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# Strongly enhanced Curie temperature in carbon-doped Mn<sub>5</sub>Ge<sub>3</sub> films

M. Gajdzik, C. Sürgers\*, M.T. Kelemen, H.v. Löhneysen

Physikalisches Institut, Universität Karlsruhe, D-76128 Karlsruhe, Germany Received 9 May 2000; received in revised form 11 July 2000

#### Abstract

The structural and magnetic properties of  $Mn_5Ge_3C_x$  films prepared at elevated substrate temperatures  $T_S$  are investigated. In particular, films with  $x \ge 0.5$  and  $T_S = 680\,\mathrm{K}$  exhibit a strongly enhanced Curie temperature  $T_C = 445\,\mathrm{K}$  compared to bulk  $Mn_5Ge_3$  with  $T_C = 304\,\mathrm{K}$  while at the same time the average Mn moment decreases from 2.6 to  $1\,\mu_B$ . Structural analysis of these films suggests that the carbon is interstitially incorporated into the voids of Mn octahedra of the hexagonal  $Mn_5Si_3$ -type structure giving rise to a lattice compression. The enhanced ferromagnetic stability in connection with the lattice compression is interpreted in terms of an Mn–Mn interaction mediated by C based on a change in the electronic structure. © 2000 Elsevier Science B.V. All rights reserved.

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# 1. Introduction

Manganese compounds exhibit a variety of properties which are of fundamental interest, such as the discovery of the colossal magnetoresistance in  $La_{1-x}Sr_xMnO_3$  [1] or the quantum-critical behavior of the weak itinerant magnet MnSi [2]. Intermetallic compounds of Mn and Ge occur in several structural phases, which mostly exhibit antiferromagnetic or ferrimagnetic order with rather low ordering temperatures. However,

In contrast, the isostructural  $Mn_5Si_3$  compound is antiferromagnetic with two different magnetic phases below Néel temperatures  $T_N = 68$  and 98 K. Consequently, a transition from antiferromagnetic to ferromagnetic behavior was found in  $Mn_5(Ge_{1-y}Si_y)_3$  alloys in dependence of y [8,9]. More remarkably, the antiferromagnetic  $Mn_5Si_3$  was reported to exhibit ferromagnetic order when doped with carbon  $(Mn_5Si_3C_x)$ , saturating at

 $<sup>\</sup>mathrm{Mn_5Ge_3}$  stands out as a ferromagnet with a Curie temperature  $T_\mathrm{C} = 304\,\mathrm{K}$  and a uniaxial magnetic anisotropy along the *c*-axis of the hexagonal structure [3,4]. The magnetic structure of this compound as studied by neutron scattering [5,6] reveals two Mn sublattices with different magnetic moments resulting in an average ordered magnetic moment of  $2.6\,\mu_\mathrm{B}/\mathrm{Mn}$ -atom [7].

<sup>\*</sup>Corresponding author. Tel.: + 49-721-608-3456; fax: + 49-721-608-6103.

*E-mail address:* christoph.suergers@physik.uni-karlsruhe.de (C. Sürgers).

a doping level x = 0.22 with  $T_C = 152$  K [10]. The interstitial incorporation of carbon into the voids of Mn octahedra expands the lattice slightly and the Curie temperature  $T_{\rm C}$  varies almost linearly with doping level x. Even more interestingly,  $T_C$ could be enhanced up to 350 K by incorporating carbon by simultaneous evaporation of Mn and SiC [11]. A shift from antiferromagnet to ferromagnet and an ensuing  $T_{\rm C}$  increase might be simply due to a lattice expansion as suggested by the famous Bethe-Slater curve [12]. Indeed, the monotonic  $T_{\rm C}(y)$  dependence in  ${\rm Mn}_5({\rm Ge}_{1-y}{\rm Si}_y)_3$  [8] can be interpreted in this way. On the other hand, the incorporation of carbon might change the electronic band structure and thus the Mn-Mn exchange. Recently, it was concluded from zero-field <sup>55</sup>Mn NMR measurements that Mn<sub>5</sub>Si<sub>3</sub>C<sub>1.5</sub> is in fact a ferrimagnet [13]. Hence, because the role of interstitial carbon in influencing the magnetic properties and stabilizing the ferrimagnetic order in Mn<sub>5</sub>Si<sub>3</sub> is presently not clear, it is important for an understanding to investigate the effect of C doping on the isostructural hexagonal ferromagnetic compound Mn<sub>5</sub>Ge<sub>3</sub>.

In this paper, we report on the magnetic and structural properties of  $\mathrm{Mn_5Ge_3C_x}$  films with a strongly enhanced Curie temperature  $T_\mathrm{C} = 445\,\mathrm{K}$  for  $x \geqslant 0.5$  compared to  $T_\mathrm{C} = 304\,\mathrm{K}$  for undoped  $\mathrm{Mn_5Ge_3}$ . Surprisingly, we find that the increase of  $T_\mathrm{C}$  upon C doping is accompanied by a decrease in the lattice constants, thus demonstrating the decisive role of carbon in altering the electronic properties.

## 2. Experimental

 $\rm Mn_5Ge_3C_x$  films (100-nm thick) were prepared by simultaneous DC- and RF-magnetron sputtering from elemental Mn, Ge, and C targets (purity 99.9%) in Ar atmosphere onto (1 $\bar{1}20$ ) sapphire substrates at different substrate temperatures  $T_s$ . They were structurally characterized by X-ray diffraction in a standard powder diffractometer with Cu-K<sub> $\alpha$ </sub> radiation. Magnetic hysteresis loops were measured for temperatures  $T = 2-500\,\rm K$  by the magneto-optical Kerr effect in transverse geometry (t-MOKE), i.e. with the magnetic field aligned in

the film plane and perpendicular to the plane of incidence. In addition, the saturation magnetic moment was obtained by SQUID magnetometry at  $T=5\,\mathrm{K}$  with the magnetic field oriented in the film plane.

#### 3. Results

Fig. 1 shows a  $\theta/2\theta$  X-ray diffractogram of an Mn<sub>5</sub>Ge<sub>3</sub>C<sub>0.75</sub> film prepared at  $T_{\rm S}=680\,{\rm K}$ . The observed diffraction lines can be indexed assuming the hexagonal Mn<sub>5</sub>Si<sub>3</sub>-type structure, apart from the strong substrate reflections and weak reflections due to precipitations of Mn<sub>5</sub>Ge<sub>2</sub> (not ferromagnetic) and elemental Ge. The structure of such ternary T<sub>5</sub>M<sub>3</sub>C<sub>x</sub> compounds, where T is a transition metal and M a metalloid, has been studied in great detail [14,15]. The atomic positions in the hexagonal structure (space group P<sub>6</sub>/mcm) are 4 Mn<sub>I</sub> in 4(d), 6 Mn<sub>II</sub> in 6(g<sub>I</sub>), and 6 Ge in 6(g<sub>II</sub>). The carbon is likely to be incorporated in voids surrounded by distorted Mn<sub>II</sub> octahedra at positions 2(b) without a change of the structure

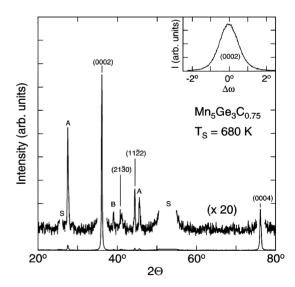


Fig. 1.  $\theta/2\theta$  X-ray diffraction pattern of an Mn<sub>5</sub>Ge<sub>3</sub>C<sub>0.75</sub> film sputtered at  $T_{\rm S}=680\,\rm K$  onto  $(1\,\bar{1}\,20)$  sapphire (Cu K<sub>x</sub> radiation). Miller indices refer to the hexagonal structure (see text). Reflections due to elemental Ge, Mn<sub>5</sub>Ge<sub>2</sub>, and the substrate are labeled A, B, and S, respectively. Inset shows the rocking curve  $(\omega$ -scan) of the  $(0\,0\,0\,2)$  Bragg reflection.

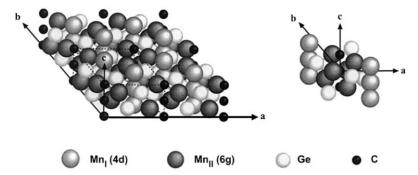


Fig. 2. Left: Structure of hexagonal  $Mn_5Ge_3C_x$  (x = 1.33). The carbon is incorporated into the voids of  $Mn_{II}$  octahedra along the c direction at positions 2(b). Right: Details of the structure showing the  $Mn_{II}$  octahedra around the C interstices.

while a certain percentage of the  $Mn_1$  4(d) positions remain unoccupied [15] (see Fig. 2). Due to the variable occupancy of the voids such phases with filled D8<sub>8</sub> structure (Nowotny phases) are generally non-stoichiometric. X-ray diffractograms of samples prepared at lower and higher  $T_s$  show the formation of a strongly disordered material or precepitation of some Ge-rich alloys, respectively. The observed intensities (Fig. 1) differ from theoretically expected values indicating a strong columnar texture along the growth direction, i.e. the hexagonal c-axis, already reported for sputtered Mn<sub>5</sub>Ge<sub>3</sub> films [16]. The texture is also inferred from the small half-width  $\Delta\omega \approx 1.2^{\circ}$  of the (0002) rocking curve (Fig. 1, inset). We mention that the powder diffraction pattern of Mn<sub>5</sub>Si<sub>3</sub>C<sub>x</sub> films prepared under the same conditions and detached from the substrate can be perfectly fitted by the Nowotny phase [17]. It is therefore very likely that this phase is also formed in  $Mn_5Ge_3C_x$  films.

The lattice parameters determined from the diffraction lines indicate a lattice *compression* caused by the incorporation of C. For x=0.75, we find c=4.996, a=7.135 and c/a=0.700, i.e. a compression in each direction compared to c=5.053, a=7.184, c/a=0.703 for  $\mathrm{Mn_5Ge_3}$  [3]. This is in strong contrast to  $\mathrm{Mn_5Gi_3C_x}$  annealed powder samples ( $x \leqslant 1$ ), where the c/a ratio increases slightly and a remains almost constant upon interstitial insertion of carbon [15].

Carbon doping induces a strong enhancement of the Curie temperature, reaching  $T_{\rm C} = 445 \, \text{K}$ , i.e. well above the room temperature. Fig. 3 shows the

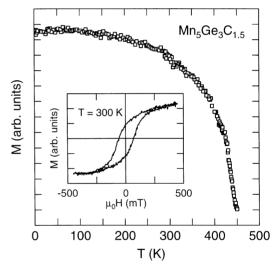


Fig. 3. Temperature dependence of the magnetization M in an applied magnetic field  $\mu_0 H = 300 \,\mathrm{mT}$  measured by t-MOKE for a  $\mathrm{Mn_5}\,\mathrm{Ge_3}\,\mathrm{C}_{1.5}$  film sputtered at  $T_\mathrm{S} = 680 \,\mathrm{K}$ . Inset shows a hysteresis loop M(H) at room temperature.

magnetization M(T) measured by t-MOKE and a hysteresis loop taken at room temperature for a  $\mathrm{Mn_5Ge_3C_{1.5}}$  film ( $T_\mathrm{S}=680\,\mathrm{K}$ ). The distinct deviation from a square loop possibly arises from the easy axis of magnetization pointing out of the film plane, i.e. along the hexagonal c-axis, due to the columnar texture, since the t-MOKE measures the in-plane component of the magnetization. On the other hand, domain formation and/or microstructure effects may also give rise to the observed hysteresis loop.

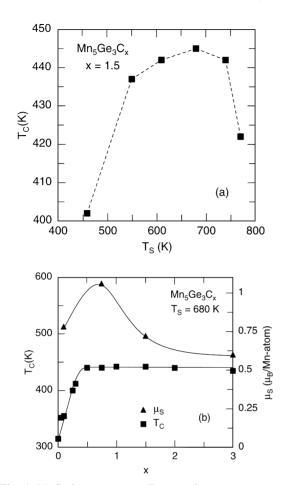


Fig. 4. (a) Curie temperature  $T_{\rm C}$  vs. substrate temperature  $T_{\rm S}$  for sputtered Mn<sub>5</sub>Ge<sub>3</sub>C<sub>1.5</sub> films. (b)  $T_{\rm C}$  and average saturation moment  $\mu_{\rm S}$  in units of  $\mu_{\rm B}/{\rm Mn}$ -atom for Mn<sub>5</sub>Ge<sub>3</sub>C<sub>x</sub> films sputtered at  $T_{\rm S}=680\,{\rm K}$  vs. carbon concentration x. Solid lines serve as a guide to the eye.

Fig. 4a shows  $T_{\rm C}$ , obtained from the extrapolation of the M(T) data to  $M(T_{\rm C})=0$  in a small applied field  $\mu_0H=5\,{\rm mT}$ , vs. substrate temperature  $T_{\rm S}$ . An optimum  $T_{\rm S}\approx 680\,{\rm K}$  can be inferred in agreement with the X-ray analysis indicating nearly single-phase material for this  $T_{\rm S}$ . The formation of the high- $T_{\rm C}$  phase is presumably thermodynamically stabilized by its low free energy together with a high adatom mobility during film growth at elevated temperatures. At lower  $T_{\rm S}$  the process is likely to be kinetically hindered whereas at higher  $T_{\rm S}$  the formation of other crystalline phases is energetically favored.

The effect of carbon doping on the magnetic behavior was investigated in more detail by preparing films with different x at  $T_{\rm S}=680\,{\rm K}.$  Fig. 4b shows  $T_{\rm C}$  and the average saturation magnetic moment  $\mu_S$  plotted vs. carbon content x.  $T_C$  increases linearly for small x and saturates at  $x \ge 0.5$ . Around  $x \approx 0.5-0.75$ , the average magnetic moment reaches a maximum of  $\mu = 1.1 \,\mu_B/Mn$ atom, considerably smaller than  $\mu_{\rm S}$  for Mn<sub>5</sub>Ge<sub>3</sub>, and decreases for larger x. This indicates that the optimum C concentration for the Mn<sub>5</sub>Ge<sub>3</sub>C<sub>x</sub> ferromagnetic phase is around x = 0.75. The saturation of carbon uptake is higher compared to  $Mn_5Si_3C_x$  sintered powder samples ( $x_{max} = 0.22$ ) [10] which indicates that the carbon is distributed homogeneously on an atomic scale during film growth, whereas in the powder samples this process is presumably diffusion limited. This may stimulate further investigations with an aim at incorporating even higher carbon concentrations by employing other preparation techniques far from equilibrium.

From neutron-diffraction data, Forsyth and Brown presented evidence that in several Mnmetalloid binary intermetallic compounds the moment reduction at different Mn sites with respect to the free-ion value is governed by the nearest-neighbor Mn-Mn interaction. Below a critical average Mn-Mn bond length of about 3.1 A, each Mn neighbor reduces the moment of the coordinated Mn atom from the ionic value by about  $2\mu_{\rm B}/A$ , [6]. Applying this empirical relationship to the present samples, an average magnetic moment of about 2.3  $\mu_B$  is expected from the measured lattice parameters. Additional strong reduction observed in the present samples can have three reasons. First, it could be due to some non-magnetic minority phases. However, this would require a high volume fraction of 50% of non-magnetic material at optimal doping  $x \approx 0.75$  which seems unlikely in view of the only weak parasitic X-ray Bragg reflections that could be attributed to Mn<sub>5</sub>Ge<sub>2</sub> or elemental Ge. Second, the relationship between average Mn-Mn bond length and magnetic moment will break down if the carbon participates in the Mn-Mn bonding because the d-band population and therefore  $\mu_{S}$  are modified by the incorporation of carbon into the magnetic host lattice. Such an effect of carbon doping on the band structure has been suggested for the cubic perovskites  $Mn_3MC$  where M is a non-transition metal [18]. Third, an antiferromagnetic coupling between the sublattices on different sites may lead to a ferrimagnet with reduced  $\mu_S$ . Clearly, the magnetic moment at the different Mn sites has to be determined by further microscopic investigations.

For larger concentrations x > 0.75 the average Mn moment decreases whereas  $T_{\rm C}$  remains constant. This behavior is attributed to the increased formation of non-magnetic phases which reduce the amount of the ferromagnetic phase and therefore the average moment, whereas the Curie temperature of the majority phase is still maintained. This is corroborated by an overall decrease of the magnetic signal in t-MOKE with increasing x. Furthermore, the half-width of the (0002) rocking curve increases with increasing x, most likely due to the decreasing columnarity of the multiphase material.

### 4. Discussion

The question arises how the ferromagnetic exchange interaction can be enhanced by the incorporation of carbon. Obviously, only the  $Mn_{II}$  atoms of the octahedra around the C site are affected via sp–d interaction or hybridization. This can also be inferred from NMR data on  $Mn_5(Ge_{1-y}Si_y)_3$  [9]. The  $Mn_{II}$  atoms with a moment of  $\approx 3\,\mu_B$  seem to be strongly ferromagnetically coupled to each other while the coupling between the  $Mn_I$  atoms with  $\mu\approx 2\,\mu_B$  is certainly weaker. Substitution of Si for Ge gives rise to a reduction of the moment at the  $Mn_I$  site whereas the  $Mn_{II}$  moments remain unaffected.

In C-doped Mn<sub>5</sub>Ge<sub>3</sub> two alternative possibilities may lead to the strongly enhanced Curie temperature, involving either a predominantly covalent bonding between Mn and C or an ionic charge transfer. The carbon and the surrounding Mn<sub>II</sub> atoms are possibly covalently bonded by sp-d hybrid orbitals and the local environment of the nearest Mn<sub>II</sub> neighbors around C is comparable to the cubic Mn<sub>3</sub>/M perovskites, where C is incorporated in octahedral interstices of the face-centered cubic

Mn/M-atom array [18]. In the perovskites the band structure is modified by the electronegative interstices so that the broad bands arising from the Mn-M bonding are split by the changed translational symmetry and the occupation of the sp states overlapping with the d bands is changed by Mn-C and M-C interactions. This may stabilize a spontaneous magnetization although the spin configuration can be complex giving rise to a ferromagnetic-to-antiferromagnetic transition at low temperatures [18]. However, for the present samples such band-structure effects are further complicated by the variable filling of the (4d) Mn<sub>I</sub> positions of the Nowotny phase but the general behavior due to the introduction of C serving as an electron acceptor might be similar to  $Mn_3MC$ .

On the other hand, in a simple ionic picture applied to undoped Mn<sub>5</sub>Ge<sub>3</sub>, charge balance requires  $6Ge^{4-}$ ,  $4Mn_I^{3+}$ , and  $6Mn_{II}^{2+}$ . The  $Mn_{II}$  valence is compatible with the magnetization density measured by neutron diffraction [6]. The addition of C will induce a charge transfer from Mn<sub>II</sub> atoms to C, assuming that C is strongly electronegative. This would, for instance, lead to a C<sup>-4</sup> ion surrounded by  $4Mn_{II}^{3+}$  and  $2Mn_{II}^{2+}$  ions at the corners of the octahedron, thus offering the possibility of double exchange leading to ferromagnetic order as originally proposed by Zener [19] and more recently investigated in the colossal magnetoresistance alloys [20]. For ferromagnetic alloys of Mn or Fe the effect of interstitial non-magnetic atoms like B, C, and N, on the magnetic properties has been recently studied theoretically by assuming a Hubbard-split band at half-filling coupled with a localized spin system via exchange coupling [21]. The coupling gives rise to the double-exchange mechanism in the limit of completely localized orbitals. Whether this model or other more realistic double-exchange models including lattice distortions can be applied to the C-doped Mn<sub>5</sub>Ge<sub>3</sub> alloys requires a detailed knowledge of the valences at the different Mn<sub>II</sub> sites. In case of a complete charge transfer we would expect a change of the average valence from 2.4 for Mn<sub>5</sub>Ge<sub>3</sub> to 3 for  $Mn_5Ge_3C_{0.75}$ . We have checked this possibility by performing preliminary X-ray absorption measurements for x = 0 and 0.75. The experiments were done at the beamline A1 at HASYLAB, Hamburg. The absorption edge shifts to higher energies in the C-doped sample by less than  $0.5\,\mathrm{eV}$  which corresponds [22] to an average valence change of less than 0.1, clearly much less than the ionic model would predict. We conclude that the double-exchange mechanism plays only a minor role in enhancing the ferromagnetism in  $\mathrm{Mn}_5\mathrm{Ge}_3\mathrm{C}_x$ .

Although the facts that (i)  $dT_{\rm c}/dp > 0$  for pure  $Mn_5Ge_3$  [23] and (ii)  $T_C$  increases while the volume decreases upon C doping appear to be superficially compatible, it is clear from the above discussion that the enhanced Curie temperature of  $Mn_5Ge_3C_x$  is not due to a simple volume effect. This is supported by the monotonic  $T_{\rm C}(y)$  dependence in  $Mn_5(Ge_{1-\nu}Si_{\nu})_3$  as soon as the antiferromagnetic Mn<sub>5</sub>Si<sub>3</sub> changes to ferromagnetism with increasing Ge concentration. We also note that taking  $dT_C/dp = 5.5 \text{ K/GPa}$  for  $Mn_5Ge_3$  [24] and assuming a bulk modulus of  $K = 110 \,\mathrm{GPa}^1$ would lead to  $dT_C/d(\Delta V/V) = -605 \,\mathrm{K}$  which is incompatible with  $dT_C/d(\Delta V/V) = -1700 \,\mathrm{K}$  derived by simply relating the volume change upon C doping to the  $T_{\rm C}$  change. This confirms our above assignment of a predominantly electronic effect as opposed to a simple volume effect.

# 5. Summary

Incorporation of carbon in  $\mathrm{Mn_5Ge_3}$  compounds leads to a compression of the crystalline lattice and to a strongly enhanced ferromagnetic stability in remarkable contrast to  $\mathrm{Mn_5(Ge_{1-y}Si_y)_3}$  compounds, where  $T_{\mathrm{C}}$  decreases with decreasing lattice parameters. We have presented evidence that this is due to an increased interaction between Mn atoms mediated by interstitially incorporated carbon. The increase in Curie temperature is accompanied by a strong decrease in the average magnetic moment, possibly indicating a transition from ferro- to ferrimagnetism upon C doping of  $\mathrm{Mn_5Ge_3}$ . Neu-

tron scattering and NMR studies are required to determine the magnetic moments at different Mn sites.

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 $<sup>^{1}</sup>$  The bulk modulus was estimated from the elastic moduli of  $Mn_{5}Si_{3}$  [25], by using the Voigt approximation.

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