

Magnetic properties of $\text{Tm}_2\text{Fe}_{17-x}\text{Si}_x$ alloys

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The cell parameters and magnetic properties of alloys with approximate composition $\text{Tm}_2\text{Fe}_{17-x}\text{Si}_x$ and various Tm:(Fe+Si) ratios are reported. All the investigated compounds were found to crystallize in the hexagonal $\text{Th}_2\text{Ni}_{17}$ type. The magnetic properties of polycrystalline samples were investigated in the temperature range 1.9-1100 K in various magnetic fields. The field dependence of the magnetization was studied at $T = 4.2$ and 77 K in a steady magnetic field up to 14 T, and additionally in a pulsed magnetic field up to 36 T at $T = 4.2$ K with a pulse duration of 10 ms. The temperature dependence of the magnetization of the alloys exhibits pronounced maxima located at various temperatures, most probably related to spin reorientation and other magnetic anomalies. These anomalies seem to indicate also ferrimagnetic character of the magnetic order, resulting from opposite directions of the magnetic moments of Fe and Tm in the investigated compounds. Additional confirmation of this supposition is the lack of substantial difference between ZFC (zero-field cooled) and FC (field cooled) runs, and the absence of full saturation of magnetization, even under the highest magnetic field. The Néel (Curie) temperatures determined from the dM/dT plots amount to 383 K for $x = 0$ and 466 K for $x = 3$. For the saturation magnetic moment in a magnetic field of about 35 T at $T = 4.2$ K the opposite tendency is observed. The highest value was determined for pristine $\text{Tm}_{2.1}\text{Fe}_{17.9}$ ($M_s = 35.2 \mu_B$) and the lowest value for $\text{Tm}_{2.1}\text{Fe}_{14.9}\text{Si}_3$.

Ternary rare-earth silicides / Magnetic properties / Magnetic phase transitions / High-magnetic field measurements

Introduction

Binary rare-earth (R) – transition metal (T) compounds of the 2:17 type, with $T = \text{Fe}$ or Co , have attracted much attention during the past decades because of their promising magnetic properties and potential application as materials for hydrogen storage. These compounds crystallize in the hexagonal (H) $\text{Th}_2\text{Ni}_{17}$ -type structure (for heavier rare-earths) or in the rhombohedral (R) $\text{Th}_2\text{Zn}_{17}$ -type structure (predominantly for lighter rare-earths). Both types are derivatives of the CaCu_5 -type structure and can be obtained by replacing one-third of the R atoms by a dumbbell of T atoms. Two different ways of this substitution lead to the above-mentioned structures (see *e.g.* [1]). The pristine compound $\text{Tm}_2\text{Fe}_{17}$ is reported to be ferromagnetic (ferrimagnetic) in accordance with coupling rules for the T and R

sublattices. However, its Curie point, like those of other 2:17 compounds of heavier rare-earths, has an anomalously low value ($T_C = 278$ K [2] without magnetic field and 285 K [3] in a magnetic field) in comparison with other R -Fe intermetallics of similarly high Fe concentration. Moreover, $\text{Tm}_2\text{Fe}_{17}$ exhibits a spin reorientation temperature at $T_{SR} = 74$ K (see *e.g.* [2]). These values are too low to be interesting for applications. This apparently originates in the short Fe-Fe separation for the dumbbell site, which weakens the overall positive exchange coupling, and lowers the Curie temperature. Negative exchange coupling is observed when the Fe-Fe spacing is smaller than about 0.242 nm. However, the $R_2\text{Fe}_{17}$ compounds possess the highest saturation magnetization among all binary R - T intermetallics [1]. As mentioned above, $\text{Tm}_2\text{Fe}_{17}$ exhibits two anomalies in the temperature dependence of magnetization [2] and Mössbauer

Table 1 Crystallographic data for Tm–Fe–Si alloys with the hexagonal $\text{Th}_2\text{Ni}_{17}$ structure at RT.

Alloy	<i>a</i> , nm	<i>c</i> , nm	<i>V</i> , nm ³	Remarks
$\text{Tm}_{2.1}\text{Fe}_{17.9}$	0.84052(2)	0.82797(3)	0.50657(3)	
$\text{Tm}_{2.1}\text{Fe}_{16.9}\text{Si}$	0.83919(2)	0.82696(3)	0.50436(3)	single crystal, <i>T</i> = 10–12 K <i>T</i> = 480 K
	0.8388	0.8218	0.5007	
	0.8426	0.8263	0.5080	
$\text{Tm}_{2.1}\text{Fe}_{15.9}\text{Si}_2$	0.83894(2)	0.82732(3)	0.50427(2)	
$\text{Tm}_{2.1}\text{Fe}_{14.9}\text{Si}_3$	0.83768(7)	0.82651(9)	0.50227(8)	
$\text{Tm}_{2.5}\text{Fe}_{15.5}\text{Si}_2$	0.84007(5)	0.82592(6)	0.50478(6)	
$\text{Tm}_{3.0}\text{Fe}_{15}\text{Si}_2$	0.84298(4)	0.82553(6)	0.50804(5)	$\text{Th}_6\text{Mn}_{23}$ -type phase (39 %)
	1.19680(4)		1.71423(9)	

hyperfine field [3]. The low-temperature transition is related to the change of the easy direction of magnetization from an easy plane to a hard *c*-axis above T_{SR} [4]. Band structure calculations have recently been performed on $R_2\text{Fe}_{17}$ compounds, where *R* is a heavy rare-earth or yttrium, by using the TB-LMTO-ASA method [5]. They allow concluding that both local *4f*-*5d* and *5d*-*3d* short-range interactions contribute to the *5d* band polarization, however, they do not provide a clear indication concerning how to improve the magnetic properties. Many investigations have shown that the T_C of 2:17 alloys can be increased by substitution of a variety of other elements for Fe. One of these elements can be Si and it is most remarkable that non-magnetic Si replacing Fe increases T_C . The effect comes from the increase of the Fe-Fe distance at the dumbbell sites and suppresses the negative contribution to the exchange (see *e.g.* [6]). Another explanation for the influence of substitution upon magnetization is given in [7] with the assumption that the substitution of *e.g.* Si for Fe raises the Fermi energy, E_F , without drastic changes of the *3d* band. At the beginning of the substitution (the beginning of raising E_F) the magnetization decreases gradually with increasing *x* (*e.g.* $\text{Y}_2\text{Fe}_{17-x}\text{Si}_x$), because the difference between the densities of states (DOS) of up-spin and down-spin at E_F is small. Subsequently, the magnetization decreases steeply with increasing *x*, because the DOS of up-spin decreases and the DOS of down-spin increases at E_F . The analysis of the Mössbauer ^{57}Fe spectrum shows that Si mainly substitutes for 12*j*-site Fe [8].

Up to now, according to our best knowledge, hexagonal ternaries with Si have been investigated in a broader composition range for *R* = Y [7–12], Sm [10], Tb [13–15], Ho [15], Lu, and U [11]. From the system Tm–Fe–Si only selected compositions have been examined [2,16,17]. Moreover, the composition of these alloys did not correspond exactly to the 2:17 stoichiometry. Finally, X-ray diffraction (XRD) and neutron diffraction (ND) of $\text{Ce}_2\text{Fe}_{17}$ suggest that in this compound some distortion of the rhombohedral structure may occur at LT [18,19]. For these reasons we decided to investigate carefully the structure and magnetic properties of Tm–Fe–Si alloys with approximate $\text{Tm}_2\text{Fe}_{17-x}\text{Si}_x$ composition with $x \leq 3$ in

broad temperature and magnetic field ranges, mostly on polycrystalline samples.

Experimental

All the samples were prepared in Lviv by melting the components in an arc furnace under an Ar protective atmosphere, and annealing them at 900°C in vacuum for 1 week. X-ray diagrams were recorded in Lviv on HZG-4a (Fe) and DRON-2.0 (Fe) diffractometers at room temperature. The purity of the samples was only checked by powder XRD. Additionally, an examination of a single crystal of $\text{Tm}_{2.1}\text{Fe}_{16.9}\text{Si}$ at low temperature (10–12 K) and above the Curie point (387 K) was carried out in Wrocław. Unfortunately the dimensions of the single crystal were not large enough for magnetic studies. The magnetization was investigated on polycrystalline samples in the temperature range 1.9–400 K, in a magnetic field of 5 kOe, in zero-field cooled (ZFC) and field cooled (FC) modes with a SQUID magnetometer, and in a magnetic field of 4.83 kOe at increasing temperature up to 1100 K. The field dependence of the magnetization at 4.2 K (not shown here) and 77 K were measured up to 140 kOe using a string magnetometer, and up to 360 kOe in a pulsed magnetic field, with a pulse duration of 10 ms [20]. All the magnetic measurements were performed in Wrocław.

Results and discussion

a. Crystallographic investigations

Earlier investigations have shown that the pristine 2:17 compounds [21], as well as the Tm–Fe–Si ternaries [16], can exist at a composition differing from the exact 2:17 stoichiometry. Thus we decided to investigate the phase equilibria in the Tm–Fe–Si system [22]. XRD examination revealed that the parent hexagonal $\text{Tm}_2\text{Fe}_{17}$ alloy exists in the range 10.5–12.5 at.% Tm (for crystallographic data see Table 1). The alloy containing 15 at.% Tm turned out to contain two phases: mainly a $\text{Tm}_2\text{Fe}_{17}$ -type phase and 39 wt.% of an impurity $\text{Th}_6\text{Mn}_{23}$ -type phase. For

further experiments we used (in principle) alloys with lower concentration of Tm since closer to the exact stoichiometry. In these alloys the vacancies of Tm atoms in $2b$ sites (16 %) are compensated by pairs of Fe atoms in $4e$ sites. The ternary $\text{Tm}_2\text{Fe}_{17-x}\text{Si}_x$ compounds with 10.5 at.% Tm were found to be single-phase, hexagonal, $\text{Th}_2\text{Ni}_{17}$ -type for Si content amounting to 15 at.%. The lattice parameters of the ternary alloy with Tm concentration of 12.5 %, as well as those observed for a compound with higher Tm content, are listed in [Table 1](#). For the atomic coordinates and the coefficients of occupancy of individual positions see [\[22\]](#).

The suggestions presented in [\[18,19\]](#), as well as the anomalies observed in magnetization at low temperature (see below) inclined us to perform XRD studies of a single crystal of $\text{Tm}_{2.1}\text{Fe}_{16.9}\text{Si}$ at 10-12 K, the lowest temperature attainable in Wrocław for structural analysis, and at a temperature above the Curie point (387 K). The information of Tereshina *et al.* [\[23\]](#) about strong negative thermal expansion along the c -axis below T_C in $R_2\text{Fe}_{17}$ was a reason to perform the high-temperature experiment. The examination in a broad temperature range did not indicate any change of the hexagonal $\text{Th}_2\text{Ni}_{17}$ -type structure. Both lattice parameters, as well as the cell volume, show an increase without any anomaly above T_C . Details of the single crystal structure determination will be published in a separate paper.

b. Magnetic properties

The temperature dependencies of the magnetization of the polycrystalline $\text{Tm}_2\text{Fe}_{17-x}\text{Si}_x$ alloys are presented in [Fig. 1a](#) and [b](#) in the temperature range 1.9-400 K and a magnetic field of 50 Oe. Note that these samples have various Tm:(Fe+Si) ratios, *i.e.* the Tm concentration changes from 10.5 to 15 at.%. However, this variation of the Tm concentration does not influence the general character of the thermomagnetic curves. In [Fig. 1a](#) the results for the pristine alloy and alloys with lower concentration of Si are collected, whereas in [Fig. 1b](#) the data for the alloys with higher Si concentration are shown. The pristine alloy and the alloy with only one Si atom per formula unit exhibit clear maxima at 140 K and 150 K, respectively. These maxima seem to be the spin reorientation temperatures, T_{SR} . Moreover, for the $\text{Tm}_{2.5}\text{Fe}_{16.5}\text{Si}$ alloy at 170 K the magnetization shows an apparent minimum, which can be considered as the compensation point in a ferrimagnetic compound. Above the temperatures of these anomalies the magnetization is weakly temperature-dependent and decreases strongly only after having reached the Curie points (383 and 387 K, respectively, see [Fig. 2](#)). The magnetization curve of the alloy with two Si atoms per formula unit demonstrates a kink at a temperature close to the maxima presented by the former two alloys, then, at about 200 K, a broad maximum is seen that could be related to T_{SR} . Another slight decrease of magnetization can be detected at $T \sim 330$ K. These results can be compared to the

earlier paper [\[2\]](#), in which the temperatures observed for the pristine compound are much lower and the low-temperature anomaly that should correspond to the spin-reorientation temperature is 74 K and T_C amounts to 278 K. For $\text{Tm}_{2.5}\text{Fe}_{15.5}\text{Si}_2$ two anomalies in magnetization were also observed [\[2\]](#): at low temperature a cusp at 182 K, which is higher than observed in the present work (~ 125 K) and $T_C = 461$ K, which is also higher than observed here (436 K). At present we cannot firmly explain these discrepancies in the location of the maxima. One reason could be the difference in experimental conditions since the data provided in [\[2\]](#) were established from Mössbauer effect measurements without magnetic field, another reason could be the small difference in composition of the investigated alloys. A similar difference in T_{SR} values was reported in [\[16\]](#) for aligned powders examined in fields parallel and perpendicular to the alignment. In [Fig. 1b](#) the magnetization of the three remaining samples, measured under the same experimental conditions as in [Fig. 1a](#), is presented. The overall character of the three $M(T)$ curves is similar. There are small cusps at about 100 K and above about 200-210 K the magnetization is weakly temperature-dependent. At present we cannot tell which anomaly is related to T_{SR} . The runs for the samples with two Tm atoms per formula unit are very similar to each other, whereas for the alloy with three Tm atoms the magnetization shows a weak decrease at about 370 K and is clearly lower than for the other two samples. This difference is most probably related to the fact that for three Tm atoms the total magnetization should be lower due to the opposite directions of the magnetic moments in the Fe and Tm sublattices under this relatively low magnetic field. Moreover, this alloy is not single-phase. In [Fig. 2](#) the measurements of magnetization in the temperature range 1.9-1100 K in a magnetic field of ~ 5 kOe for zero-field cooled (ZFC) samples are presented. The results for field cooled (FC) materials are almost identical to those presented in [Fig. 2](#) and are omitted here. One can see that the magnetization increases from relatively low values at low temperature to a maximum, which we consider under these experimental conditions as the temperature of spin reorientation, T_{SR} ([Table 2](#)). Then the magnetization suddenly decreases, going through the Curie point. The Curie points were determined from the dM/dT vs. T dependence (not shown) and are listed in [Table 2](#). One can see that the value of the Curie points increases with increasing Si concentration, as observed for other 2:17 derivatives [\[1,3,6\]](#). A clear difference in the shape of the $M(T)$ plot was observed for $\text{Tm}_2\text{Fe}_{14}\text{Si}_2$ [\[16\]](#), but that experiment was carried out on an aligned powder. As mentioned above, the Curie point and T_{SR} of the pristine 2:17 compound determined in the present work are higher than reported up to now [\[2,6,17\]](#), however, both T_C and T_{SR} of the not numerous ternaries reported earlier are of the same order of magnitude [\[2,16,17\]](#) as our results.

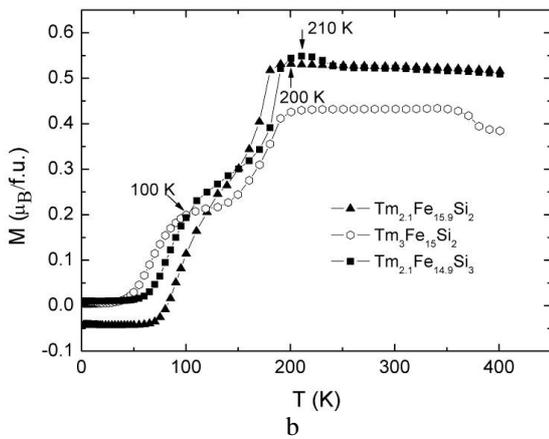
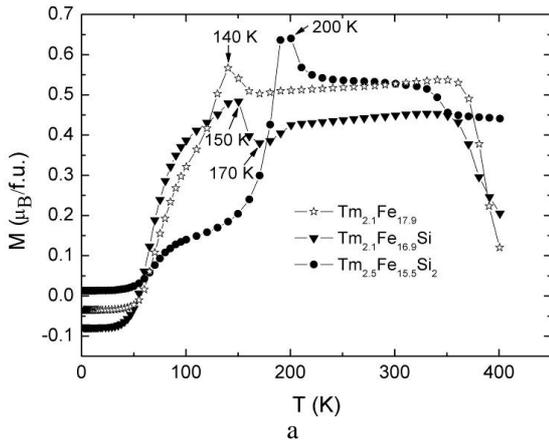


Fig. 1 Magnetization of $\text{Tm}_{2.1}\text{Fe}_{17.9}$, $\text{Tm}_{2.1}\text{Fe}_{16.9}\text{Si}$ and $\text{Tm}_{2.5}\text{Fe}_{15.5}\text{Si}_2$ vs. temperature measured in a magnetic field of 50 Oe (a); magnetization of $\text{Tm}_{2.1}\text{Fe}_{15.9}\text{Si}_2$, $\text{Tm}_{2.1}\text{Fe}_{14.9}\text{Si}_3$ and $\text{Tm}_{3.0}\text{Fe}_{15}\text{Si}_2$ vs. temperature measured in a magnetic field of 50 Oe (b).

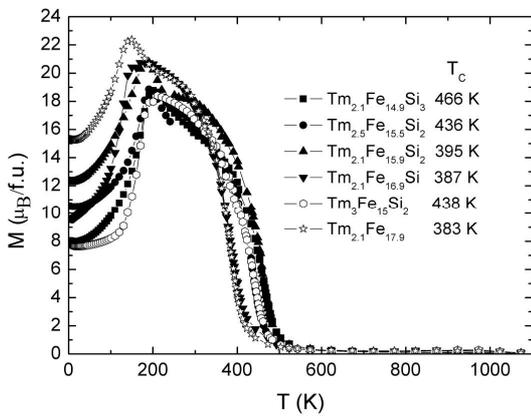


Fig. 2 Magnetization of Tm–Fe–Si alloys vs. temperature measured in a magnetic field of 5.0 kOe.

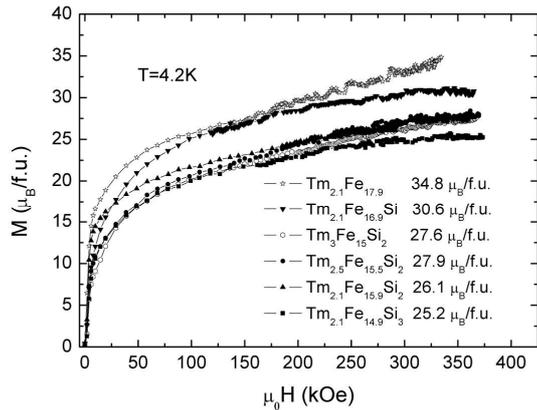


Fig. 3 Magnetization of Tm–Fe–Si alloys vs. a pulsed magnetic field at 4.2 K. The values of the “saturation” magnetic moments are listed in a separate column.

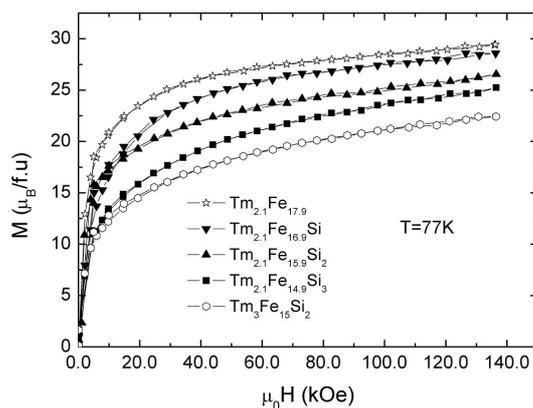
In some of the investigated alloys additional anomalies were observed in the thermomagnetic curves, but without neutron diffraction or Mössbauer effect experiments one cannot give a reasonable explanation. Some indication is provided by experiments on the $\text{Lu}_2\text{Co}_{17-x}\text{Si}_x$ system [24], for which the authors suggest the presence of two second-order spin reorientation phase transitions.

The conclusion from our thermomagnetic curves is that ternaries with substituting Si can have some very limited importance for application because of their relatively high Curie temperatures.

The magnetic field dependence of the magnetization of the ternaries measured at 4.2 K in a steady magnetic field does not exhibit saturation. After a rapid increase of the magnetization at low magnetic field, the increase of the magnetization with increasing magnetic field is much smoother. A small hysteresis is observed and the remanence is rather low (not shown). Fig. 3 presents the magnetization vs. a pulsed magnetic field up to 360 kOe at 4.2 K. It is seen that the curves do not exhibit saturation, even in the highest magnetic field. The almost linear behavior found for the pseudo-binary alloys in the high-field region can be attributed to a rotation of the magnetic moments from their easy direction to the direction of the applied field. The numbers collected in Table 2 are the values obtained in the highest magnetic field. Note that for different samples different magnetic fields have been applied. In spite of this limitation we tried to evaluate the contribution of the Tm atoms in these ternaries. Since Y is a nonmagnetic element, the magnetic properties of Y_2Fe_{17} can be considered to represent the contribution of the Fe-sublattice to the magnetization of $\text{Tm}_{2.1}\text{Fe}_{17.9}$. The saturation magnetic moment of Y_2Fe_{17} according to [6] amounts to $35.7 \mu_B/\text{f.u.}$ at RT and to $34.0 \mu_B/\text{f.u.}$ in $\text{Lu}_2\text{Fe}_{17}$ at LT [21]. For $\text{Tm}_{2.1}\text{Fe}_{17.9}$ we obtain the value $\sim 35 \mu_B/\text{f.u.}$ This minute difference does not allow drawing any firm conclusion. Another observation concerning

Table 2 Magnetic data for Tm–Fe–Si alloys.

Alloy	T_{SR} , K	T_{C} , K	M_{s} , μ_{B} /f.u.	Remarks
$\text{Tm}_{2.1}\text{Fe}_{17.9}$	140	383	34.8	
$\text{Tm}_{2.1}\text{Fe}_{16.9}\text{Si}$	150	387	30.6	minimum in $M(T)$ at 170 K
$\text{Tm}_{2.1}\text{Fe}_{15.5}\text{Si}_2$	~200	395	26.1	
$\text{Tm}_{2.1}\text{Fe}_{14.9}\text{Si}_3$	200	466	25.2	
$\text{Tm}_{2.5}\text{Fe}_{15.5}\text{Si}_2$	200	436	27.9	cuspl at ~120 K
$\text{Tm}_{3.0}\text{Fe}_{15}\text{Si}_2$	200	438	27.6	cuspl at 100 K

**Fig. 4** Magnetization of Tm–Fe–Si alloys vs. a steady magnetic field at 77 K.

the decrease of the “saturation” magnetic moment after substitution of Si for Fe confirms the earlier information about the decrease of magnetization after dilution of the transition metal by a nonmagnetic component, however, this diminution is not proportional to the concentration of the substituting element (see *e.g.* [25]). Fig. 4 shows the magnetization of selected alloys measured in a steady magnetic field at $T = 77$ K. One can see that these plots are very similar to that obtained at 4.2 K. This observation confirms that in a magnetic field above about 20 kOe the magnetic order of the investigated alloys does not change substantially with temperature at LT.

Conclusions

1. The lattice parameters of the $\text{Tm}_2\text{Fe}_{17-x}\text{Si}_x$ alloys were determined at room temperature. The parameter a decreases with increasing x ; the parameter c demonstrates, in principle, the same tendency for the alloys with the same concentration of Tm.
2. The lattice parameters of a single crystal $\text{Tm}_{2.1}\text{Fe}_{16.9}\text{Si}$ were determined at $T = 10$ – 12 K and at $T = 480$ K (above the Curie point).
3. Magnetization measurements were carried out in a broad temperature and magnetic field range. All the alloys exhibit the presence of two anomalies in the temperature dependence of magnetization corresponding to the Curie (Néel) point, T_{C} , and a

temperature of spin reorientation, T_{SR} . T_{C} and T_{SR} , as expected, increase with increasing Si concentration. On the contrary, the values of the magnetic moment obtained in the highest magnetic field decrease with increasing Si content.

4. The magnetic parameters of the investigated compounds do not qualify them as useful materials, however, the observed features allow them to be considered as very interesting from a fundamental point of view. These materials need complementary measurements, such as for example Mössbauer effect studies or neutron diffraction on single crystals.

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