Substitutional and interstitial modification of Nd_5Fe_{17}

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Abstract

The intermetallic compound Nd_5Fe_{17} has potentially important magnetic properties and is being investigated as a possible permanent magnet material, since the partial replacement of Nd by Sm leads to uniaxial anisotropy. We have produced and studied materials with compositions $Nd_5(Fe,X)_{17}$ (X = Co, Mn, Ti, Cr, Al), $(Nd,Sm)_5Fe_{17}$, and $(Nd,Sm)_5(Fe,X)_{17}$ (X = Co, Ti). Only $(Nd,Sm)_5(Fe,Ti)_{17}$ has substantially improved magnetic properties in comparison with binary Nd_5Fe_{17}. Ti substitution, however, leads to a more rapid formation of the 5:17 phase than in the binary compound. This paper also reports structural and magnetic measurements on deuterided Nd_5Fe_{17} and $(Nd,Sm)_5(Fe,Ti)_{17}$. The deuterides are similar to the hydrides in that 14–16 atoms are absorbed per formula unit. The increases in the lattice parameters were larger than in the binary compound: $\Delta a/a = 4.3\%$ and $\Delta c/c = 4.6\%$ for $Nd_5Fe_{17}D_{15.5}$ and $\Delta a/a = 5.3\%$ and $\Delta c/c = 5.2\%$ for $(Nd,Sm)_5(Fe,Ti)_{17}D_{14}$. For Nd_5Fe_{17}, deuterium absorption results in a ~6% increase in M_s, while, for $(Nd,Sm)_5(Fe,Ti)_{17}$, M_s increases by less than 1%. These measurements are consistent with Mössbauer spectroscopy results presented here. These results are discussed in light of recent band structure calculations. $(Nd,Sm)_5(Fe,Ti)_{17}$ was reacted with NaN_3 in an attempt to produce a stable 5:17 nitride. Materials reacted at temperatures 523–623 K for 3–20 h showed multiple phases. One of the phases had $T_c \sim 571$ K.

1. Introduction

Compounds which are being examined for new permanent magnets are principally those derived from the $CaCu_5$ structure, including the 2:17, 1:12, and 3:27 phases. The coordination of the atoms in these compounds is substantially similar to that in the $Nd_2Fe_{14}B$ phase [1], which is the most important high energy product magnet phase in current use. In view of the similarity of these compounds, it seems unlikely that greatly enhanced performance will be achieved through modifications beyond those already explored in the 2:17 phases (interstitial and substitutional changes) and in $Nd_2Fe_{14}B$.

Nd_5Fe_{17} is a stable phase [2,3] with potentially important magnetic properties. The compound is ferromagnetically ordered with a Curie temperature above 500 K. Although the pure Nd compound has basal plane anisotropy, partial replacement of the Nd by Sm induces uniaxial anisotropy. The anisotropy field of $Sm_{3.3}Nd_{1.7}Fe_{17}$ has been estimated [4] to be on the order of 177 kOe, with a Curie temperature of about 540 K. Binary Nd_5Fe_{17} can be hydrided [5] and the hydride ($Nd_5Fe_{17}H_{16}$) has $T_c \sim 573$ K and M_s about 20% larger than the non-hydrided material. Mössbauer spectroscopy studies [5,6] of the binary compound suggest that the Fe moments are larger than in other binary NdFe alloy systems, which increases its interest as a potential high energy product magnetic material.

The crystal structure of Nd_5Fe_{17} was solved by Moreau et al. [7] using single crystal X-ray diffraction. The structure contains 14 crystallographically distinct Fe sites and 7 Nd sites in a wide variety of environments having greater diversity than in the $CaCu_5$ derived structures. The local environments of the Fe atoms are expected to influence the magnetic exchange interactions and the local moments on the Fe sites. High resolution neutron diffraction was used [8,9] to refine the crystallographic

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and magnetic structure of Nd_5Fe_{17}. The results of the Rietveld refinement yield improved precision for the Fe coordinates relative to the X-ray study. Thus, the Fe moment distribution in binary Nd_5Fe_{17} was found to be in good agreement with recent electronic structure calculations [10]. The site occupancy of Sm in $(Nd,Sm)_5(Fe,Ti)_{17}$ was also studied by high resolution neutron diffraction [11]. It was found that Sm prefers the Nd-24l site, which is characterized by very short R−R bonds. Sm was also found on the Nd-2a and Nd-6g sites, with few Fe neighbors [11].

Although some of these materials have been studied previously using neutron diffraction [11,12], it is the purpose of this paper to present all results on interstitial and substitutional modifications of Nd_5Fe_{17}. This paper thus reports structural and magnetic measurements on deuterided Nd_5Fe_{17} and $(Nd,Sm)_5(Fe,Ti)_{17}$. The deuterides are similar to the hydrides in that 14–16 atoms are absorbed per formula unit. Another interesting candidate for interstitial modification of Nd_5Fe_{17} is nitrogen. Previous attempts [5,14] to interstitially modify Nd_5Fe_{17} using gaseous nitrogen were not successful, but reaction with NaN_3 [15] has proven to be an interesting alternative to gaseous reaction in the case of the 2:17 phases. Recent calculations of the magnetovolume effect in Nd_5Fe_{17} [16,17] have predicted a ~34% increase in the average Fe total exchange parameter for a ~6% volume expansion. Thus, we attempted to interstitially modify the 5:17 phase by reacting it with NaN_3.

2. Experiment

The samples used in this work involved transition metal and Sm substitutions: $Nd_5(Fe,X)_{17}$ (X = Co, Mn, Ti, Cr, Al), $(Nd,Sm)_5Fe_{17}$, and $(Nd,Sm)_5(Fe,X)_{17}$ (X = Co, Ti) and were produced by combining high purity elements in an arc furnace. In the case of the samples containing Sm, it was necessary to add extra quantities of Sm to compensate for weight loss during the fusion. Assuming the weight loss during fusion to be due only to the Sm, the final compositions of these samples were calculated. Calculated compositions were generally in very good agreement with compositions determined in a scanning electron microscope (SEM) by energy dispersive spectroscopy (EDS) measurements. After melting, samples were wrapped in Ta foil, sealed in quartz tubes under an Ar atmosphere, and annealed at 953 K for up to 60 days. Thermomagnetic analysis (TMA) curves showed the samples to be single phased. Final compositions and Curie temperatures of samples studied are given in Table 1 below.

Deuterium was introduced into the samples at room temperature in a Sievert system. From the pressure drop occurring in the system, it was estimated that the binary sample absorbed about 15.5 deuterium atoms per formula, while the Sm, Ti substituted sample absorbed approximately 14 deuterium atoms per formula.

The nitrogen pressure–temperature isochore for Nd_5Fe_{17} has been shown previously [5]. Initial nitrogen absorption starts around 523 K and gradually increases with temperature. Slightly above 673 K, there is a change in the slope of the absorption curve corresponding to the beginning of disproportionation, which continues to higher temperatures. The proximity of the disproportionation event to the initial nitrogen absorption is similar to the situation encountered in Sm_2Fe_{17}. In this work, reaction with NaN_3 was carried out for both binary Nd_5Fe_{17} and $(Nd_{0.7}Sm_{0.3})_5(Fe_{0.95}Ti_{0.05})_{17}$. Annealed buttons of the alloy were reduced to < 100 μm powder by hand grinding with an agate mill. NaN_3 powder of the same granulometry was added, supposing that roughly 16 N atoms per formula could be accommodated in the lattice. After wrapping the powder in a Ta foil and sealing it in a steel tube under a nitrogen atmosphere, the mixture was reacted at temperatures 523–623 K for 3–20 h. After the reaction, the sodium was removed and the powder was then washed, dried, and pressed into pellets for measurement.

Magnetization measurements were carried out using a vibrating sample magnetometer (VSM) mounted either in a 2T electromagnet or a 9T superconducting coil. X-ray diffraction measurements were made on a Philips model PW1710 diffractometer with CuK_α radiation, using a zero-background sample holder. Data were obtained for $5° < 2\theta < 80°$ at intervals of $0.05°$ with a holding time of 40 s at each angle. The Rietveld analysis of the X-ray data was performed using the DBWS program.

3. Results and discussion

3.1. 5:17 Phase formation

Samples with compositions $Nd_5(Fe,X)_{17}$ (X = Co, Mn, Ti, Cr, Al) were produced as described above (additional descriptions at Ref. [13]). TMA measurements did not show a magnetic transition for Cr and Al substitutions, indicating either that the 5:17 phase did not form or that the Curie temperature was below room temperature. In the case of both Co and Mn substitutions, substantial T_c reductions were observed: $\Delta T_c = -120$ K for 5% Co and $\Delta T_c = -54$ K for 5% Mn. See Table 1 below. Only Ti substitutions resulted in improved T_c values. Sm substitution eventually results in uniaxial anisotropy [4] for elevated Sm content (above $Sm_{3.3}Nd_{1.7}Fe_{17}$). Several lower Sm substitutions had been investigated earlier in order to avoid heavy neutron absorption [11]. Thus, emphasis was given to substitutions of both Sm and Ti, since it is probable that a practical material based on the 5:17 phase would involve these two substitutions. Additionally, in the case of Sm,Ti substituted samples, the time necessary to form the 5:17 phase was reduced from nearly 60 days to only 32 days. Fig. 1 shows the evolution of a sample initially containing the 2:17 phase toward the 5:17 phase.

3.2. Structure

The Ti-containing samples investigated here show small amounts of an unidentified second phase, estimated to be less than 5% in volume. A sample with 10% Ti had been found to contain about 3% $TiFe_2$ [11], but none of the 2:17 phase, which had been identified in the Ti-free samples. As Sm is added to the alloy, the lattice parameters decrease, as expected. The cell volume of the Ti-containing sample was found to be larger than that of the Ti-free sample [11], indicating that Ti had been incor-

Table 1
Compositions and Curie temperatures of the precursor (without deuterium or nitrogen) samples studied

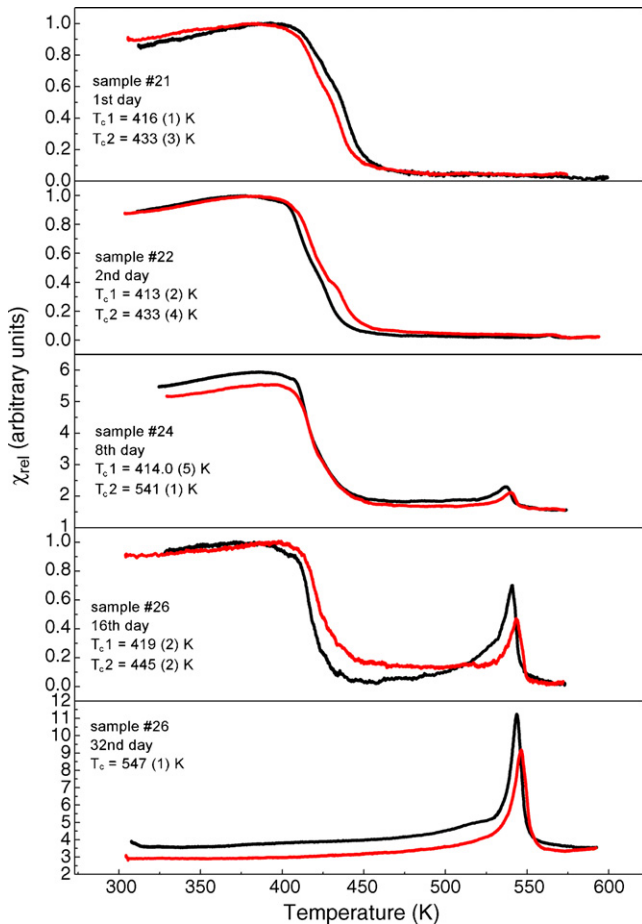| Sample number	Composition	T_c (K)
1	Nd_5Fe_{17}	508
2	$Nd_5(Fe_{0.95}Co_{0.05})_{17}$	388
3	$Nd_5(Fe_{0.90}Co_{0.10})_{17}$	462
4	$Nd_5(Fe_{0.95}Mn_{0.05})_{17}$	454
5	$Nd_5(Fe_{0.90}Mn_{0.10})_{17}$	414
12	$Nd_{4.5}Sm_{0.5}Fe_{17}$	511
13	$Nd_4Sm_1Fe_{17}$	514
14	$Nd_{3.5}Sm_{1.5}Fe_{17}$	533
15	$Nd_{3.0}Sm_{2.0}Fe_{17}$	514
16	$Nd_{3.0}Sm_{2.0}Fe_{17}$	539
19	$Nd_{3.5}Sm_{1.5}Fe_{15.3}Ti_{1.7}$	543
20	$Nd_{3.5}Sm_{1.5}Fe_{16.15}Ti_{0.85}$	540

Fig. 1. Formation of the 5:17 phase (with $T_c = 547$ K) in Sm,Ti substituted samples as seen in TMA measurements. Heating (red) and cooling (black) curves show good agreement. (For the interpretation of the reference to colour in this figure legend, the reader is referred to the web version of the article).

Fig. 2. X-ray diffraction data and Rietveld refinement for (a) Nd_5Fe_{17}; (b) $Nd_5Fe_{17}D_{15.5}$; (c) $(Nd,Sm)_5(Fe,Ti)_{17}$; and (d) $(Nd,Sm)_5(Fe,Ti)_{17}D_{14}$.

porated into the unit cell. Previously, it had been found that, in the Ti-free samples, Sm appears to have a high affinity for the 2a site, which has only rare earth nearest neighbors. Significant amounts of Sm were also found at the 24l site, which has unusually short bonds to its rare earth neighbors, and at the 6g site. When Ti is added, substantial Sm is also encountered at the 4d site, the most highly coordinated site. The highest concentrations of Ti are observed at the $12k_1$, $12k_2$, $12j_1$, and $12j_2$ sites which are 12-fold coordinated with 9 Fe neighbors. This is quite different from the situation in the 2:17 phase where Ti is incorporated at the 6c dumbbell sites which have the longest average bond lengths and 14 neighbors.

The 5:17 structure absorbs substantial quantities of hydrogen or deuterium. Previously we had reported [5] the formation of the hydride $Nd_5Fe_{17}H_{16}$. This is a much larger hydrogen absorption than that which occurs during the formation [18] of a hydride from Nd_2Fe_{17} ($Nd_2Fe_{17}H_x$, $0 \le x \le 5$) or $Nd_2Fe_{14}B$ ($Nd_2Fe_{14}BH_3$). Our results for deuterium are consistent with those of Ref. [5]: $Nd_5Fe_{17}D_{15.5}$ and $(Nd,Sm)_5(Fe,Ti)_{17}D_{14}$ correspond to a very large deuterium absorption. A recent discussion of interstitial sites in the 5:17 lattice [12] noted that it was a very close packed structure, possessing only two possible interstitial sites in the Nd column which could accommodate up
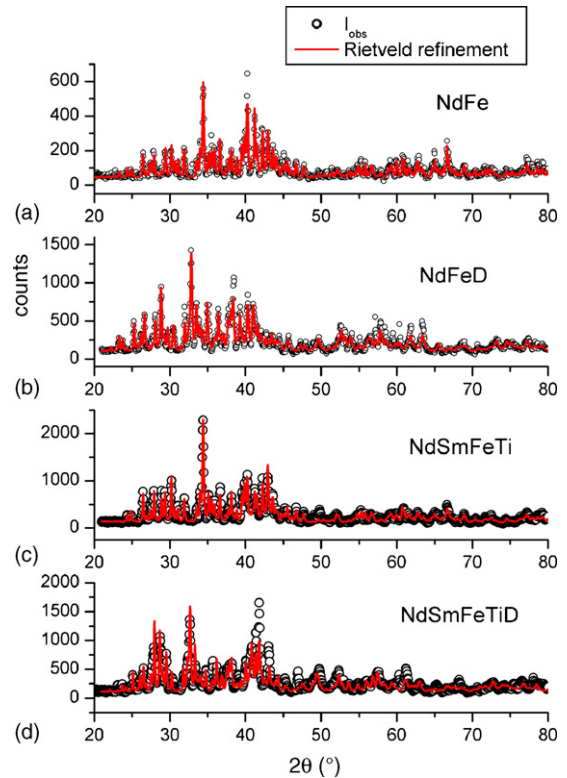
to 14 atoms per unit cell (2 atoms with a Wigner–Seitz radius of 0.73 Å and 12 atoms with radius of about 0.85 Å). However, we have observed H/D absorption corresponding to 168–192 atoms per unit cell, since there are 12 formulas per unit cell of Nd_5Fe_{17}. Since hydrogen absorption requires a hole with a radius of about 0.40 Å [19], we conclude that, on a smaller scale, the 5:17 structure is very open. Neutron diffraction experiments were analyzed using Fourier maps in an attempt to locate the D in the 5:17 structure [19]. About 15 sites, partially or fully occupied by D, were identified, the vast majority being tetrahedral sites, with one octahedral site and some other unusual environments with five metal neighbors.

Lattice parameters were determined from the Rietveld analysis of the X-ray data [12]. See Fig. 2. Values for all samples are given in Table 2, where the increase in a and c for the deuterides can be found. There is good agreement with values obtained previously [19] from neutron data. We see that D absorption resulted in large lattice increases, comparable to that seen previously for the hydrides [5]. As was observed in that case, the 5:17 structure has been maintained.

3.3. Magnetization

The magnetization was measured as a function of magnetic field for $T = 4.2$–300 K. The data were then extrapolated to infinite fields using the function $M = M_s (1 - a/H - b/H^2)$ to obtain M_s. The results for $\mu_o M_s$ (T) are shown in Fig. 3. We see that for Nd_5Fe_{17}, deuterium absorption results in a ~6% increase in M_s, but for $(Nd,Sm)_5(Fe,Ti)_{17}$, M_s increases by less than 1%.

Table 2
Room temperature lattice parameters for the 5:17 alloys, obtained from a Rietveld analysis of X-ray diffraction data

Nd_5Fe_{17}	$a = 20.23$ Å	$c = 12.34$ Å		
$Nd_5Fe_{17}D_{15.5}$	$a = 21.1$ Å	$c = 12.9$ Å	$\Delta a/a = 4.3\%$	$\Delta c/c = 4.6\%$
$(Nd,Sm)_5(Fe,Ti)_{17}$	$a = 20.23$ Å	$c = 12.36$ Å		
$(Nd,Sm)_5(Fe,Ti)_{17}D_{14}$	$a = 21.3$ Å	$c = 13.0$ Å	$\Delta a/a = 5.3\%$	$\Delta c/c = 5.2\%$

Previously [5] we had observed that the hydride ($Nd_5Fe_{17}H_{16}$) has $T_c \sim 573$ K and M_s about 20% larger than the non-hydrided material. The unexpectedly small increase in M_s for the Sm,Ti substituted material with deuterium addition was checked by Mössbauer spectroscopy. Room temperature spectra are shown in Fig. 4.

The Mössbauer spectra shown in Fig. 4 are of considerable complexity due to the substitution of Ti for Fe and Sm for Nd. Furthermore, they bear little resemblance to spectra previously presented for Nd_5Fe_{17} [6] and $Nd_5Fe_{17}H_x$ [5]. In the case of the binary compound, a substantial increase in the hyperfine field was observed upon hydrogenation, consistent with the 20% increase in M_s observed for those materials. A qualitative analysis of the spectra of Fig. 4 leads to the conclusion that the local environments of the Fe atoms are substantially different than in the binary compound and that deuteration does not affect them as hydrogenation does the binary compound. The relative constancy of the hyperfine field for the materials of Fig. 4 is consistent with the results obtained for M_s on these materials.

Calculations [16,17] of the electronic structure of Nd_5Fe_{17} have been performed to investigate the magnetovolume effect in this material. Calculations for the room-temperature, experimental lattice volume and as well as for a volume increased by 6% were performed to study the dependence of T_c and M_s on volume. The calculated density of states (DOS) shows a high degree of spin polarization with the main peaks of the majority spin below the Fermi level, while the minority spin DOS have deep minima at the Fermi energy. The majority spin DOS were found to be more sensitive to the volume and hybridization with neighboring atoms. Those calculations predicted a modest 6% increase in the local magnetic moments and magnetization for the 6% volume increase. For the hydride, we observed a 20% increase in M_s associated with $\Delta v/v \sim 13.8\%$, in rough agree-
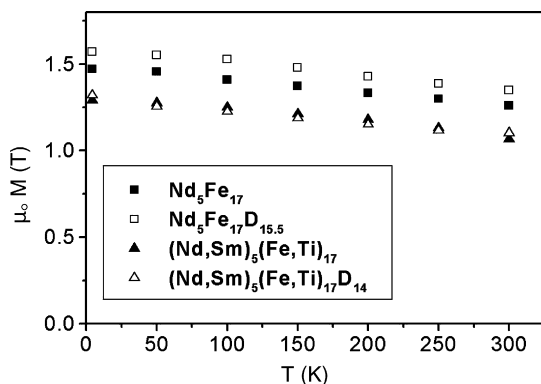
ment with the calculations. However, for the binary deuteride, $\Delta M_s \sim 6\%$ M_s is associated with $\Delta v/v \sim 13.2\%$. For the deuteride of the Sm,Ti substituted material, a change $\Delta M_s \sim 1\%$ M_s is associated with $\Delta v/v \sim 15.7\%$. As we remarked previously, the Mössbauer spectra shown in Fig. 4 bear little resemblance to spectra previously presented for Nd_5Fe_{17} [6] and $Nd_5Fe_{17}H_x$ [5]. These latter results already suggest a profoundly different average local environment for the Fe atoms in the Sm,Ti substituted material compared to the binary compound, while the changes caused by deuteriding also result in greatly modified hyperfine fields. The fact that the 5:17 structure is maintained suggests that volume effects themselves may be secondary, while changes in hybridization due to the presence of hydrogen or deuterium orbitals may be the more important factor. The variety of results (saturation moment, hyperfine field) obtained for the four cases considered here leave no doubt that we are not dealing with a simple magnetovolume effect.
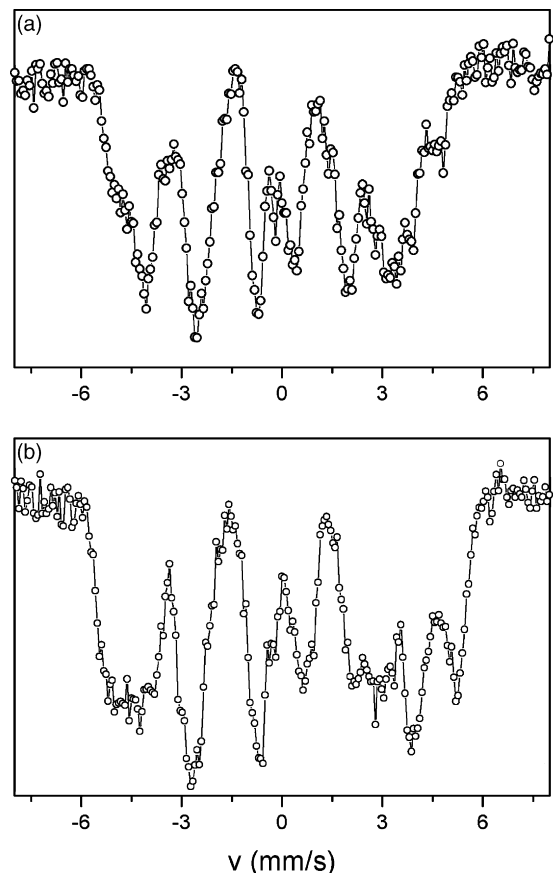

Fig. 4. Mössbauer spectra at room temperature for $(Nd,Sm)_5(Fe,Ti)_{17}$ and $(Nd,Sm)_5(Fe,Ti)_{17}D_{14}$. Solid lines are a guide to the eye.

Fig. 3. Saturation magnetization M_s vs. temperature for the samples studied.

Table 3
Results of reacting $(Nd_{0.7}Sm_{0.3})_5(Fe_{0.95}Ti_{0.05})_{17}$ with NaN_3

Sample	T_{anneal} (K)	t_{anneal} (hours)	T_{c1} (K)	T_{c2} (K)
4	573	20	512	–
5	573	3	507	537
7	623	3	534	571
8	623	20	517	562
9	623	6	539	571

T_c values were obtained from a derivative of the $M(T)$ cooling curve. Measurements made with a VSM.

3.4. Nitrogen modification

Coey and Sun [20] have shown how interstitial modification with nitrogen can dramatically improve the magnetic properties of the 2:17 phase. However, previous attempts [5,14] to interstitially modify Nd_5Fe_{17} using gaseous nitrogen were not successful. In this work, reaction with NaN_3 was carried out for both binary Nd_5Fe_{17} and $(Nd_{0.7}Sm_{0.3})_5(Fe_{0.95}Ti_{0.05})_{17}$. This technique had previously [21,22] proven successful in introducing nitrogen into $NdFe_{11}$. Annealed buttons of the alloy were reduced to powder with a diameter less than $100\,\mu m$ by hand grinding with an agate mill. NaN_3 powder of the same granulometry was added, supposing that roughly 16N atoms per formula could be accommodated in the lattice. After wrapping the powder in a Ta foil and sealing it in a steel tube under a nitrogen atmosphere, the mixture was reacted at temperatures 523–623 K for 3–20 h. After the reaction, the sodium was removed and the powder was then washed, dried, and pressed into pellets for measurement. Some results are presented in Table 3 above.

The magnetization $\mu_o M$ versus temperature T for $(Nd_{0.7}Sm_{0.3})_5(Fe_{0.95}Ti_{0.05})_{17}$, nitrided by reaction with NaN_3 at 623 K for 6 h, is shown in Fig. 5. Calculating a derivative of this curve we detect two events corresponding to $T_{c1} = 539$ K and $T_{c2} = 571$ K. T_{c1} corresponds to the Curie temperature of the non-nitrided material, while T_{c2} must correspond to the nitrided phase. Some α-Fe is also present in this sample. This result shows that the Curie temperature of the 5:17 phase can be elevated about 30 K by reaction with NaN_3, bringing it close to that of $Nd_2Fe_{14}B$.

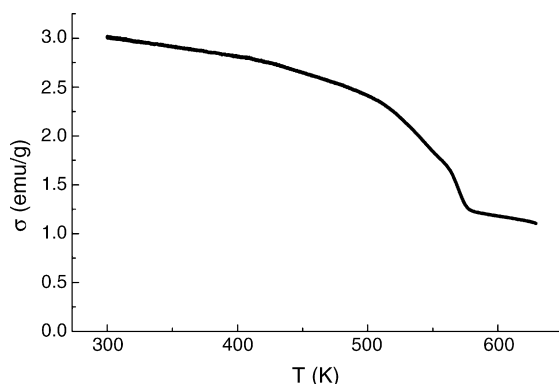Fig. 5. Magnetization $\mu_o M$ vs. temperature T for $(Nd_{0.7}Sm_{0.3})_5(Fe_{0.95}Ti_{0.05})_{17}$ nitrided by reaction with NaN_3.

4. Conclusions

We have produced and studied materials with compositions $Nd_5(Fe,X)_{17}$ ($X =$ Co, Mn, Ti, Cr, Al), $(Nd,Sm)_5Fe_{17}$, and $(Nd,Sm)_5(Fe,X)_{17}$ ($X =$ Co, Ti). Only $(Nd,Sm)_5(Fe,Ti)_{17}$ has substantially improved magnetic properties in comparison with binary Nd_5Fe_{17}. This paper also reports structural and magnetic measurements on deuterided Nd_5Fe_{17} and $(Nd,Sm)_5(Fe,Ti)_{17}$. Large lattice parameter changes were observed for all materials with deuterium absorption. However, our measurements show that the magnetic properties may be more sensitive to hybridization changes than suggested by recent band structure calculations. $(Nd,Sm)_5(Fe,Ti)_{17}$ was reacted with NaN_3 in an attempt to produce a stable 5:17 nitride. Materials reacted at 623 K for 6 h showed multiple phases. One of the phases had $T_c \sim 571$ K, suggesting that this might be a promising route toward obtaining a practical magnetic material.

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