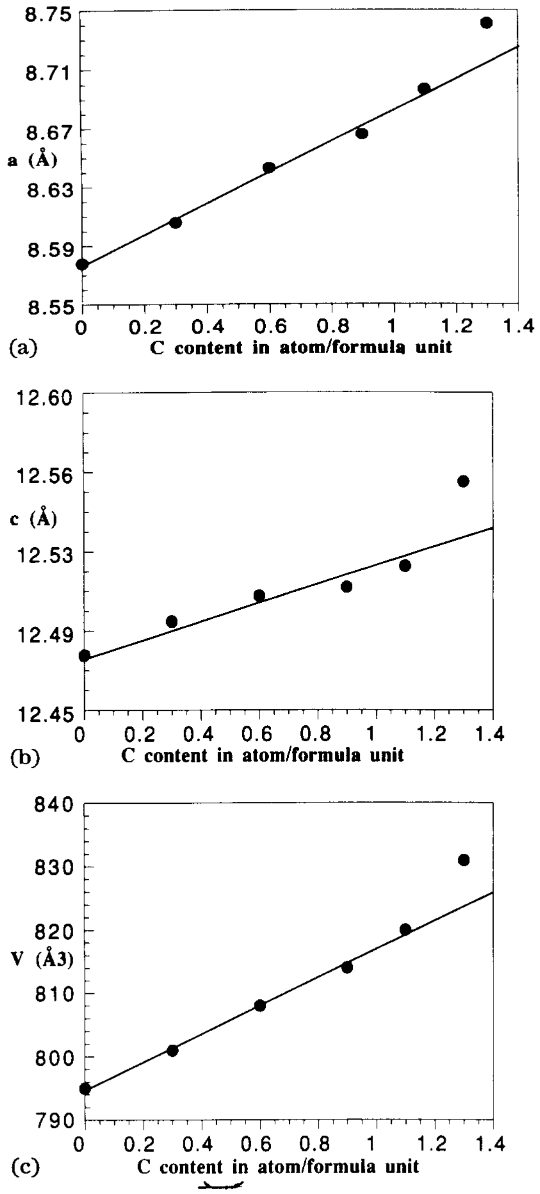

Fig. 1. Concentration dependence of the cell parameters (a, c and volume) of $Th_2Fe_{17}C_x$ with the amount of carbon uptake at room temperature.

observed but occurs for the largest values of x only (2.5–2.9 N per formula unit).

We therefore come to the conclusion that in the $Th_2Fe_{17}C_x$ series and for the composition range studied, no direct correlation can be established between the strongly increasing Curie temperature and the dumbbell Fe–Fe distance.

TABLE 2

2 K refined structural parameters for $Th_2Fe_{17}C_x$ with $x=0$–1.25 and the magnetic moments (in μ_B) measured on the iron sites; n are the relative site occupancies

Atom	Site	x	y	z	n	m (μ_B)
Th_2Fe_{17} at 2K						
Th	6c	0	0	0.3396(6)	0.1667	0
Fe(1)	6c	0	0	0.0939(3)	0.1667	2.81(12)
Fe(2)	9d	0.5	0	0.5	0.25	2.14(12)
Fe(3)	18f	0.2875(3)	0	0	0.5	1.83(9)
Fe(4)	18h	0.5018(2)	0.4982(2)	0.1583(3)	0.5	1.74(9)
$Th_2Fe_{17}C_{0.3}$ at 2 K						
Th	6c	0	0	0.3396(6)	0.1667	0
Fe(1)	6c	0	0	0.0929(4)	0.1667	2.46(16)
Fe(2)	9d	0.5	0	0.5	0.25	2.11(16)
Fe(3)	18f	0.2856(3)	0	0	0.5	1.79(10)
Fe(4)	18h	0.5015(2)	0.4985(2)	0.1567(3)	0.5	1.61(13)
C	9e	0.5000	0	0	0.023(3)	0
$Th_2Fe_{17}C_{0.6}$ at 2 K						
Th	6c	0	0	0.3430(6)	0.1667	0
Fe(1)	6c	0	0	0.0953(4)	0.1667	3.17(14)
Fe(2)	9d	0.5	0	0.5	0.25	2.11(13)
Fe(3)	18f	0.2844(3)	0	0	0.5	1.81(11)
Fe(4)	18h	0.5018(2)	0.4982(2)	0.1562(3)	0.5	1.76(11)
C	9e	0.5000	0	0	0.052(1)	0
$Th_2Fe_{17}C_{0.9}$ at 2 K						
Th	6c	0	0	0.3406(8)	0.1667	0
Fe(1)	6c	0	0	0.0965(5)	0.1667	2.86(18)
Fe(2)	9d	0.5	0	0.5	0.25	2.23(16)
Fe(3)	18f	0.2843(4)	0	0	0.5	1.83(12)
Fe(4)	18h	0.5025(3)	0.4975(3)	0.1567(4)	0.5	1.79(12)
C	9e	0.5000	0	0	0.070(3)	–
$Th_2Fe_{17}C_{1.2}$ at 2 K						
Th	6c	0	0	0.3409(7)	0.1667	0
Fe(1)	6c	0	0	0.0960(5)	0.1667	2.91(15)
Fe(2)	9d	0.5	0	0.5	0.25	2.43(15)
Fe(3)	18f	0.2830(4)	0	0	0.5	1.97(10)
Fe(4)	18h	0.5027(2)	0.4973(2)	0.1557(4)	0.5	1.81(10)
C	9e	0.5000	0	0	0.090(4)	–
“*$Th_2Fe_{17}C_{1.5}$*” *at 2 K* (the refinement leads to the formula $Th_2Fe_{17}C_{1.25}$ and a $BaCd_{11}$ type impurity)						
Th	6c	0	0	0.3425(10)	0.1667	0
Fe(1)	6c	0	0	0.0953(6)	0.1667	3.00(22)
Fe(2)	9d	0.5	0	0.5	0.25	2.37(21)
Fe(3)	18f	0.2825(5)	0	0	0.5	1.83(15)
Fe(4)	18h	0.5028(4)	0.4972(4)	0.1555(5)	0.5	1.94(16)
C	9e	0.5000	0	0	0.103(3)	–

TABLE 3

300 K refined structural parameters for $Th_2Fe_{17}C_x$ with $x = 0–1.25$ and magnetic moments (in μ_B) measured on the iron sites; n are the relative occupancies. (The Debye–Waller factors are given in Å^2.)

Atom	Site	x	y	z	B	n	m (μ_B)
Th_2Fe_{17} at 300 K							
Th	6c	0	0	0.3430(6)	0.4	0.1667	0
Fe(1)	6c	0	0	0.0943(4)	0.7	0.1667	–
Fe(2)	9d	0.5	0	0.5	0.7	0.25	–
Fe(3)	18f	0.2874(3)	0	0	0.5	0.5	–
Fe(4)	18h	0.5021(2)	0.4979(2)	0.1574(3)	0.7	0.5	–
$Th_2Fe_{17}C_{0.3}$ at 300 K							
Th	6c	0	0	0.3424(8)	0.5(3)	0.1667	0
Fe(1)	6c	0	0	0.0936(5)	0.6	0.1667	0.70(15)
Fe(2)	9d	0.5	0	0.5	0.6	0.25	0.70(15)
Fe(3)	18f	0.2820(4)	0	0	0.4(2)	0.5	0.70(15)
Fe(4)	18h	0.5033(3)	0.4967(3)	0.1576(4)	0.4(2)	0.5	0.70(15)
C	9e	0.5	0	0	0.6	0.023(3)	–
$Th_2Fe_{17}C_{0.6}$ at 300 K							
Th	6c	0	0	0.3452(7)	0.6	0.1667	0
Fe(1)	6c	0	0	0.0936(5)	0.6	0.1667	1.20(09)
Fe(2)	9d	0.5	0	0.5	0.6	0.25	1.20(09)
Fe(3)	18f	0.2843(4)	0	0	0.6	0.5	1.20(09)
Fe(4)	18h	0.5039(3)	0.4960(3)	0.1583(4)	0.6	0.5	1.20(09)
C	9e	0.5	0	0	0.6	0.052(1)	–
$Th_2Fe_{17}C_{0.9}$ at 300 K							
Th	6c	0	0	0.3412(8)	0.6	0.1667	0
Fe(1)	6c	0	0	0.0939(6)	0.6	0.1667	0.97(10)
Fe(2)	9d	0.5	0	0.5	0.6	0.25	0.97(10)
Fe(3)	18f	0.2792(4)	0	0	0.6	0.5	0.97(10)
Fe(4)	18h	0.5041(3)	0.4959(3)	0.1546(4)	0.6	0.5	0.97(10)
C	9e	0.5	0	0	0.6	0.076(3)	–
$Th_2Fe_{17}C_{1.2}$ at 300 K							
Th	6c	0	0	0.3481(6)	0.6	0.1667	0
Fe(1)	6c	0	0	0.0943(4)	0.6	0.1667	1.46(6)
Fe(2)	9d	0.5	0	0.5	0.6	0.25	1.46(6)
Fe(3)	18f	0.2817(3)	0	0	0.6	0.5	1.46(6)
Fe(4)	18h	0.5045(2)	0.4955(2)	0.1619(3)	0.6	0.5	1.46(6)
C	9e	0.5	0	0	0.6	0.090(1)	–
$Th_2Fe_{17}C_{1.5}$ at 300 K							
Th	6c	0	0	0.3457(8)	0.6	0.1667	0
Fe(1)	6c	0	0	0.0927(6)	0.6	0.1667	1.37(11)
Fe(2)	9d	0.5	0	0.5	0.6	0.25	1.37(11)
Fe(3)	18f	0.2810(5)	0	0	0.6	0.5	1.37(11)
Fe(4)	18h	0.5045(3)	0.4955(3)	0.1594(5)	0.6	0.5	1.37(11)
C	9e	0.5	0	0	0.6	0.097(3)	–

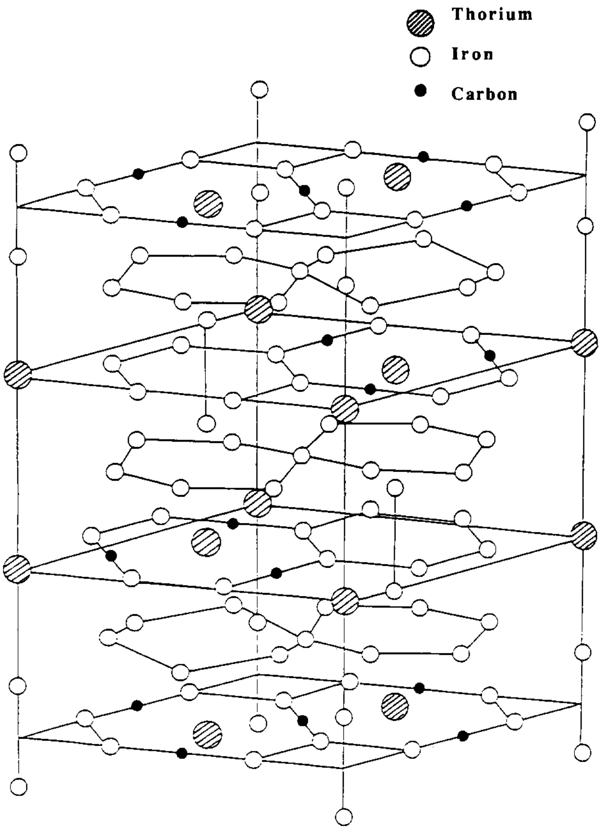

Fig. 2. Schematic representation of the $Th_2Fe_{17}C_x$ rhombohedral structure. The medium and small circles refer to thorium and iron respectively. The black circles refer to the carbon atoms.

3.2. Magnetic properties

As reported in a recent paper [6], the insertion of carbon leads to an increase of both the Curie temperature and the magnetization of the Th_2Fe_{17} compounds. This behaviour has been explained in terms of magneto-volume effects. In order to refine the magnetic moments of the iron sites, a collinear magnetic structure (with M perpendicular to c) was assumed, owing to the iron sublattice anisotropy that favours easy basal plane behaviour. No magnetic moment was allowed on the thorium atom since it is assumed to exhibit a tetravalent character in such intermetallic compounds, despite the fact that a possible value of 0.2 μ_B can exist, but is not measurable by powder neutron diffraction. The iron magnetic moments refined at 300 K and 2 K for all the carbon concentrations are reported in Tables 2 and 3 respectively. Neutron powder diffraction patterns are shown in Figs. 3 and 4.

The iron sites directly bonded with carbon (Fe_3(18f), Fe_4(18h)) have significantly lower magnetic moments than observed on the two other iron sites Fe_1(6c) and Fe_2(9d). The former magnetic moments are quite comparable

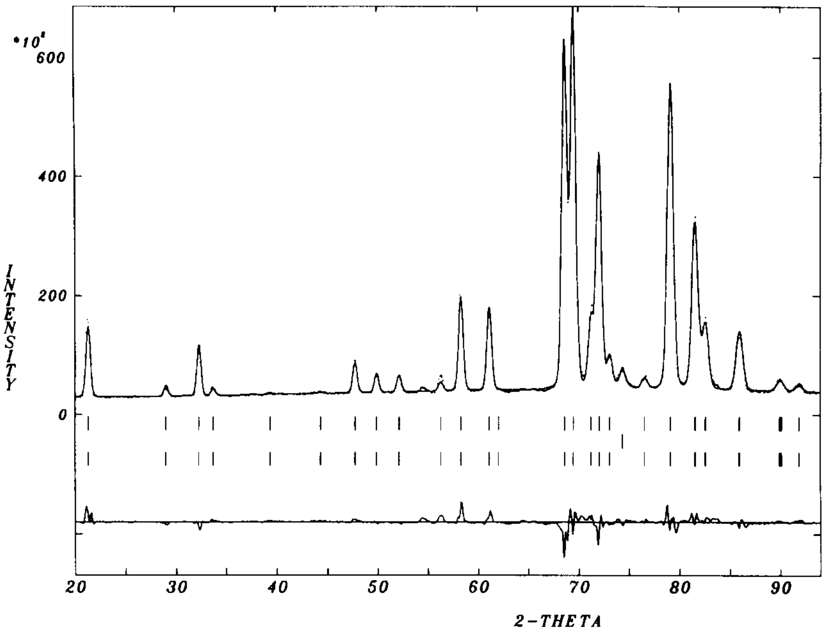

Fig. 3. Neutron powder diffraction pattern of $Th_2Fe_{17}C_{1.2}$ at room temperature. The points and the line refer to the recorded pattern and the calculated fit respectively. The difference pattern is plotted in the lower part of the figure with the same scale. The first and third set of bars refer to the nuclear and magnetic diffraction lines of $Th_2Fe_{17}C_{1.2}$ respectively. The second set refers to α-iron present as impurity.

to $M = 1.8$ μ_B/Fe encountered in Fe_3C [14]. This confirms the similarities in the interatomic distances and it corresponds to a d–p bonding overlap [13].

As observed in hydrides and nitrides of R_2Fe_{17} compounds [16] the dumbbell iron site 6c exhibits by far the largest magnetic moment. The latter site Fe_2(9d) has a magnetic moment close to that of α-iron, and has been the most enhanced by carbon insertion.

In this carbide series, magnetic measurements have revealed that the saturation magnetization M_s as well as the Curie temperature increase with the carbon concentration, and a linear increase with the cell volume was observed [6]. The mean iron moment value deduced from the neutron diffraction experiments performed at 2 K agrees well with the bulk measurements (Table 4). In fact the local values we determined are always larger by about 0.15 μ_B than the macroscopic ones. This slight deviation falls within the standard error, but such a systematic shift could originate from negative conduction electron polarization.

The net magnetization increase from $x = 0$ to $x = 1.2$ is 4 μ_B per unit formula, or $\Delta M/M_{x=0} = 17\%$. In the Th_2Fe_{17} nitride an increase of 24% has been observed [6]. This increase is by far much larger than what was previously found at low temperatures in the $R_2Fe_{17}X_x$ (X = H and N) where the increase does not exceed 5% [17].

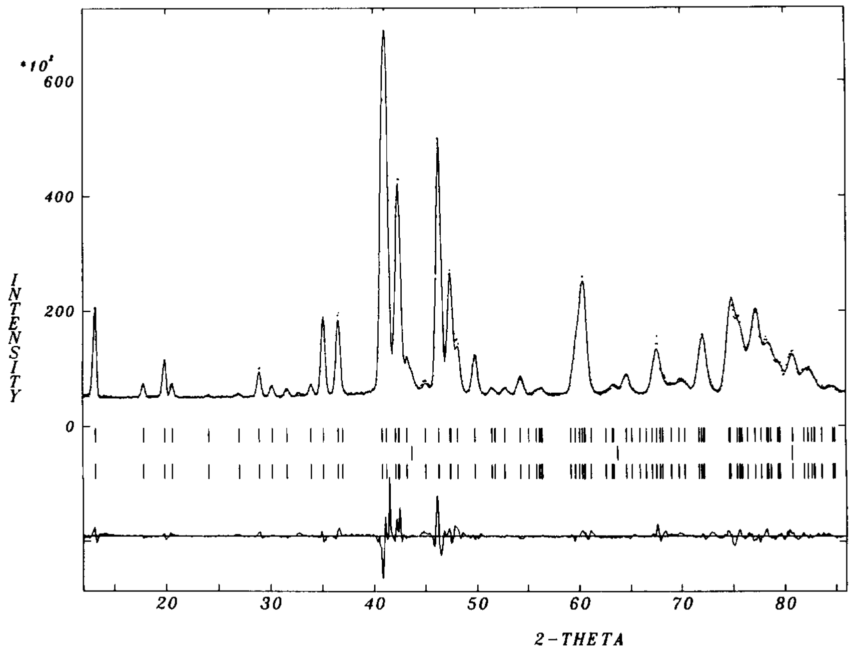

Fig. 4. Neutron powder diffraction pattern of $Th_2Fe_{17}C_{0.6}$ at 2 K. The points and the line refer to the recorded pattern and the calculated fit respectively. The difference pattern is plotted in the lower part of the figure with the same scale. The first and third set of bars refer to the nuclear and magnetic diffraction lines of $Th_2Fe_{17}C_{0.6}$ respectively. The second set refers to α-iron present as an impurity.

TABLE 4

Comparison of the average magnetic moment obtained by neutron diffraction experiment with that of bulk magnetization measurements

Compounds	m_{Fe} (μ_B) 5 K H = 5.5 T (from ref. 6)	m_{Fe} (μ_B) 2 K Rietveld refinement
Th_2Fe_{17}	1.79	1.83(10)
$Th_2Fe_{17}C_{0.3}$	1.79	1.86(10)
$Th_2Fe_{17}C_{0.6}$	1.87	2.01(10)
$Th_2Fe_{17}C_{0.9}$	1.91	2.01(10)
$Th_2Fe_{17}C_{1.2}$	2.03	2.10(10)

3.3. The limit of stability of $Th_2Fe_{17}C_x$: the $BaCd_{11}$-type carbide

The sample of nominal composition $Th_2Fe_{17}C_{1.5}$ has been found to consist of $Th_2Fe_{17}C_{1.25}$ and $ThFe_{11}C_{1.3}$, in agreement with a previous X-ray analysis [6]. The structure of the new ternary carbide has been refined as being isotypic with $LaMn_{11}C_{2-x}$ [18]. Here again carbon occupies a slightly distorted octahedron formed by four iron and two thorium atoms. The structural and magnetic parameters will be reported in a forthcoming paper [12].

4. Conclusion

This work performed on a series of $Th_2Fe_{17}C_x$ carbides ($x=0$ to 1.25) showed that the carbon atoms occupy the 9e octahedral site exclusively. This induces an anisotropic cell parameter expansion, mainly affecting the basal plane. The limit of stability of the ternary carbide corresponds to the formula $Th_2Fe_{17}C_{1.25}$, carbon-rich samples exhibiting the presence of a $BdCd_{11}$ type of interstitial compound [6]. The Fe–C bonding distances measured in $Th_2Fe_{17}C_x$ closely correspond to those observed in Fe_3C [14, 15], the Th–C bond being weaker than in the binary Th–C. The magnetic moments measured at 2 K on the four iron sites are different in magnitude. When bonded with carbon, the magnetic moments of iron are significantly weaker. The mean magnetic moment agrees well with that found from magnetization measurements. It depends sensitively on the amount of carbon accommodated in the cell.

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