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Interstitial carbonation of the Sm_2Fe_{17} phase by reaction with hydrocarbons

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Abstract

Interstitial Sm_2Fe_{17} carbides were synthesized by reacting Sm_2Fe_{17} powder with various hydrocarbons at about 550–600 °C. The resulting Sm_2Fe_{17} carbide retains its rhombohedral structure with an expanded lattice with parameters $a = 8.744$ Å and $c = 12.572$ Å. The Curie temperature of the carbide is 400 °C. The carbide is strongly uniaxial, exhibiting an anisotropy field of about 120 kOe. The reaction kinetics between Sm_2Fe_{17} and hydrocarbon gases were studied in a constant-volume reactor (Isochoro-Thermal Analyser) by monitoring the pressure and temperature. X-ray diffraction and thermomagnetic analysis have been employed for phase analysis of the carbonation products. The estimated content in the resulting carbides ($Sm_2Fe_{17}C_{2.6}$) is much higher than that obtainable by conventional melting methods ($Sm_2Fe_{17}C_1$).

1. Introduction

The R_2Fe_{17} (R, rare earth) compounds can be interstitially modified to form hydrides, nitrides or carbides ($R_2Fe_{17}(H,N,C)_x$). Interstitial hydrides [1] and nitrides [2] can be prepared by direct reaction of the parent compound with hydrogen or nitrogen gas. Interstitial carbides [3] can be prepared by direct melting of the constituent elements followed by high temperature heat treatment (1000–1200 °C). The R_2Fe_{17} compounds with rare earths lighter than gadolinium exhibit the rhombohedral Th_2Zn_{17}-type structure, while those with rare earths heavier than dysprosium exhibit the hexagonal Th_2Ni_{17}-type structure. Both crystal structures may coexist for gadolinium, terbium and dysprosium as well as for yttrium. Lanthanum does not form such compounds. The most interesting ones in terms of potential permanent magnet applications are the interstitial Sm_2Fe_{17} nitride and carbide. Both exhibit large anisotropy fields and high Curie temperatures [4, 5]. Recently, the gas phase carbonation method was used by Coey *et al.* [6], Bo Ping and Gui-Ghuan Liu [7], Liao *et al.* [8], Hong Sun *et al.* [9] and Chen *et al.* [10] for the preparation of R_2Fe_{17} carbides exhibiting superior intrinsic magnetic properties to those prepared by conventional melting. The present study deals exclusively with the Sm_2Fe_{17} carbides prepared by gas phase carbonation with the use of several hydrocarbons, the corresponding reaction kinetics and the intrinsic magnetic properties.

2. Experimental details

The Sm_2Fe_{17} alloy was prepared by arc melting the constituent elements (purity better than 99.9 wt.%) in a pure argon gas atmosphere. During melting, a 5 wt.% excess of samarium was added to account for the samarium loss due to vaporization. The as-prepared Sm_2Fe_{17} alloy was heat treated at 1000 °C for about 1 week in order to produce an Sm_2Fe_{17} single-phase alloy. A powder of particle size less than 45 μm was prepared from this alloy and was used for all the carbonation experiments. The purities of the hydrocarbon gases (methane, *n*-butane and acetylene) and liquids (*n*-hexane, *m*-xylene, toluene and benzene) were greater than 99.9 vol.% and 99.98. wt.% respectively.

The constant-volume reactor isochoro-thermal analyser (ITA) which was used in the present study is similar to the thermopiezic analyser (TPA) used by other researchers (*e.g.* Coey *et al.* [6] and Liao *et al.* [8]) for the study of gas–solid reactions. The constant volume of the ITA was about 2.3 cm^3 and only about 5% of the total volume was heated. Heat was supplied by a precisely controlled programmable furnace capable of reaching a temperature of 1000 °C. The pressure was measured with a pressure sensor capable of detecting pressure differences of about 100 Pa. The temperature was measured to an accuracy of ± 1 °C. The pressure *vs.* temperature data were collected in a computer and analysed taking into consideration the pressure variations due to the thermal effects (heated

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volume). By utilizing the ideal gas law, the data were transformed and plotted in terms of gas mole ratios (present:initial) as a function of temperature. The ideal gas law was applied in this case and, considering the relatively low pressure and high temperature conditions (25–102 kPa and 20–1000 °C respectively), was considered to be a good approximation. The reaction taking place in the ITA apparatus is a "batch reaction" and will be referred to herein as "closed system".

Reactions were also performed by flowing hydrocarbon gas over Sm_2Fe_{17} powder at selected temperatures between 250 and 800 °C. These are "continuous gas flow reactions" and will be referred to herein as "open system". In the case of the liquid hydrocarbons, argon gas was used as a carrying agent (bubbling through the liquid at about 50 °C).

Magnetization measurements (M *vs.* H) were obtained with a vibrating sample magnetometer (VSM) in magnetic fields up to 15 kOe. Thermomagnetic analysis (TMA) was performed with a Faraday balance between the temperatures of 25 and 850 °C and in a magnetic field of about 1 kOe. The anisotropy field was estimated by applying the extrapolation method to magnetization *vs.* magnetic field (up to 15 kOe) data obtained parallel and perpendicular to the crystallographic *c* axis of aligned powders which were premagnetized in a pulse field of 75 kOe. X-ray powder diffraction patterns were obtained using a Philips automated diffractometer with Cu Kα radiation monochromated by a graphite single crystal. X-ray diffraction patterns were obtained for both random powders and magnetically aligned powders fixed in epoxy resin.

3. Results and discussion

3.1. The $Sm_2Fe_{17}+CH_4$ system

The $Sm_2Fe_{17}+CH_4$ system has been studied extensively in the present study in terms of the reaction kinetics and products obtained as a function of temperature. ITA isochore traces (closed system) for this system are shown in Figs. 1(a)–1(e). They include the isochore for pure CH_4 (Fig. 1(a)) as well as those for $Sm_2Fe_{17}+CH_4$ (Figs. 1(b)–1(e)) with different CH_4:Sm_2Fe_{17} mole ratios and under different initial pressures of CH_4. X-ray diffraction patterns of the reaction products between Sm_2Fe_{17} powder and CH_4 (open system) at 250, 400, 500, 550, 600 and 800 °C are shown in Figs. 2(b)–2(g) respectively. The reaction products in both open and closed systems are basically the same. The difference is that in the closed system the effect of hydrogen is more profound.

In the absence of Sm_2Fe_{17} powder, pure CH_4 (Fig. 1(a)) seems to decompose (intrinsically) at 850 °C according to the reaction

$$CH_4(g) \longleftrightarrow C(s) + 2H_2(g)$$

Carbon was observed visually to be deposited on the inner surface of the quartz tube where the reaction took place. It is very possible that the quartz surface might serve as a catalyst for the decomposition reaction. Under these conditions the extent of the reaction is very small (less than 3% at 950 °C).

The ITA isochore traces in Figs. 1(b)–1(e) suggest that in the presence of Sm_2Fe_{17} the decomposition of CH_4 is initiated below 350 °C. At this temperature most of the H_2 produced is absorbed by the Sm_2Fe_{17} to form Sm_2Fe_{17} hydrides. This is the reason why the total number of gaseous species decreases instead of increasing (since 1 mol CH_4 produces 2 mol H_2). Even under these relatively low pressure conditions a portion of the Sm_2Fe_{17} hydrides decomposes into α-Fe + SmH_2. The production of α-Fe + SmH_2 increases as the reaction temperature increases. The X-ray diffraction pattern in Fig. 2(b) shows that interstitial hydrogenation together with the production of α-Fe + SmH_2 take place at a temperature as low as 250 °C. At 400 °C (Fig. 2(c)) the Sm_2Fe_{17} phase coexists with α-Fe + SmH_2 (open system), which suggests that most of the hydrogen has desorbed from the Sm_2Fe_{17} phase, with some decomposition occurring too. In the case of the ITA experiments (closed system) the $Sm_2Fe_{17}H_x$ continues to exist with lower hydrogen content. The relative amount of decomposition products (α-Fe + SmH_2) with respect to the Sm_2Fe_{17} phase increases up to the temperature of 500 °C (Fig. 2(d)) and no interstitial Sm_2Fe_{17} carbides are produced. However, carbon was seen visually to be deposited on the powder.

Interstitial Sm_2Fe_{17} carbides appeared at the reaction temperatures of 550 and 600 °C (Figs. 2(e) and 2(f)). This was confirmed by the lattice expansion ($a = 8.738$ Å, $c = 12.542$ Å as compared with $a = 8.549$ Å, $c = 12.441$ Å for the parent compound) and the increase in Curie temperature (400 °C as compared with 135 °C for the parent compound). It is obvious that diffusion of carbon into the Sm_2Fe_{17} structure becomes appreciable at temperatures above 500 °C. Below this temperature the carbon produced is being deposited on the particle surface, where it accumulates without diffusion. In the ITA experiments (closed system) an Sm_2Fe_{17} carbohydride was found instead of an Sm_2Fe_{17} carbide. This was confirmed by X-ray diffraction patterns of the as-prepared samples and of samples subjected to a vacuum at 250 °C for 1 h. The Sm_2Fe_{17} carbohydrides show a lattice expansion in the *c* direction with respect to the Sm_2Fe_{17} carbides. Details of this study will be published elsewhere.

At temperatures above 600 °C a complete decomposition of the Sm_2Fe_{17} carbides (hydrides) occurs in the presence of hydrogen, the major products being

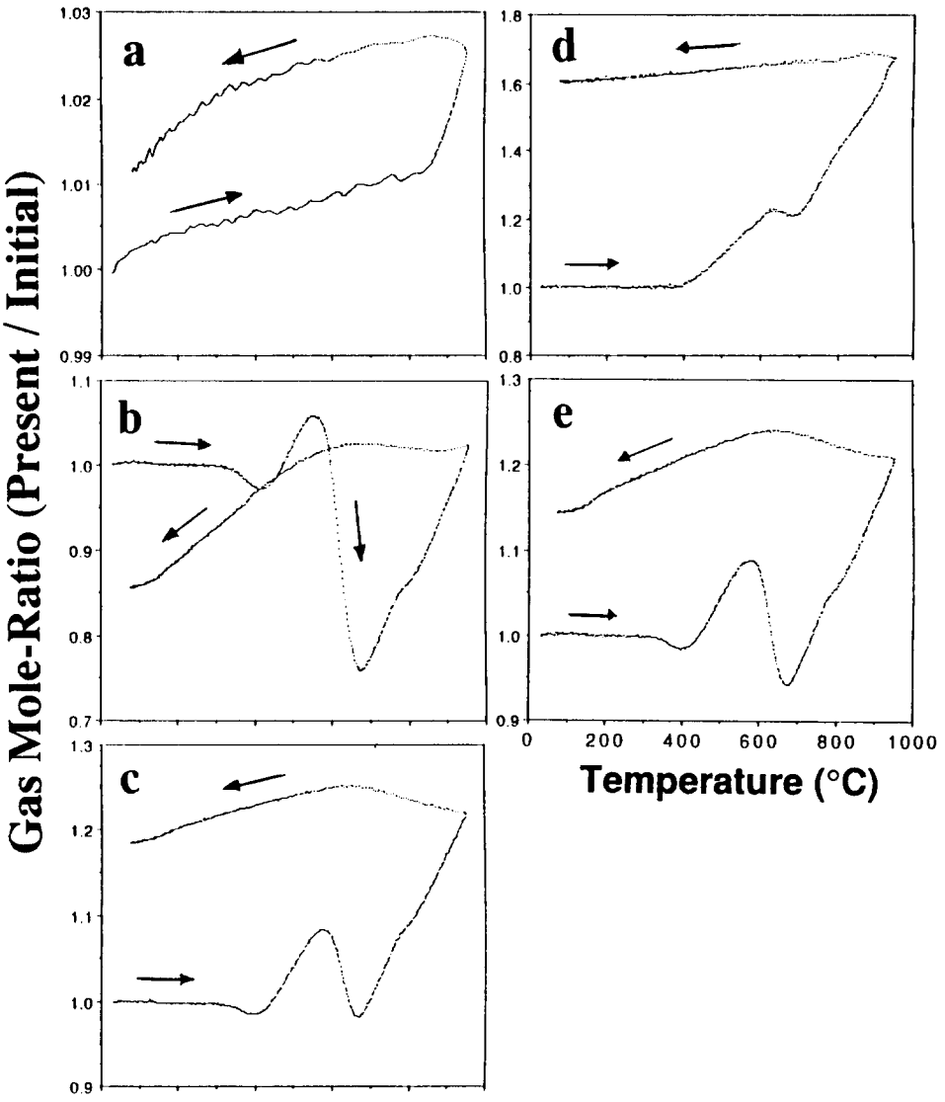

Fig. 1. ITA isochores obtained for the $Sm_2Fe_{17}+CH_4$ system with different Sm_2Fe_{17}:CH_4 mole ratios and under different initial gas pressures (P_0): a, pure CH_4, $P_0=102.0$ kPa; b, Sm_2Fe_{17}:$CH_4=1.961$, $P_0=101.4$ kPa; c, Sm_2Fe_{17}:$CH_4=4.053$, $P_0=101.9$ kPa; d, Sm_2Fe_{17}:$CH_4=3.052$, $P_0=25.4$ kPa; e, Sm_2Fe_{17}:$CH_4=3.093$, $P_0=87.0$ kPa.

Fe_3C, α-Fe and samarium hydrides (Fig. 2(g)). Other products such as samarium carbohydrides may also be present in this range. The Sm_2O_3 (b.c.c. and monoclinic) observed in the X-ray diffraction pattern shown in Fig. 2(g) is attributed to the oxidation of samarium carbohydrides during exposure to atmospheric conditions. The major absorption shown in the isochores at around 600 °C is due to the absorption of hydrogen by the samarium involved in the decomposing Sm_2Fe_{17} carbohydride.

At temperatures above 700 °C more and more hydrogen is being produced (Figs. 1(b)–1(e)) as a result of further decomposition of CH_4 and dehydrogenation of samarium hydrides.

The effect of the CH_4:Sm_2Fe_{17} mole ratio (under the same initial gas pressure) during heating is shown in the isochores of Figs. 1(b) and 1(c). For the lower ratio (Fig. 1(b)) the availability of the Sm_2Fe_{17} makes the hydrogen absorption greater at around 400 °C (by Sm_2Fe_{17}) and 600 °C (decomposition). Also, the total number of gaseous species at the end of the reaction is less than the initial value in the case of the smaller CH_4:Sm_2Fe_{17} mole ratio (Fig. 1(b)). This occurs because the majority of the hydrogen produced is absorbed by samarium to form samarium hydrides.

The effect of the initial CH_4 pressure (for the same CH_4:Sm_2Fe_{17} mole ratio) is shown in the isochores of Figs. 1(d) and 1(e). When 1 mol CH_4 decomposes, 2 mol H_2 are produced. Therefore the extent of the reaction is thermodynamically favoured at lower pressures. The reaction under 25.4 kPa (Fig. 1(d)) produces more gaseous species than the reaction under 87.0 kPa

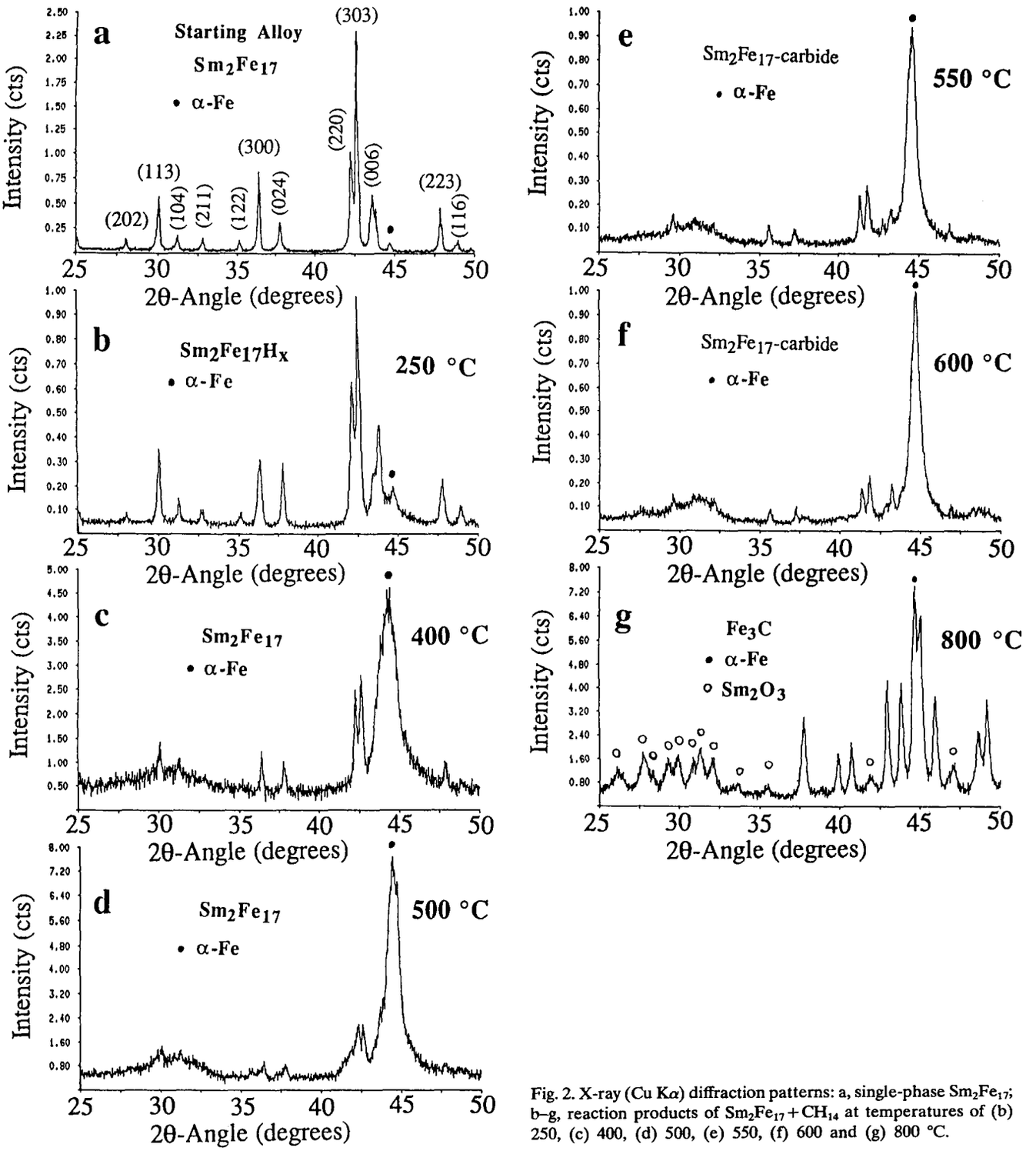

Fig. 2. X-ray (Cu Kα) diffraction patterns: a, single-phase Sm_2Fe_{17}; b–g, reaction products of $Sm_2Fe_{17}+CH_4$ at temperatures of (b) 250, (c) 400, (d) 500, (e) 550, (f) 600 and (g) 800 °C.

(Fig. 1(e)). In this case the rate of production of hydrogen is greater than the rate of absorption of hydrogen. This results in a diminishment in the hydrogen absorption dips on the isochore traces as shown in Fig. 1(d).

There is another common feature of the isochore traces other than the two dips due to the hydrogen absorption, namely the "kink" observed at around 800–850 °C. This might be due to the dissociation of samarium dihydride or/and the intrinsic decomposition of CH_4 (see Fig. 1(a)).

The ITA isochore traces obtained in the case of acetylene and especially *n*-butane exhibited similar features to those obtained in the case of methane.

3.1.1. Summary of reaction kinetics

On the basis of the information obtained from the ITA isochores, X-ray diffraction, *M vs. T* behaviours and visual observation, the reaction occurring in the $Sm_2Fe_{17}+CH_4$ open and closed systems can be summarized as follows.

(1) Two reactions take place constantly in the temperature range between 200 and 950 °C. (i) CH_4 decomposes on the surface of solid particles to produce $C(s)+2H_2(g)$. (ii) Solid carbon is physically deposited on the surface of the solid particles.

(2) Specific reactions occur depending on the temperature range.

(a) Between 200 and 400 °C

Open or closed system. (i) A portion of the hydrogen produced is absorbed interstitially by Sm_2Fe_{17} to form $Sm_2Fe_{17}H_x$. (ii) A portion of the $Sm_2Fe_{17}H_x$ decomposes into α-Fe + SmH_2. Overall reaction:

$$Sm_2Fe_{17}+CH_4 \longleftrightarrow Sm_2Fe_{17}H_x+\alpha\text{-Fe}+SmH_2+C+H_2$$

(b) Between 400 and 500 °C

Open system. (i) The majority of the $Sm_2Fe_{17}H_x$ desorbs hydrogen to form Sm_2Fe_{17}. Some of the $Sm_2Fe_{17}H_x$ decomposes to produce α-Fe + SmH_2. Overall reaction:

$$Sm_2Fe_{17}H_x+CH_4 \longleftrightarrow Sm_2Fe_{17}+\alpha\text{-Fe}+SmH_2+C+H_2$$

Closed system. (i) A fraction of the $Sm_2Fe_{17}H_x$ persists (with a smaller value of x), while there is also some decomposition to produce α-Fe + SmH_2. Overall reaction:

$$Sm_2Fe_{17}H_x+CH_4 \longleftrightarrow Sm_2Fe_{17}H_y+\alpha\text{-Fe}+SmH_2+C+H_2$$

(c) Between 500 and 600 °C

Open system. (i) Some of the hydrogen reacts directly with Sm_2Fe_{17} to produce α-Fe + SmH_2. (ii) Interstitial Sm_2Fe_{17} carbides ($Sm_2Fe_{17}C_x$) are produced by diffusion of carbon into the Sm_2Fe_{17} structure. Overall reaction:

$$Sm_2Fe_{17}+CH_4 \longleftrightarrow Sm_2Fe_{17}C_x+\alpha\text{-Fe}+SmH_2+C+H_2$$

Closed system. (i) Some of the $Sm_2Fe_{17}H_y$ decomposes into α-Fe + SmH_2. (ii) Interstitial Sm_2Fe_{17} carbohydrides ($Sm_2Fe_{17}C_yH_x$) are produced by diffusion of carbon into the $Sm_2Fe_{17}H_y$ structure. Overall reaction:

$$Sm_2Fe_{17}H_y+CH_4 \longleftrightarrow Sm_2Fe_{17}C_xH_y+\alpha\text{-Fe}+SmH_2+C+H_2$$

(d) Between 600 and 700 °C

Open or closed system. (i) The $Sm_2Fe_{17}C_x$ or $Sm_2Fe_{17}C_xH_y$ decomposes in the presence of hydrogen to produce $Fe_3C+\alpha$-Fe + samarium hydrides. Overall reaction:

$$Sm_2Fe_{17}C_y(H_y)+CH_4 \longleftrightarrow Fe_3C+\alpha\text{-Fe}+SmH_{2+x}+C+H_2$$

(e) Above 700 °C

Open or closed system. (i) Hydrogen desorption of samarium hydride SmH_{2+x} into samarium hydride $SmH_{2-x}+H_2$. Subsequent partial disociation of SmH_{2-x} into $Sm+H_2$ at about 750–850 °C. (ii) Partial intrinsic decomposition of CH_4 to produce $C(s)+2H_2(g)$ at about 800–850 °C. (iii) Possible production of some samarium carbides. Overall reaction:

$$SmH_{2+x}+CH_4 \longleftrightarrow \alpha\text{-Fe}+SmH_{2-x}+SmC_y+C+H_2(g)$$

3.2. Reaction of Sm_2Fe_{17} with other hydrocarbons

The methane-produced Sm_2Fe_{17} carbide contains a substantial amount of α-Fe (see *e.g.* Fig. 2(e)). It is desirable, though, to produce an almost single-phase Sm_2Fe_{17} carbide for future application of the carbonation process for the production of permanent magnets. In the light of this prospect, other hydrocarbons have been examined for the preparation of Sm_2Fe_{17} carbides. The idea was the following. The α-Fe precipitates during the carbonation process because of the presence of hydrogen. Hydrogen is produced during decomposition of methane, which is a saturated hydrocarbon (H:C = 4.0). Therefore other hydrocarbons could be used which during decomposition produce smaller H:C ratios and/or for which the produced hydrogen recombines selectively with other gaseous species (unsaturated hydrocarbons) to form other gas products instead of reacting with Sm_2Fe_{17}. Hydrocarbons such as *n*-butane (H:C = 2.5), *n*-hexane (H:C = 2.0), *m*-xylene (H:C = 1.25), toluene (H:C = 1.14), benzene (H:C = 1.0) and acetylene (H:C = 1.0) were chosen in this respect; the last four are unsaturated hydrocarbons involving double or triple bonds. The X-ray diffraction patterns of the *n*-butane-, toluene- and acetylene-produced Sm_2Fe_{17} carbides are shown in Figs. 3(a)–3(c) respectively. It is generally observed that as the H:C ratio decreases, the production of undesirable α-Fe also decreases. In particular, reactions involving unsaturated hydrocarbons show less production of α-Fe. Specifically, acetylene was found to be the most effective hydrocarbon for the carbonation process, producing the least amount (less than 5 wt.%) of α-Fe (Fig. 3(c)). This particular sample was investigated further in terms of carbon concentration and magnetic and structural properties.

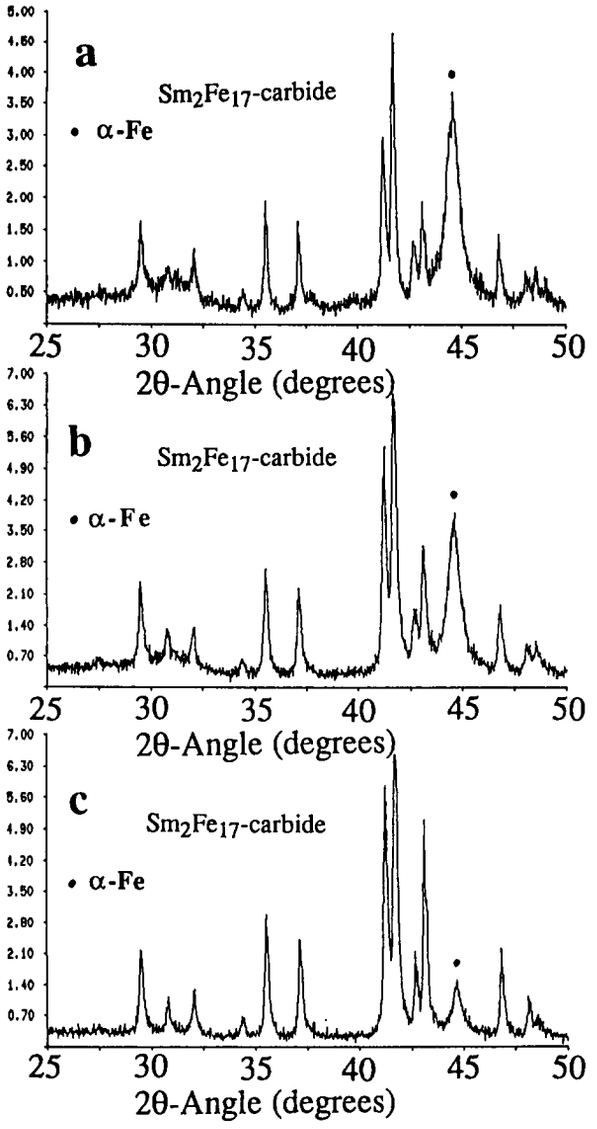

Fig. 3. X-ray (Cu Kα) diffraction patterns of Sm_2Fe_{17} carbides prepared at 600 °C for 1 h under flow of (a) *n*-butane, (b) toluene and (c) acetylene.

3.3. *Acetylene-produced $Sm_2Fe_{17}C_x$*

3.3.1. *Concentration of interstitial carbon*

The determination of carbon concentration by the weight difference before and after carbonation is not a reliable method because of the physically deposited carbon on the surface of the particles. However, it has been found that for carbon concentrations $x \leqslant 1$ the volume of the rhombohedral cell of the arc-melted carbide is proportional to its interstitial carbon concentration. On the basis of the assumption that the linearity extends to higher carbon concentrations, it is possible to estimate the carbon concentration in the acetylene-produced carbide or any carbide if the volume of its rhombohedral cell is known.

The lattice parameters and volumes of the rhombohedral cell of $Sm_2Fe_{17}C_x$ with three different carbon concentrations are listed in Table 1. The parameters for the carbide with $x=1$ were obtained after Kou *et al.* [5]. The samples with $x=0$ and 0.5 were prepared in our laboratory by arc melting and subsequent heat treatment at 1000–1200 °C for several days. By fitting the data for the rhombohedral cell volume as a function of interstitial carbon concentration (Fig. 4) and substituting for the cell volume of the acetylene-produced carbide, it is estimated that its carbon concentration is $x=2.6$. Therefore the composition of the acetylene-produced carbide is $Sm_2Fe_{17}C_{2.6}$.

TABLE 1. Rhombohedral lattice parameters (a, c) and cell volumes (V) of $Sm_2Fe_{17}C_x$ for different carbon concentrations (x)

x	a (Å)	c (Å)	V (Å^3)
0.00	8.549	12.441	787.53
0.50	8.599	12.444	796.87
1.00	8.634	12.460	804.44
2.60	8.744	12.572	832.42

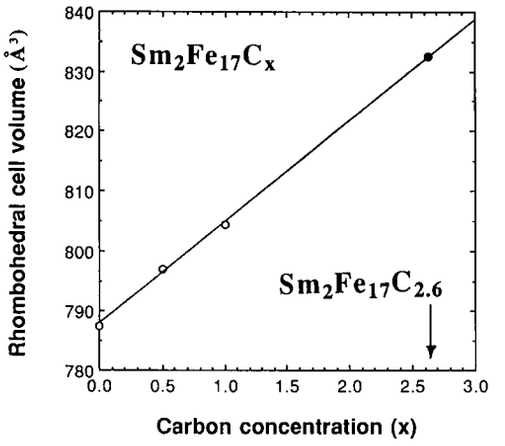

Fig. 4. Rhombohedral cell volume as a function of interstitial carbon concentration (x) of $Sm_2Fe_{17}C_x$. The solid circle corresponds to the acetylene-produced carbide.

It is known that carbon and nitrogen are located at the 9*e* interstitial sites of the rhombohedral structure. The nitrogen concentration is known to be $x=2.3$–2.7 [11, 12] and therefore is about the same as the carbon concentration in the carbide. The value of $x=2.6$ is about the same as that ($x \approx 2.5$) obtained by Liao *et al.* [8] but higher than that ($x \approx 2$) reported by Coey *et al.* [6]. Hong Sun *et al.* [9] reported a value of $x=2.3$. These discrepancies are apparently due to the different methods employed for the estimation of the interstitial carbon content. Nevertheless, they all indicate that gas phase carbonation produces interstitial carbides with high carbon content.

3.3.2. *Anisotropy field*

The acetylene-produced Sm_2Fe_{17} carbide exhibits strong uniaxial magnetocrystalline anisotropy. This was

confirmed by the c axis texture observed in the X-ray diffraction pattern of the aligned powder (Fig. 5). Magnetization curves (Fig. 6) obtained parallel and perpendicular to the c axis of the Sm_2Fe_{17} carbide show an estimated anisotropy field of 120 kOe. This is an approximate value because the maximum available magnetic field was only 15 kOe, which is much lower than the anisotropy field. Bo Ping and Gui-Ghuan Liu [7] and Liao *et al.* [8] reported very similar values of 135 and 140 kOe respectively. The anisotropy field of the carbide is compatible with that obtained for the Sm_2Fe_{17} nitrides (140 kOe) [4]. The high uniaxial anisotropy is attributed to the presence of interstitial N or C atoms around the Sm ions, which give rise to a large negative A_2^0 crystal field parameter [13–15]. The saturation magnetization of the carbide at 25 °C in an applied magnetic field of 15 kOe is 128 e.m.u. g^{-1}.

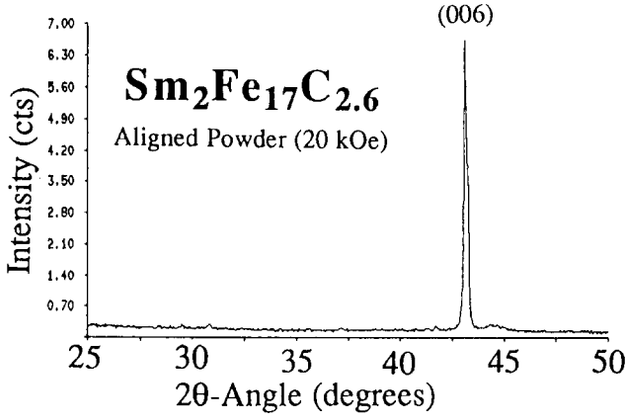

Fig. 5. X-ray (Cu Kα) diffraction pattern of $Sm_2Fe_{17}C_{2.6}$ aligned powder.

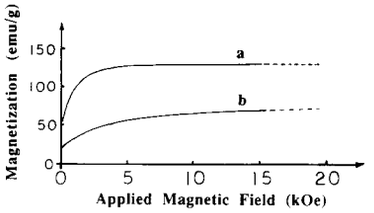

Fig. 6. Magnetization *vs.* magnetic field behaviour used for estimation of the anisotropy field of $Sm_2Fe_{17}C_{2.6}$: a, parallel to the c axis; b, perpendicular to the c axis. The sample was premagnetized in a pulse field of 75 kOe.

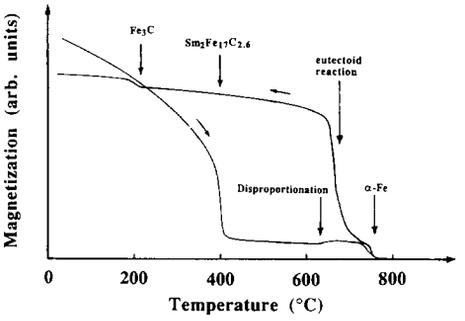

Fig. 7. TMA trace of $Sm_2Fe_{17}C_{2.6}$.

3.3.3. *Thermomagnetic analysis*

Thermomagnetic analysis (TMA) of the acetylene-produced carbide shows that its Curie temperature is 400 ± 2 °C (Fig. 7). This value is in agreement with those obtained by Coey *et al.* [6] ($T_c = 400$ °C), Bo Ping and Gui-Ghuan Liu [7] ($T_c = 397$ °C) and Chen *et al.* ($T_c = 407$ °C). On the other hand, the value of $T_c = 487$ °C was reported by Liao *et al.* [8]. On heating, the carbide decomposes at about 650 °C to form α-Fe as indicated by the increase in the magnetization on the TMA trace. On cooling, it was found that the metastable Fe_3C ($T_c = 230$ °C) is also one of the decomposition products. Other possible decomposition products (in the absence of hydrogen) are samarium carbides and Fe–Sm–C ternaries. The inflection point at 727 °C is most likely due to the eutectoid reaction [16]

$$\gamma\text{-Fe} \longleftrightarrow \alpha\text{-Fe} + Fe_3C$$

4. Summary

Interstitial Sm_2Fe_{17} carbides were prepared by reacting Sm_2Fe_{17} powder with hydrocarbons at 550–600 °C. An almost single-phase carbide (less than 5 wt.% α-Fe) can be prepared by using acetylene. The composition of the carbide is estimated to be $Sm_2Fe_{17}C_{2.6}$. The byproducts of the reaction are α-Fe and samarium carbohydrides. At temperatures higher than 600 °C, decomposition of the carbide occurs (in the presence of hydrogen) to form Fe_3C, α-Fe and samarium carbohydrides. The Curie temperature of the carbide is 400 °C, the saturation magnetization is 128 e.m.u. g^{-1} (25 °C, 15 kOe) and its estimated anisotropy field about 120 kOe at 25 °C. On the basis of the resulting intrinsic magnetic properties, the produced carbide becomes a potential candidate for permanent magnets. Consequently, the gas phase carbonation of Sm_2Fe_{17} by reaction with hydrocarbons becomes a promising method for the production of such permanent magnets.

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