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Local strain-induced ferromagnetism in inhomogeneous Fe-implanted silicon carbide

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Abstract

Despite an intense research on diluted magnetic semiconductors since twenty years ago, the origin and nature of observed ferromagnetism in these materials remain controversial, the presence of transition metal precipitates hindering to reveal unambiguously the intrinsic ferromagnetic behavior of the semiconducting matrix. In this work, we have investigated the magnetic properties of Fe-doped SiC, composed of Fe-rich nanoparticles and some diluted Fe atoms in the SiC matrix. By a careful analysis of the experimental magnetization data, a magnetic contribution from the diluted Fe atoms in the SiC matrix was evidenced, with a Curie temperature (T_C) value of ~ 115 K. Such a value is much higher than those reported in similar III-V diluted magnetic semiconductors such as a few at.% Mn-doped InP ($T_C = 20-40$ K). This magnetic ordering temperature of the diluted Fe atoms is shown to be independent of the amorphous or crystalline state of the SiC matrix, but depends strongly on the Fe-rich nanoparticle size, with a drastic reduction of T_C down to 45 K observed in the presence of Fe-rich nanoparticles with diameter higher than 10 nm.

Keywords: Diluted Magnetic Semiconductor (DMS), silicon carbide (SiC), Superconducting Quantum Interference Device magnetometry (SQUID), spin electronics.

29 **Introduction**

30 Light doping of semiconductors with a 3d magnetic element (Cr, Mn, Fe, Co) may confer
31 ferromagnetic (FM) properties to the semiconductor material, leading to diluted magnetic
32 semiconductors (DMS) able to be incorporated and operational in spintronic devices [1-2].
33 Since almost two decades, many advances have been made in the growth and annealing of
34 these materials in order to minimize the presence of secondary phases or compensating
35 defects which are detrimental to the desired properties with the goal to achieve Curie
36 temperature (T_C) value higher than 300 K. Many authors have reported observation of
37 ferromagnetism in such materials above room temperature (RT). However, most of these
38 observations later turned out to be partial or full consequence of the presence of precipitates
39 or inclusions of secondary phases of transition metal (TM) compounds in solid solution to
40 which the DMS specimens belong [3-4]. These circumstances stimulated the appearance of
41 theoretical works showing that the FM ordering in such specimens can be partly explained
42 from the exchange interaction between charge carriers and doping magnetic ions [5-6].
43 Despite this intense research in DMS field, no T_C higher than RT has now been obtained and
44 the origin and nature of observed ferromagnetism in these materials remain controversial [7].
45 Recently, a theoretical work [8] has addressed the nature of the ferromagnetism observed in
46 2.5-5 at.%Mn-doped InP [9] and another one [10] based on a self-consistent local random
47 phase approximation (SC-LRPA) approach shed a new light on the complex topic of effects
48 of magnetic inhomogeneities in DMS [11].

49 Among many candidates considered for spin electronics applications (III-V, II-VI
50 compounds) the wide bandgap semiconductor silicon carbide (SiC) has a long history of
51 materials research and device development and SiC-based components have already been
52 commercialized for high frequency and high power applications. Many theoretical works [12-
53 17] have shown that a magnetic moment may be attributed to TM atoms in different SiC
54 polytypes and that electronic and magnetic properties of DMS are significantly influenced by
55 the lattice relaxation [14-17]. Some authors [18] have claimed observation of ferromagnetism
56 in Mn- and Fe-implanted 6H-SiC with T_C as high as 250 K. Typical FM order was also
57 established at around 250 K at as low Mn-doped concentration as 10^{-4} molar fraction in 4H-
58 SiC [19]. These authors found that defects-related effects other than the inserted Mn atoms
59 play the most important role in the magnetic ordering. In addition, Liu et al experimentally
60 evidenced ferromagnetism in 4H-SiC after neutron irradiation [20], suggesting that Si and C
61 vacancies may also play an important role in magnetism in SiC. Despite this significant effort

62 to the theoretical and experimental investigations of TM-doped SiC and other DMS materials,
63 the mechanisms responsible for the polarization and ordering in these materials are still far
64 from being clear [7,15].

65 Ion implantation is now an essential industrial standard technique in doping
66 semiconductors and can be useful for obtaining high-spin configuration in SiC-based DMS
67 [15]. In previously published works, some authors [21-24] have shown that the formation of
68 Fe₃Si nanoparticles is mainly responsible for the magnetic properties observed in 6H-SiC
69 crystals implanted with Fe ions at the 6 at.% concentration and annealed at high temperature
70 (up to 1573 K). However, for Song *et al.* [25] the presence of Fe₃Si is not the nature origin of
71 FM ordering in Fe-doped SiC, and traces of Fe-doping in SiC induce a high temperature FM
72 arrangement. In recent communications [24,26,27], we reported atom probe tomography
73 (APT) investigations on such materials, evidencing for a random distribution of Fe atoms in
74 the SiC matrix and some Fe-rich nanoparticles. The FM behavior observed in hexagonal SiC
75 implanted with 2-6 at.% Fe ions is mainly due to the contribution of Fe-rich nanoparticles, but
76 also to an important part of diluted Fe atoms in the SiC matrix which can get a FM behavior
77 [27-29].

78 The aim of the present work is to fully characterize the magnetic behavior of Fe-
79 implanted SiC (Fe:SiC) with the two main populations of Fe atoms observed by APT in this
80 system: Fe-rich nanoparticles and diluted Fe atoms in the SiC matrix. In the first part,
81 experimental protocols are reported with relevant microstructural details of typical samples.
82 Magnetometry results are presented with a complete treatment of the diamagnetic and
83 paramagnetic contributions of the substrate to the measured signals. In many previously
84 published works on this topic, this point is not well documented and/or taken account
85 consideration, especially in TM-implanted samples in which the implanted thickness is much
86 smaller (a few 100 nm) than the substrate one (a few 100 μm). When only correcting
87 magnetization curves from the diamagnetic component of the substrate, the omission of the
88 paramagnetic one may introduce a strong paramagnetic component in the temperature
89 dependence of the net magnetization curves as we will show below. In the second and third
90 parts, we present and discuss temperature dependence of the magnetization curves of typical
91 samples. Modeling the Fe-rich nanoparticles contribution to the net magnetization of the
92 studied samples allows to isolate the magnetic behavior of diluted Fe atoms and to compare it
93 with the results of recent theoretical works [8,12,15]. It is shown that such a behavior as that
94 observed in Fe-implanted SiC is common to other systems with similar electronic structure

95 like Mn-implanted InP [8], leading to a relatively high $T_C \sim 115$ K for FM diluted Fe atoms in
96 SiC. At last, our results show an influence of big FM Fe-rich nanoparticles on the Curie
97 temperature of the FM diluted Fe atoms.

98 **I) Experimental**

99 Lightly doped (a few 10^{+18} cm^{-3}) n- and p-type bulk 6H-SiC wafers ($e \sim 350$ μm) from CREE
100 company have been implanted with Fe ions (^{56}Fe and also ^{57}Fe ions to enhance the statistics
101 of the ^{57}Fe Mössbauer spectra). The implantation temperature was sufficiently high (653 K or
102 823 K) to prevent amorphization of SiC [30]. Each sample has been implanted with multiple
103 energy Fe ions at different energies and fluences (from 30 to 160 keV at a few 10^{+15} to a few
104 10^{+16} Fe ions/ cm^2) in order to get a uniform Fe atom concentration $C_{\text{atFe}} \sim 5.0$ (+/-1.0)% from
105 20 to 90 nm under the sample surface. One sample was intentionally implanted at low
106 temperature (473 K) and higher fluence in order to get an amorphous SiC matrix.

107 Post-implantation RTA (15 K/s) was performed at 1173 K, 1273 K or 1573 K with N_2
108 circulation in order to prevent accidental pollution. Actual Fe atom concentration profile and
109 average value were measured by Rutherford Backscattering Spectroscopy (RBS) [31] and by
110 EDS respectively.

111 The microstructural characterization of the as prepared samples was made by SEM
112 and APT [32] to get a detailed picture of the Fe atoms distribution inside the SiC matrix.
113 Finally, we used XRD to determine the surface normal strain profile along the c -axis of
114 hexagonal SiC crystal within the implanted region [33-36] (See Supplementary Material for
115 discussion).

116 The magnetic properties of the samples were studied by Superconducting QUantum
117 Interference Device (SQUID), and ^{57}Fe conversion electron Mössbauer spectrometry (CEMS)
118 which is sensitive to the near surface implanted region (~ 100 nm) [37,38]. The CEMS spectra
119 were fitted with the Gunnlaugsson model [39] allowing to determine the proportion of
120 implanted Fe atoms lying at substitutional position (noted Fe_S) into the SiC matrix. Previous
121 works have shown that Fe atom more easily substitutes to Si rather than to C atom in SiC
122 [13,16,40]. Thus, all along this work Fe_S denotes diluted Fe atoms at the Si sites in the SiC
123 matrix. It could also be noted that there are crystallographic inequivalent Si (and C) sites
124 when the first- and second-nearest neighbours are considered in 6H(4H)-SiC: two (6H-SiC) or
125 one (4H-SiC) with cubic-like symmetry for one with hexagonal-like symmetry [41].

126

127 **I-1) Microstructural overview of some typical samples especially discussed in this work**

128

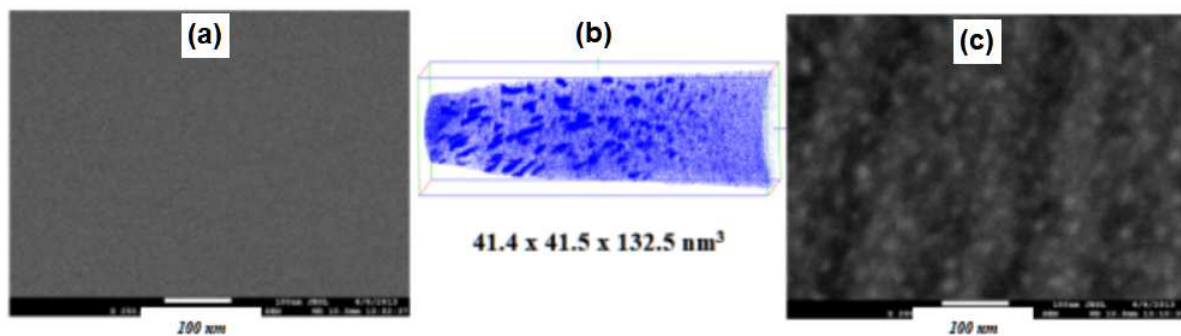
129 Table 1: Characteristics of the studied samples. S_{max} is the maximum value of the surface
130 normal strain (see Supplementary Material); Fe-nanop is the proportion of Fe-implanted
131 atoms in the Fe-rich nanoparticles as obtained from APT and Fe_s is the proportion of Fe-
132 implanted atoms at the Si sites in the SiC matrix as obtained from CEMS as explained above.

SAMPLE	C_{atFe} (%)	T-implantation (K)	T-annealing (K)	S_{max} (%)	Fe-nanop (%)	Fe_s (%)
Sample 1	13	473	as-implanted	-	-	-
Sample 2	6	823	as-implanted	4	27	53
Sample 3	6	823	1173	3.5	44	43
Sample 4	4	823	1273	3.5	-	38
Sample 5	6	823	1573	3	66	24
Sample 6	2	823	1573	-	60	35

133

134 - The as-implanted sample at 473 K ($C_{atFe} \sim 13\%$), under the SiC amorphization critical
135 temperature (~ 500 K) [30], shows an amorphous SiC matrix (RBS/Channeling and XRD)
136 and uniform contrast in SEM (no visible Fe-rich nanoparticles) (Sample 1; Fig.1-a).

137



138

139 Fig. 1: (a): Z-contrast image (SEM with BEI mode; backscattered electrons; HV = 20 kV) of sample 1;
140 (b): APT image revealing Fe atoms spatial distribution in sample 3; (c): Z-contrast image of sample 5

141 (the wave contrast is related to the p-type ($> 10^{19} \text{ cm}^{-3}$; Al doping) epitaxy ($e \sim 200 \text{ nm}$) on n-type
142 bulk substrate).

143

144 - The as-implanted sample at 823 K ($C_{\text{atFe}} \sim 6\%$) shows a crystalline SiC matrix; slight
145 contrasts are visible in SEM; small Fe-rich nanoparticles of mean diameter (diam) $\sim 1\text{-}6 \text{ nm}$
146 are detected by APT (core composition: FeSi₂; FeSi) [24,28] (Sample 2).

147 All the annealed samples at a given temperature (1173 K, 1273 K or 1573 K) with
148 $C_{\text{atFe}} \sim 5.0 (\pm 1.0\%)$ exhibit virtually similar microstructure independently of the implantation
149 temperature (653 K or 823 K) and thus of their as-implanted state. It has been ensured that for
150 more than 4 minutes RTA, there is no evolution of the microstructure and magnetic
151 properties. Thus, all RTA were done for a 4 minutes duration.

152 - The sample implanted at 823 K ($C_{\text{atFe}} \sim 6\%$) and annealed at 1173 K shows a crystalline
153 SiC matrix with a weak extra XRD peak (other than allowed 6H-SiC peaks; interreticular
154 distance $d \sim 0.2 \text{ nm}$ and Scherrer diameter $\sim 5 \text{ nm}$). Slight contrasts are visible in SEM; Fe-
155 rich nanoparticles (diam $\sim 1\text{-}8 \text{ nm}$) are detected by APT (core composition: FeSi; Fe₅Si₃;
156 Fe₂Si) [24,28] (Sample 3; Fig. 1-b).

157 - The sample implanted at 653 K ($C_{\text{atFe}} \sim 4\%$) and annealed at 1273 K shows a crystalline
158 SiC matrix with an extra XRD peak ($d \sim 0.2 \text{ nm}$ and Scherrer diameter $\sim 6 \text{ nm}$) and pockets
159 on which a translation Moiré appears in TEM ($d \sim 0.2 \text{ nm}$); slight contrasts are visible in SEM
160 [23,28] (Sample 4).

161 - The sample implanted at 823 K ($C_{\text{atFe}} \sim 6\%$) and annealed at 1573 K shows a crystalline
162 SiC matrix with an extra XRD peak ($d \sim 0.2 \text{ nm}$ and Scherrer diameter $> 6 \text{ nm}$) and pockets
163 on which a translation Moiré appears in TEM ($d \sim 0.2 \text{ nm}$). Clear contrasts are visible in
164 SEM; Fe-rich nanoparticles (diam $\sim 1\text{-}8 \text{ nm}$) and a few big (diam $\sim 9\text{-}12 \text{ nm}$) Fe₃Si
165 nanoparticles are detected by APT (core composition: FeSi; Fe₅Si₃; Fe₂Si; Fe₃Si) [24,28]
166 (Sample 5; Fig.1-c).

167 - The sample implanted at 823 K ($C_{\text{atFe}} \sim 2\%$) and annealed at 1573 K (Sample 6) with small
168 (diam $< 8 \text{ nm}$) superparamagnetic Fe-rich nanoparticles has been previously studied in
169 [26,27,29].

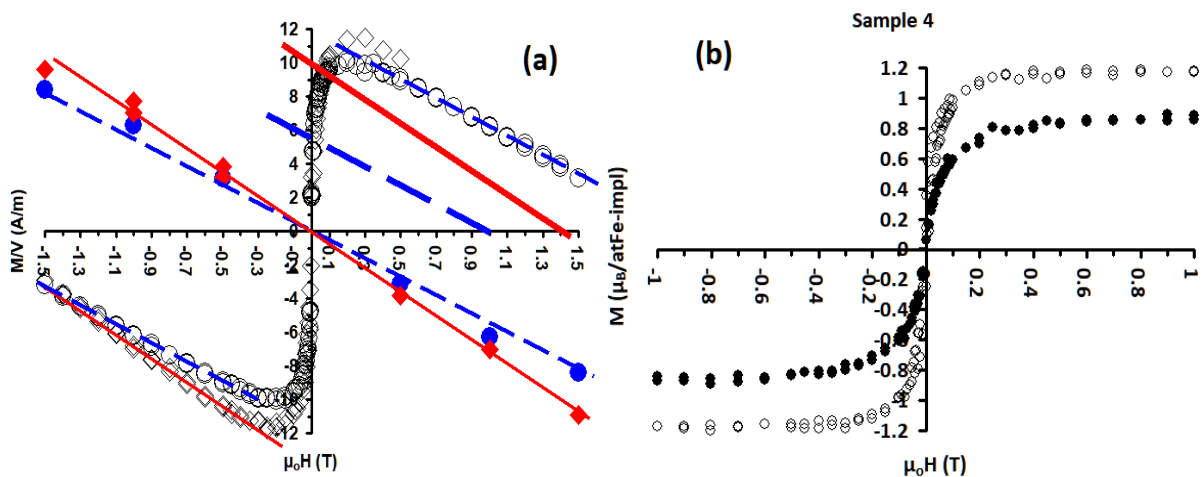
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I-2) Magnetometry

171 Magnetization loops $M(H)$ were measured at 10 K and 250 K by SQUID with an applied field
 172 $\mu_0 H$ varying from 0 to ± 2 T. As we can see on Fig. 2-a, the slope of SQUID signals at high
 173 field (> 0.3 T) is clearly different at 10 K and 250 K. For unimplanted and Fe-implanted
 174 samples, its value is ~ -5.5 (A/m)/T at 10 K and ~ -7.13 (A/m)/T at 250 K. Observation of
 175 the same slope for unimplanted and implanted samples shows no (or very weak) contribution
 176 of paramagnetic Fe-implanted atoms. Thus, this slope is mainly due to the diamagnetic
 177 component and to the paramagnetic defects of the bulk substrate 6H-SiC. These values lead to
 178 a slope $\sim -7.2 + 17 / T(K)$ [(A/m)/T] what is in the range of what is observed in the literature
 179 for SiC. For example, the value of the calculated diamagnetic component for SiC is -7.0
 180 (A/m)/T [42] and the paramagnetic component corresponds to a concentration of
 181 paramagnetic defects $\sim 2.7 \times 10^{18} \text{ cm}^{-3}$ for $3.2 \times 10^{18} \text{ cm}^{-3}$ observed by Wang [43] in lightly
 182 doped 6H-SiC from KMT company. Thus, all along this work we use the above value of the
 183 slope to properly eliminate the substrate contribution from the measured SQUID signals of the
 184 studied samples. Doing this for all the studied samples, saturation magnetization is observed
 185 and reached at $\mu_0 H \sim 0.5$ T at 10 K and 250 K, as shown on Fig. 2-b for sample 4.

186

187



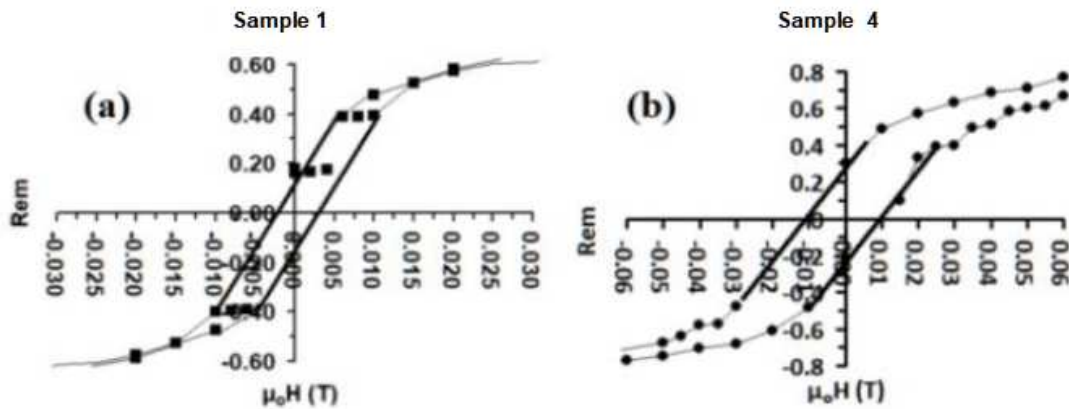
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189 Fig. 2: (a): SQUID magnetization curves of four SiC samples: unimplanted sample (full circles) and
 190 Fe-implanted sample (sample 1, empty circles) measured at 10 K: slope = -5.5 (A/m)/T (dotted lines);
 191 unimplanted sample (full rhombus) and Fe-implanted sample (sample 5, empty rhombus) measured at
 192 250 K: slope = -7.13 (A/m)/T (continuous lines). (b): Magnetic moment of sample 4 as a function of

193 the applied field $\mu_0 H$ in Tesla (T): measured at $T = 10$ K (empty circles) and $T = 250$ K (full circles).
194 The value of the magnetic moment is given in μ_B per Fe-implanted atom ($\mu_B/\text{at-Fe impl}$) where μ_B is
195 the Bohr magneton.

196

197 Open cycles are observed at 10 K (Fig. 3). At 250 K the cycles are closed or under
198 resolution, except for sample 5. For sample 1 (Fig. 3-a) with no Fe-rich nanoparticle, the FM
199 behavior at 10 K is clearly evidenced.



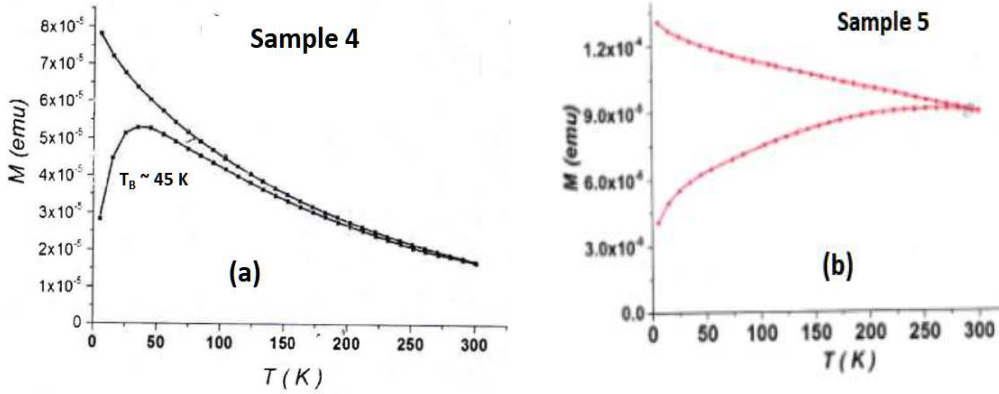
200

201 Fig. 3: FM loops at 10 K. (a): Sample 1: Magnetization remanence (Rem) $\sim 10\%$ and coercive field
202 (H_C) ~ 2.5 mT; (b): Sample 4: $Rem \sim 25\%$ and $H_C \sim 10$ mT.

203

204 For the other samples containing Fe-rich nanoparticles, this demonstrates the FM
205 behavior at 10 K and the superparamagnetic behavior at 250 K, what is confirmed by FC/ZFC
206 curves at low field as shown on Fig. 4 with a blocking temperature $T_B \sim 45$ K for sample 4
207 (Fig. 4-a).

208



209

210

Fig. 4: FC/ZFC curves at low field ($\mu_0H = 0.01$ T). (a): Sample 4; (b): Sample 5.

211

For sample 5 with big (diam ~ 9 -12 nm) Fe_3Si nanoparticles, the ZFC curve shows no maximum and magnetization increases continuously with increasing temperature (Fig. 4-b). This shows the FM character of this sample as for a solid sample with $T_B > 300$ K. Indeed, a slight open cycle is observed at 250 K ($Rem \sim 10\%$ with a $H_C \sim 5$ mT) and CEMS at 300 K shows a clear signature of a FM Fe_3Si component [28].

216

Due to the complex Stoner-Wohlfarth behavior of the superparamagnetic Fe-rich nanoparticles at low temperature and low field, remanent magnetization could be difficult to interpret for samples containing Fe-rich nanoparticles due to the mixing of their FM behavior for $T < T_B$ with that of diluted Fe atoms as shown in Fig. 3-a for sample 1 with no Fe-rich nanoparticle. Thus, in order to properly quantify the magnetic behavior of the different Fe phases observed in the studied samples (Fe-rich nanoparticles and diluted Fe atoms in the SiC matrix) magnetization curves as a function of temperature $M(T)$ have been measured at saturation with an applied field $\mu_0H = 0.5$ T from 10 K to 250 K.

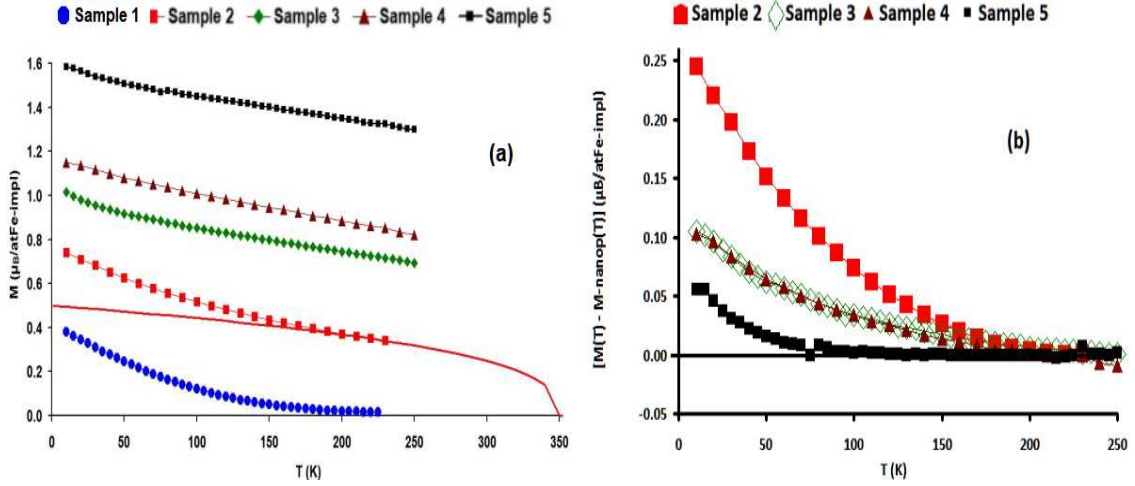
224

II) Results and discussion

225

As shown on Fig. 5-a, the $M(T)$ curve of sample 1 (with no Fe-rich nanoparticle) reaches ~ 0 at high temperature with a “highly unconventional non-Brillouin-function-like character” [8]. In other samples with Fe-rich nanoparticles $M(T)$ is smoother and higher, revealing the magnetization of superparamagnetic Fe-rich nanoparticles with a $T_C \geq 350$ K.

228



229

230 Fig. 5: (a): Saturation magnetization of samples 1-5 as a function of temperature. Line: Brillouin-
 231 function-like magnetization of Fe-rich nanoparticles for sample 2 calculated from equation (1) with
 232 $M(0)\text{-nanop} = 0.5 \mu_B/\text{atFe-impl}$ and $T_C\text{-nanop} = 350$ K (see later and Table 2). (b): $[M(T) - M\text{-}$
 233 $\text{nanop}(T)]$ for samples 2 to 5.

234 In order to identify the contribution of the Fe-rich nanoparticles and the contribution
 235 of the diluted Fe atoms in the SiC matrix to the net magnetization, it is possible to describe the
 236 temperature dependence of the saturation magnetization of the superparamagnetic Fe-rich
 237 nanoparticles by the following relation:

$$238 \quad M\text{-nanop}(T) = M(0)\text{-nanop} (1 - T/T_C\text{-nanop})^\beta \quad (1)$$

239 where $M(0)\text{-nanop}$ is the value of their saturation magnetization at $T = 0$ K, $T_C\text{-nanop}$ is their
 240 Curie temperature, and β is the critical exponent with $\beta = 0.36$ for Heisenberg ferromagnets
 241 [44-45]. As previously noted in [29], equation (1) is unable to reproduce the full behavior of
 242 $M(T)$ curves namely at low temperature as shown in Fig.5 for sample 2. Then, fitting the
 243 maximum number of points of high temperature part of all these curves with equation (1)
 244 leads to the values of $M(0)\text{-nanop}$ and $T_C\text{-nanop}$ reported in Table 2.

245

246 Table 2: $M(0)\text{-nanop}$ and $T_C\text{-nanop}$ are relative to Fe-rich nanoparticles. $M(0)\text{-diluted}$ and $T_C\text{-diluted}$
 247 are relative to diluted Fe atoms in the SiC matrix and were obtained as explained in the text below.
 248 The values of $M(0)\text{-nanop}$ and $M(0)\text{-diluted}$ are in $\mu_B/\text{atFe-impl}$.

SAMPLE	$M(0)\text{-nanop}$	$T_C\text{-nanop}$ (K)	$M(0)\text{-diluted}$	$T_C\text{-diluted}$ (K)
Sample 1	-	-	0.40	110

Sample 2	0.5	350	0.275	110
Sample 3	0.89	500	0.10	110
Sample 4	1.055	515	0.11	120
Sample 5	1.535	700	0.07	45
Sample 6	1.285	615	0.14	120

249

250 Subtracting this contribution from the $M(T)$ curves for samples 2 to 6, a residual contribution
251 appears which can be identify with the contribution of the diluted Fe atoms in the SiC matrix
252 ($M(T)$ -diluted). All these $M(T)$ -diluted = $M(T) - M\text{-nanop}(T)$ curves (Fig. 5-b) exhibit the
253 same behavior as the $M(T)$ curve of sample 1 (Fig. 5-a) showing that diluted Fe atoms of
254 these samples with nanoparticles have the same magnetic behavior as Fe atoms in sample 1
255 with no nanoparticle.

256

257 **II-1) Theoretical analysis**

258 Los et al [15] theoretically studied purely diluted 4 at.%Fe-doped hexagonal SiC (with no
259 nanoparticules or other kind of defects) with *ab initio* calculations taking “fully account for
260 the effects of crystal lattice reconstruction and electronic structure changes caused by
261 substitution of Si atoms by Fe atoms”. They show that “substitution of 4% of Si atoms in the
262 SiC lattice with Fe atoms leads to an emergence of two impurity bands in the gap close to the
263 valence band top. The Fermi level is then pinned in the middle of the lower-energy Fe band
264 with close to 100% spin polarization” together with a lattice relaxation along the **c**-axis of the
265 hexagonal SiC crystal. Fe atom being at a hexagonal-like Si site, the C atom above the Fe atom
266 among the four C atoms forming the elemental tetrahedra around Fe atom have the strongest
267 influence on the electronic and magnetic configurations. While with Fe atom at a cubic-like Si
268 site there is no atomic relaxation for the non magnetic state (NM), in the high spin magnetic
269 state (M), the equilibrium distance between Fe atom and C atom above it in the tetrahedron is
270 significantly increased. Fe atom is staying practically in place and this C atom is moving
271 about 0.01 nm away along the **c**-axis of the hexagonal SiC crystal from the unrelaxed Fe-C
272 distance of ~ 0.19 nm (= Si-C distance in SiC) for a strain value of $\sim 5\%$. The NM state
273 becomes the lower energy state for a strain less than 2.5% as shown on Fig. 3 of [15]. In the

274 M state, such value of strain corresponds to the surface normal strain measured by XRD along
 275 the *c*-axis of the hexagonal SiC crystal (3-4%; see Table 1 and Supplementary Material).

276 On the other hand, in a recent theoretical work on the origin of ferromagnetism in III-
 277 V DMS, Bouzerar *et al* [8] combine several theoretical approaches to explain the magnetic
 278 properties of such systems, and in particular on 2.5-5 at.%Mn-implanted InP [9]. It is shown
 279 that the “highly unconventional non-Brillouin-function-like character” of the *M*(*T*) curve for
 280 this system is strongly related to “the extreme sensitivity of the position of the Mn acceptor
 281 level”. Moreover, “spin-resolved density of states (DOS) in (In,Mn)P itinerant carriers are
 282 always fully polarized and the Fermi level lies in a well defined impurity band not totally
 283 separated from the valence band” [8]. The electronic structure of 2.5-5 at.%Mn-implanted InP
 284 and of 4 at.%Fe-doped hexagonal SiC are thus very similar, allowing analysis of our
 285 experimental results in the framework of Refs [8] and [15].

286 For comparison, the ordinate axis of calculated curves of [8] were normalized to that
 287 of the experimental ones of [9]. As shown in Fig. 6-a the value $T_C \sim 40$ K for 5 at.%Mn-
 288 implanted InP reported in [9] and calculated in [8] corresponds to about the quarter of the
 289 maximum value of the experimental *M*(*T*) curves and of the normalized calculated ones at
 290 low temperature (large arrows on Fig. 6-a).

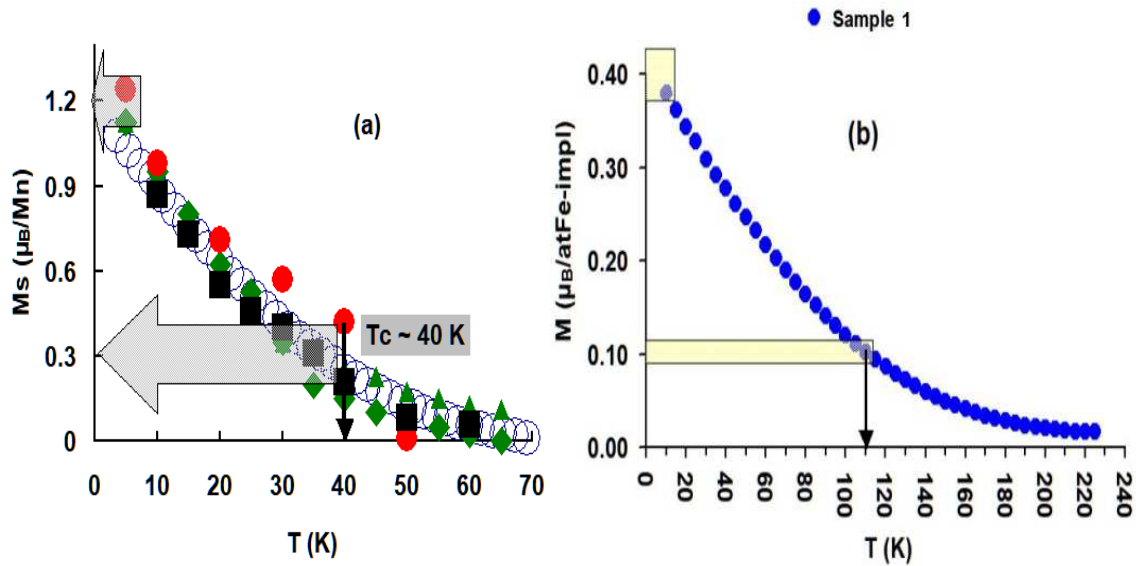
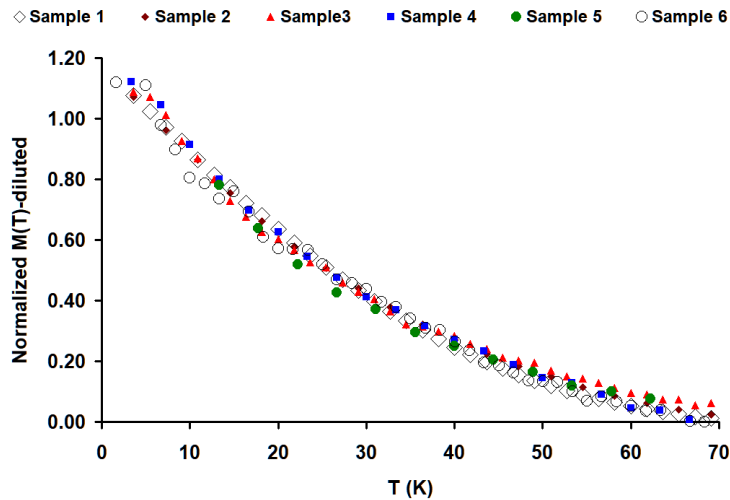


Fig. 6: (a): 5 at.%Mn-implanted InP saturation magnetization $M_s(\mu_B/Mn)$ as a function of temperature: SQUID (full circles) and XMCD signal (squares) [9]; normalized calculations MC (triangles) and LRPA (rhombus) [8]; normalized *M*(*T*) curve of sample 1 (empty circles). (b): experimental saturation magnetization *M*(*T*) curve of sample 1.

291

292 With this criterion, we can evaluate the value $T_C \sim 110$ K for sample 1 (Fig. 6-b).
 293 Normalizing the experimental $M(T)$ curve of sample 1 with: $Mx1.2/0.4$ and $Tx40/110$, we can
 294 see that the normalized curve of this sample exactly overlaps the experimental curves of 5
 295 at.%Mn-implanted InP and the normalized calculated ones (Fig. 6-a). This confirms that
 296 (In,Mn)P and Fe:SiC have similar electronic structure and magnetic behavior. We conclude
 297 that sample 1 has a DMS behavior with $T_C \sim 110$ K.

298 The $M(T)$ -diluted curves for samples with nanoparticles (Fig. 5-b) can then be
 299 analyzed as explained above for sample 1. Thus, normalizing these curves with $Mx1.2/M(0)$ -
 300 diluted and $Tx40/T_C$ -diluted with the set of parameters given in Table 2, as we have done
 301 above for sample 1, all the normalized curves remarkably overlap the normalized curve of
 302 sample 1 as shown in Fig. 7. This shows that the magnetic behavior of diluted Fe atoms in
 303 Fe:SiC can be described and explained, as for the III-V system (In,Mn)P, in the framework of
 304 the theoretical work of [8], independently of the presence of Fe-rich nanoparticles. We are
 305 thus able to evaluate the mean value of the magnetic moment of the diluted Fe atoms ($M(0)$ -
 306 diluted) and their Curie temperature (T_C -diluted) as reported in Table 2.



307
 308 Fig. 7: Normalized $M(T)$ -diluted curves as explained in the text.

309
 310 Only the diamagnetic component of the substrate has been taken into account in
 311 previous work [29] and then, as mentioned above, a strong paramagnetic component appears
 312 on the $M(T)$ curve of sample 6. In the present work, subtracting the paramagnetic contribution
 313 of the substrate from magnetometry measurements leads to the normalized $M(T)$ -diluted
 314 curve of Fig. 7 together with the set of parameters reported in Table 2 for this sample.

315

316 II-2) Discussion on Curie Temperature

317 The values of T_C -nanop reported on Table 2 are in the range of the FM compounds present in
318 the Fe-rich nanoparticles evidenced by APT in the studied samples [24,27]: $T_C(\text{Fe}_5\text{Si}) \sim 385$
319 K; $T_C(\text{Fe}_2\text{Si}) \sim 550$ K; $T_C(\text{Fe}_3\text{Si}) \sim 840$ K. The mean value of T_C -diluted is $\sim 115(+/-5)$ K in
320 all samples independently of the amorphous or crystalline state of the SiC matrix and of the
321 presence of small (diam < 10 nm) superparamagnetic Fe-rich nanoparticles, except for sample
322 5 with a few big (diam ~ 9 -12 nm) strongly FM Fe_3Si nanoparticles. In that case, T_C -diluted is
323 strongly reduced to 45 K, indicating a strong influence of strongly FM big Fe_3Si nanoparticles
324 on the FM behavior of diluted Fe atoms. It can be mentioned that the sample 6 ($C_{\text{atFe}} \sim 2\%$)
325 annealed at the same temperature (1573 K) as sample 5 ($C_{\text{atFe}} \sim 6\%$) exhibits only small
326 (diam < 8 nm) superparamagnetic Fe-rich nanoparticles [27] and T_C -diluted ~ 120 K (Table
327 2), confirming the strong influence of strongly FM big Fe-rich nanoparticles on the magnetic
328 behavior of diluted Fe atoms in Fe:SiC.

329 II-3) Discussion on TM magnetic moment

330 The mean value of the magnetic moment of Fe atoms in Fe-rich nanoparticles can be
331 evaluated as: $M(0)\text{-Fe-nanop} = M_0\text{-nanop} / \text{Fe-nanop}$ as reported in Table 3 where results
332 obtained from other techniques are also given (Fe-nanop and Fe_s , see Table 1). The median
333 value of $M(0)\text{-Fe-nanop}$ on the five samples containing nanoparticles (samples 2 to 6) is ~ 2.1
334 μ_B , as currently observed for Fe atoms in the high spin state in well defined solid FM
335 compounds mainly detected by APT in the core of the Fe-rich nanoparticles. In a previous
336 work [27], we have estimated *a priori* the mean value of the magnetic moment of Fe atoms in
337 Fe-rich nanoparticles from its mean value in these solid FM compounds. The value of the
338 magnetic moment ($\sim 1.2 \mu_B$) [27] appears now to be underestimated and therefore
339 overestimated values of the magnetic moment of diluted Fe atoms were obtained. In the
340 present work, modeling the contribution of the Fe-rich nanoparticles to the saturation
341 magnetization with equation (1) and taking into account the proportion of Fe-implanted atoms
342 in the Fe-rich nanoparticles obtained from APT (Fe-nanop) get a direct access to the mean
343 value of the magnetic moment of Fe atoms in Fe-rich nanoparticles ($M(0)\text{-Fe-nanop}$) with no
344 hypothesis.

345 Table 3: Magnetic moment of Fe atoms

SAMPLE	M(0)-nanop (μ_B /atFe-impl)	Fe-nanop	M(0)-Fe-nanop (μ_B)	M(0)-diluted (μ_B /atFe-impl)	Fe _s	M(0)-substit (μ_B)
Sample 1	-	-	-	0.40	-	-
Sample 2	0.5	27	1.85	0.275	53	0.52
Sample 3	0.89	44	2.02	0.10	43	0.23
Sample 4	1.055	-	-	0.11	38	0.29
Sample 5	1.535	66	2.33	0.07	24	0.29
Sample 6	1.285	60	2.14	0.14	35	0.40

346

347 Now, taking into account CEMS results, we can also calculate the mean value of the
348 magnetic moment of diluted Fe atoms at the Si sites in the SiC matrix as: $M(0)\text{-substit} =$
349 $M(0)\text{-diluted} / Fe_s$. As shown on Table 3 the median value of $M(0)\text{-substit}$ is $\sim 0.375 \mu_B$.
350 Now, we have to remember that there are crystallographic inequivalent Si (and C) sites when
351 the first- and second-nearest neighbours are considered in 6H(4H)-SiC: two (6H-SiC) or one
352 (4H-SiC) with cubic-like symmetry for one with hexagonal-like symmetry [41]. Thus, the
353 mean probability of Fe substitution at an hexagonal-like Si site, which is the only one likely to
354 be magnetic as mentioned above [15], is between $1/3$ and $1/2$, and thus the mean value of the
355 magnetic moment of a diluted Fe atom at a hexagonal-like Si site becomes $\sim 0.375 \times (2 \text{ or } 3$
356 $(\sim 2.5)) \sim 0.94 \mu_B$. In addition, Los *et al* have shown that for such a Fe atom in hexagonal SiC,
357 the energy separation between NM and M is very low (20 meV in 4H-SiC [15]) and M can be
358 FM or antiferromagnetic (AFM) [15-16]. Thus, at a non-zero temperature a diluted Fe atom in
359 a hexagonal-like Si site may be NM, FM or AFM with nearly equal probability ($\sim 1/3$) [15].
360 The mean value of the magnetic moment of a FM diluted Fe atom is then $\sim 0.94 \times 3 \sim 2.8 \mu_B$
361 in very good agreement with the calculated value for such a Fe atom in 2 at%Fe-doped 6H-
362 SiC ($2.76 \mu_B$) [12]. In addition, as shown in [15], depending on the strain state of the
363 crystalline SiC matrix as observed by XRD, the value of the magnetic moment of a FM
364 diluted Fe atom may fluctuate between 2.95 and $3.1 \mu_B$ in 4H-SiC. Such considerations may
365 explain the factor of 2 between the values of experimental magnetization ($\sim 1.2 \mu_B/\text{Mn}$) [9]
366 and the estimated ones ($2.4 \mu_B/\text{Mn}$) [8,9] in (In,Mn)P as suggested by Los *et al* in [16] for
367 GaN-based DMS materials.

368 Furthermore, it can be noted that the mean value of the magnetic moment of Fe atoms
369 in the amorphous sample 1 ($M(0)\text{-diluted} \sim 0.4 \mu_B$) is almost the same as the median value of
370 $M(0)\text{-substit}$ ($\sim 0.375 \mu_B$) in crystalline 6H-SiC matrix. We might imagine that each TM atom

371 in the amorphous SiC is located in elemental tetrahedron at a site like a hexagonal Si site in
372 crystalline SiC matrix. In that case $F_{es} = 100\%$ for sample 1 and $M(0)$ -substit = $M(0)$ -diluted
373 ($\sim 0.4 \mu_B$). This might be suggested by the fact that at the first steps of annealing of an
374 amorphous SiC sample, tiny crystallites of different polytypes are observed [46]. In
375 amorphous SiC, the long range order is broken but short range order may exist and then the
376 discussion of the above paragraph holds and leads to a mean value of the magnetic moment of
377 a FM diluted Fe atom in sample 1 as: $0.4 \times 2.5 \times 3 \sim 3 \mu_B$. We can thus conclude that the
378 mean value of the magnetic moment of a FM diluted Fe atom in SiC is ~ 2.8 - $3.1 \mu_B$
379 independently of the amorphous or crystalline state of the SiC matrix.

380 **III) Discussion on some typical experimental results**

381 Ferromagnetism with a T_C as high as 250 K has been claimed [18] for Mn- and Fe-
382 implanted 6H-SiC. It can be noted that the temperature dependence of the difference between
383 FC and ZFC magnetization for 5 at.%Fe-implanted 6H-SiC at 623 K and annealed at 973 K
384 (with crystalline SiC matrix and no detected secondary phase) shown in Fig. 3 of [18] has the
385 same behavior as shown in Fig. 6. Following the same normalization procedure as explained
386 above, it appears that we may determine $T_C \sim 100$ - 150 K. Experimental results of [18] for the
387 5 at.%Fe-implanted sample: $M(\text{saturation}) \sim 0.6 \mu_B/\text{atFe}$ and $H_C \sim 5$ mT at 10 K (Fig. 2 of
388 [18]) and $T_C \sim (100$ - $150)$ K are coherent with those observed in this work. The same behavior
389 is roughly seen on Fig. 4 of [18] for the 5 at.%Mn-implanted sample, with $T_C \sim (200$ K) and
390 $H_C(10$ K) ~ 15 mT and about half the magnetization of 5 at.%Fe-implanted sample (Fig.3 of
391 [18]). The discussion of the above paragraph leads to a mean value of the magnetic moment
392 of a FM diluted Mn atom in 5 at.%Mn-implanted SiC as: $(0.6 / 2) \times 2.5 \times 3 \sim 2.25 \mu_B$ in very
393 good agreement with the calculated value for such a Mn atom in 2 at.%Mn-doped 6H-SiC
394 ($2.17 \mu_B$) [12].

395 Ferromagnetism above RT has also been claimed [47] for 7-10 at.%Cr-doped
396 amorphous SiC films grown on Al_2O_3 substrate with an average magnetic moment in the
397 range 0.17 - $0.47 \mu_B$ and $H_C \sim 3$ - 15 mT between 300 K and 5 K (Fig. 5-6 of [47]). Taking into
398 account the above discussion, we may roughly estimate a mean value of the magnetic moment
399 of FM Cr in SiC as: $((0.17+0.42)/2 = 0.32) \times 2.5 \times 3 \sim 2.4 \mu_B$. This value is higher to the
400 values reported for spin moment of a Cr ion in tetrahedral coordination when doped on the
401 Si site ($2.0 \mu_B$) [47], as well as for Cr atom in 2 at.%Cr-doped 6H-SiC ($1.5 \mu_B$) [12]. In the
402 corresponding work, there is no correction of the paramagnetic contribution to the SQUID

403 signal, thus the experimentally estimated values of saturation magnetization are higher than
404 the actual ones. Nevertheless, these values and those of H_C are consistent with those observed
405 for Fe and Mn and we suppose that results described in [47] may be understood in the
406 framework of the theoretical models [8] and [15] as proposed in this work.

407 From this study and particular examples of refs. [18] and [47], it appears that the FM
408 behavior of diluted TM atoms in SiC does not depend on the crystalline state of the SiC
409 matrix nor on the nature and concentration of the TM dopant in the range of 2-13 at.%, but
410 more on the local relaxation of the SiC lattice due to the substitution of a TM atom at the Si
411 lattice site. The local relaxation may depend on the nature of the TM atom and explain the
412 little differences observed on the values of H_C and T_C and between the calculated and
413 experimentally estimated values of magnetic moments. Moreover, it has been suggested that
414 uniaxial strain as observed in this work can efficiently control the value of T_C resulting from
415 the hole-mediated interaction between magnetic ions in DMS [48]. It would be therefore
416 interesting to perform transport measurements on the Fe-implanted 6H-SiC samples presented
417 in this work in order to study the coupling between magnetic Fe atoms and itinerant carriers.
418 Work is in progress on this topic by our team.

419 **Conclusion**

420 In this paper, we have reviewed some experimental and theoretical results about Fe-(and Mn-
421 Cr-) implanted SiC. We have shown that Fe:SiC has similar electronic structure and magnetic
422 behavior to (In,Mn)P. Combining many selected experimental techniques and properly
423 treating magnetometry results together with the help of new theoretical approaches adapted to
424 the studied system allow a direct access to:

- 425 1- The mean value of the magnetic moment of Fe atoms in magnetic Fe-rich
426 nanoparticles ($\sim 2.1 \mu_B$) currently observed for Fe atoms in the high spin state in well
427 defined solid FM compounds mainly detected in the core of the Fe-rich nanoparticles.
- 428 2- The mean value of the magnetic moment of FM diluted Fe atoms ($\sim 2.8 \mu_B$) ($2.2 \mu_B$
429 for Mn) in very good agreement with the calculated value for such TM atoms in
430 hexagonal SiC. This value together with the temperature dependence of their
431 magnetization strongly agree with recent theoretical works on such DMS systems
432 taking into account both the carrier-mediated ferromagnetism and the correct location
433 of the transition metal dopant binding energy in the host semiconductor, allowing us to

434 conclude that FM diluted Fe atoms in 2-13 at.%Fe-implanted SiC have a DMS
435 behavior.

436 The magnetic moment of FM diluted Fe atoms in SiC appears due to local strain
437 relaxation around the implanted species. It is also independent of the amorphous or crystalline
438 state of the SiC matrix and of the presence of small (diameter < 10 nm) superparamagnetic
439 Fe-rich nanoparticles. However, the value of their T_C is strongly dependent on the presence of
440 larger (diameter > 10 nm) FM Fe-rich nanoparticles, decreasing from ~ 115 K to 45 K in
441 presence of Fe_3Si nanoparticles. This experimental work may contribute to a better
442 understanding of the complex topic of the effects of nanometric magnetic inhomogeneities of
443 diluted magnetic systems.

444

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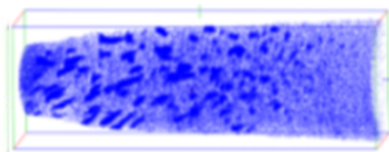
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Fe-doped SiC

● Fe atoms



$41.4 \times 41.5 \times 132.5 \text{ nm}^3$

$M (\mu_B/\text{Fe at.})$

