Local structural, magnetic and magneto-optical properties of Mn-doped SiC films prepared on a 3C–SiC(001) wafer

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Abstract. We report on a systematic study of local structural, magnetic and magneto-optical properties of Mn-doped SiC films synthesized on a 3C–SiC(001) homoepitaxial wafer by an annealing method. A thin Mn layer was deposited on the SiC wafer, and then annealing was performed to diffuse the Mn atoms into the SiC epitaxial layer. Transmission magnetic circular dichroism and magnetization investigations demonstrated ferromagnetic behavior up to 300 K, although the x-ray diffraction studies indicated that the domain phase of the synthesized layer was antiferromagnetic $\alpha$-Mn. A detailed extended x-ray-absorption fine structure measurement further revealed that the synthesized sample, in fact, comprises two uncoupled phases: one is the top antiferromagnetic $\alpha$-Mn layer and the other one is Mn atoms, which may randomly substitute the Si and C atoms in the 3C–SiC lattice. Combining these experimental results with recent theoretical calculations, we strongly propose that the observed room-temperature ferromagnetism was of intrinsic origin, arising from the ‘embedded Mn atoms’ in the 3C–SiC lattice, instead of other magnetic clusters.

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1. Introduction

The search for spintronics materials that combine semiconducting and ferromagnetic properties, namely, diluted magnetic semiconductors (DMSs), is currently the most topical field in magnetism [1]. Since the discovery of a ferromagnetic order in Mn-doped InAs by Ohno et al [2], many other DMSs have been discovered, mostly III–V and II–VI types [3, 4]. Among these materials reported so far, Mn-doped GaAs has been found to be ferromagnetic with highest Curie temperature, \( T_c \sim 172 \text{ K} \). However, for practical spintronics applications of ferromagnetic DMSs, intensive research to realize room-temperature ferromagnetism must be conducted in areas such as improving deposition techniques and exploring new host materials, etc. Toward to this end, considerable effort has been made and several materials have been reported to exhibit ferromagnetism above room temperature [5]–[8], but few of them have been confirmed to intrinsic nature of ferromagnetism. In the case of the IV-group semiconductors, Si, Ge etc, as host materials of DMSs, results in this context are still relatively limited in spite of the fact that the emerging field of spintronics would be dramatically boosted if room-temperature ferromagnetism could be added to the IV-group semiconductors that are compatible with current semiconductor technologies [9]–[11].

Among the IV-group semiconductors, silicon carbide (SiC) is a promising material for power electronics due to its useful fundamental properties such as wide band gap, high breakdown electric field and high thermal conductivity [12]. In particular, cubic SiC (3C–SiC) is the most promising material for active devices because of its low work function and the highest electron mobility among the various polytypes of SiC. Nevertheless, very little attention has been paid to potential ferromagnetic DMS behavior in SiC. Only recently, several groups [13]–[17], including ours [18]–[20], have explored SiC-based DMSs doped with Ni, Fe or Mn, raising optimism about the possibility of the intrinsic nature of room-temperature ferromagnetism therein. It is interesting to note that the Mn-doped SiC-based DMSs were theoretically predicted to have half-metallicity, stable high \( T_c \) ferromagnetism and relatively large magnetic moments [16]. These characteristics are desirable for realizing spintronics devices. In our earlier studies, we have synthesized the Mn-doped SiC films on the hexagonal polytypes of 4H–SiC(0001) wafer by using an annealing method [18]. It has been revealed that the synthesized Mn-doped SiC showed a \( T_c \) of 300 K, although the x-ray diffraction (XRD) and selected-area diffraction (SAD) measurements suggested that paramagnetic tetragonal Mn$_5$Si$_2$ compound was a dominant phase. As the origin of the ferromagnetism, we suggested that a small amount of carbon incorporated into the paramagnetic Mn$_5$Si$_2$ host induced ferromagnetic order in Mn$_5$Si$_2$. Moreover, we found that the Mn atoms in the interlayer are incorporated solely...
on the interstitial site in the hexagonal 4H–SiC lattice rather than substitute Si- or C-site, thus showing superparamagnetic behavior [19].

It is well known that 3C–SiC is the only cubic (zincblende) structure among the various polytypes of SiC. Compared with the hexagonal polytypes of SiC (for example, 4H–SiC and 6H–SiC), 3C–SiC has a relatively small interstitial site from a crystallographical point of view, affording the exciting possibility of achieving a SiC-based high-$T_c$ ferromagnetic DMS with transition metal doping. For this propose, we have used the 3C–SiC wafer as the host material. In our recent study, for the first time, Mn-doped SiC films with a $T_c$ at $\sim$245 K were successfully synthesized on a cubic 3C–SiC homo-epitaxial wafer using an ion-implantation technique [20]. As a second approach, in this study, we synthesized a Mn-doped SiC film showing room-temperature ferromagnetism on the cubic 3C–SiC wafer using an annealing method. Particular attention was paid to its structural, magnetic and magneto-optical properties. The magnetic and magneto-optical results in combination with a detailed local structural analysis strongly suggested that the observed ferromagnetism in our sample was of intrinsic origin, arising from the ‘embedded Mn atoms’ in the 3C–SiC lattice, instead of other magnetic clusters.

2. Experimental

3C–SiC(001) substrates having a 5 $\mu$m SiC homo-epitaxial layer were used for this study. This epi-layer was grown by HOYA Company. For the homo-epitaxial layer, the conduction type is $n$-type and the carrier density is $1 \times 10^{16}$ cm$^{-3}$. The typical size of the substrates is $10 \times 10$ mm$^2$. Growth of Mn was carried out in an ultrahigh vacuum (UHV) multichamber molecular-beam epitaxy system (Eiko Engineering Ltd). Before Mn growth, the 3C–SiC substrate was thermally cleaned at 1000 $^\circ$C for 10 min to remove the thin oxide layer on the surface. Then a thin Mn layer of thickness 50 nm was deposited on the clean 3C–SiC substrates using a Knudsen cell in the growth chamber. During the deposition, the substrate temperature was kept at 300 $^\circ$C, which is the optimum temperature for Mn growth on 4H–SiC(0001) [21]. The Mn cell temperature was kept at 800 $^\circ$C during growth. The growth rate was set at 20 nm h$^{-1}$. After Mn deposition, in situ annealing was performed at 1000 $^\circ$C for 3 min to diffuse the Mn atoms into the 3C–SiC homo-epitaxial layer.

The structural properties of the sample were investigated by ex situ XRD with Cu K$_{\alpha}$ radiation, where out-of-plane ($2\theta$/$\theta$ scan) and in-plane ($2\theta\chi$/$\theta$ scan) diffraction measurements were performed to detect diffraction signals from lattice planes perpendicular and parallel to the sample surfaces, respectively. The magnetization measurements up to 380 K were carried out in a Quantum Design superconducting quantum interference device (SQUID) magnetometer. All data presented here were corrected for the diamagnetic background of the substrate according to the following procedure: we measured the magnetization of the respective sample up to the highest magnetic field available (5 T). At this high field, the diamagnetic contribution from SiC dominates the signal. A linear fit yields the slope of the signal at high fields, which in all cases was virtually identical to that of bare 3C–SiC substrates. A straight line with the slope determined from the fit was then subtracted from the raw data. Prior to measuring the temperature dependence of the magnetization, the sample was first cooled from room temperature to 2 K either under a field (field cooled (FC)) or at zero field (zero field cooled (ZFC)). In the case of ZFC measurements, the sample is demagnetized under a oscillatory magnetic field at room temperature before cooling it down to 2 K. The transmission magnetic circular dichroism (MCD) measurement was performed to investigate the magneto-optical
properties. For this study, a magnetic field up to 1 T was applied perpendicular to the sample surface.

The information about the local atomic order around the Mn atoms was provided by extended x-ray absorption fine structure (EXAFS) measurements. The EXAFS technique is suited for probing the local environment surrounding specific atoms in a complex materials system, and as confirmed by previous experiments, Mn substitution for Ga in GaAs host is believed to be responsible for the observed ferromagnetism. The EXAFS measurements were performed at beam line BL12C at the Photon Factory in Tsukuba with a Si (111) double crystal monochromator and a bent cylindrical mirror using synchrotron radiation from the 2.5 GeV storage ring. The EXAFS spectra were measured in the fluorescence-detection mode. Intensity of incident x-ray beam was monitored by a nitrogen-filled ionization chamber, while the x-ray fluorescence signal was detected by an array of 19 elements of Ge solid state detectors. All the EXAFS measurements were performed at 70 K in order to reduce thermal vibration.

3. Results and discussion

The sample was first subjected to an extensive investigation by high-resolution XRD in a wide angular range. Figure 1(a) shows the typical XRD patterns in out-of-plane (θ–2θ) geometry for the 3C–SiC(001) wafer and the synthesized sample, respectively. These two diffraction patterns well coincide other than a very weak diffraction peak around 2θ = 62.46°, which corresponds to bcc α-Mn (006). Moreover, the out-of-plane parameter of the bcc α-Mn film deduced from the XRD analysis is 8.92 Å, which is in good agreement with the bulk value of 8.91 Å [22]. It means that a phase-pure α-Mn film with c-axis parallel to the film surface is obtained. In figure 1(b), we show an in-plane (φ–2θχ scan) XRD pattern for the same sample. There is only a very weak diffraction peak can be found at 2θ = 62.46°, which can be reasonably ascribed to bcc α-Mn (600). We can therefore conclude that the bcc α-Mn is the dominant phase in the synthesized sample, and any traces of other phases were not found within the detection limit of XRD.

To quantify the in-plane distribution of α-Mn crystals, another type of in-plane scan, referred to as a rocking scan, was conducted on the same sample, where the detector was fixed at θ = 31.23° (corresponding to (006) reflection) and only the sample was rotated around the axis. The result is displayed in the left inset of figure 1(b). We found that the typical full-width at half-maximum (FWHM) of the x-ray rocking curve for the (006) reflection is 0.51. The peak broadening may be explained in terms of lattice relaxation of α-Mn due to carbon incorporation.

Figure 2 shows the magnetization loops obtained for the synthesized sample at different temperatures. The magnetic field was applied perpendicular to the sample plane. At all temperatures, the magnetization saturates at high magnetic fields and exhibits a hysteresis at lower fields. These two features indicate ferromagnetic behavior. When examined closely it is found that the shapes of the hysteresis loops display a dual-loop characteristic at temperatures lower than 300 K. This is a clear indication of the fact that there is another ferromagnetic and/or superparamagnetic signal superimposed on the ferromagnetic signal. This conclusion is further supported by the upper-left inset of figure 2, which shows the saturation magnetization (Ms) as a function of temperature. It is clear from this plot that Ms falls sharply at low temperatures and then decreases slowly as the temperature is increased. A change of slope at a temperature of ~50 K is clearly visible. Finally, at 300 K, Ms drops eventually to zero, indicating the Curie point (Tc) of the ferromagnetic magnetic component to be around that temperature.

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Figure 1. XRD patterns of (a) out-of-plane ($\theta$–$2\theta$) scan for 3C–SiC (001) wafer and synthesized sample; (b) the in-plane ($\phi$–$2\theta\chi$ scan) for synthesized sample. The right inset in (b) shows the scattering geometry, and the left inset in (b) shows an x-ray rocking curve of the sample, (0 0 6)-Bragg reflection.

Figure 3 shows the FC and ZFC magnetization curves. The plot shows an interesting tendency, which is different from that for the usual superparamagnetic materials, where the FC and ZFC bifurcate at the blocking temperature. The plots here can be understood, if we consider the sample as a mixture of ferromagnetic and superparamagnetic materials with a transition temperature around 300 K. The FC and ZFC curves remain separated throughout the entire temperature range from 2 to 300 while they coincide at around 300 K. The separation between the FC and ZFC curves indicates a hysteretic behavior, which is consistent with our observation shown in figure 2. Additionally, the origin of the kink at $\sim$50 K for the FC curve is not yet clear. As we mentioned above, it seems that a second phase with a transition temperature of $\sim$50 K is additionally present in the system since a steep step around that temperature is found, when we measured $M_s$ as a function of temperature (see inset figure 2(b)). A similar second steep increase
Figure 2. Magnetization hysteresis loops of synthesized sample measured at various temperatures. The inset shows the temperature dependence of the saturation magnetization ($M_s$).

Figure 3. Temperature dependence of ZFC and FC magnetization under a magnetic field of 100 Oe. The magnetic fields were applied parallel to the sample plane.

of $M_s$ at low temperatures in combination with the appearance of a concave shoulder in FC curve has been recently found in Mn-doped Ge films [5] and is considered to be characteristic for a DMS based on the model of bound magnetic polarons (BMPs) [23]. However, for a more profound assignment to BMPs, a detailed analysis including ac susceptibility measurements would be required, which lies beyond the scope of this paper.

New Journal of Physics 10 (2008) 055006 (http://www.njp.org/)
From the foregoing, it is clear that the hysteresis loops have been observed in SQUID measurement for the synthesized sample. The SQUID measurement probes the average magnetic properties of a sample with a size of about $5 \times 5 \text{mm}^2$ and is sensitive to detect small amounts of ferromagnetic secondary phase or precipitations. However, this technique lacks the spatial and energy resolutions to discriminate these spurious phases which may confuse the data interpretation. For example, if the possible magnetic clusters or nanoparticles have a similar $T_c$ to that of the expected DMS phase, standard temperature-dependent magnetometry is not useful for addressing this issue. Compared to SQUID, the MCD technique is not only able to detect the presence of different phases, but also shows photon energy selectivity. This is based on the fact that the MCD signal is proportional to $dk/dE$, where $k$ is the absorption coefficient and $E$ is the photon energy \[24\]. Photon energies at which optical transition occurs will be accompanied by a strong MCD response. Therefore, the MCD measurements were performed in order to distinguish the magnetic properties which are intrinsic in the pure diluted phase of Mn-doped SiC from those of possible precipitates of other compounds. Figure 4(a) shows the schematic illustration of the transmission MCD measurements set-up in this work. The magnetic field up to 1 T was applied perpendicular to the sample plane. The MCD spectra of the 3C–SiC wafer and synthesized sample at 300 and 100 K are compared in figure 4(b). For the 3C–SiC wafer, no noticeable structure in the vicinity of the band gap of 3C–SiC ($E_g = 2.39 \text{ eV}$ at 300 K) was observed at 300 and 100 K. For the synthesized sample, the MCD spectrum at 300 K roughly corresponded with that of the 3C–SiC wafer. This is considered to be a reasonable result since the $T_c$ of the synthesized sample is 300 K. On the other hand, the MCD spectrum of
the synthesized sample at 100 K showed a positive large intensity in all photon energy ranges. The MCD signal is directly related to the Zeeman splitting of the band structure caused by the s, p–d exchange interaction of DMSs [24]. Its intensity linearly depends on the Zeeman splitting energy that is proportional to the magnetization. Thus, the magnetization process of a ferromagnetic DMS can be obtained from the magnetic field dependence of the MCD intensity. In figure 4(c), we show the magnetic field dependence of the MCD intensity at different photon energies measured at 100 K. Clear hysteresis loops showing uniform shape were obtained at the respective photon energy, and the loops were found to be independent of the photon energy. If the sample contains magneto-optically active precipitates, the shape of the magnetic field dependence of MCD loop should change with photon energy. The MCD loops measured at any photon energy can be superposed upon a single loop, indicating the observed MCD spectra come from a single material.

Since the MCD intensity is proportional to the magnetization, the magnetic field dependence of the MCD intensity should be in good agreement with the magnetic field dependence of magnetization (\(M–H\) curve). Thus, in figure 4(c), we also show the normalized \(M–H\) curve measured by using the SQUID at 100 K. As expected, the MCD loops coincide well with the \(M–H\) curve, although there is only a slight difference in the coercive field.

The origin of ferromagnetism in transition-metal-doped semiconductors has been controversial due to the possible presence of ferromagnetic transition metal, metal oxide particles and/or metal-related compounds [25]. However, the possibility that the ferromagnetism originates from a Mn-related secondary phase formed by precipitation during the sample preparation can be disregarded for several reasons as follows. Firstly, within the detection limit of the performed structural investigations in this study, no phase other than metallic \(\alpha\)-Mn was detected in the synthesized sample. Secondly, the metallic \(\alpha\)-Mn and most Mn oxides are not ferromagnetic. Thirdly, no ‘pure’ Mn–Si related compounds having a Curie temperature higher than 50 K are known to exist [26]. Finally, it is important to point out that carbon-doped non-ferromagnetic Mn–Si compounds such as Mn5Si3 [27] and Mn5Si2 [18] have been recently found to show a ferromagnetism with high \(T_c\). In the present study, however, we can also rule out this scenario for the following reasons: (i) we did not detect any trace of Mn–Si compounds by both XRD and EXAFS measurements as will be shown below; (ii) even if one assumes the formation of nanosized carbon-doped Mn-Si clusters, which are beyond the detection limit for both techniques above mentioned, one can only expect either paramagnetic or superparamagnetic behavior depending on cluster size; (iii) in fact, we did not observe the existence of such Mn-Si clusters by high resolution transmission electron microscopy and (iv) for simplicity, we have removed the top metallic layer by chemical etching. In figure 5(a), we show the magnetization loop obtained at 2 K for the sample after etching. The observed clear hysteresis loop indicates that the etched sample still shows ferromagnetic behavior. Figure 5(b) shows the temperature dependence of the magnetization (\(M–T\)) in the ZFC process with an applied field of 0.01 T. The \(T_c\) of the etched sample was evaluated to be around 300 K. Based on this simple decomposition and previous structure investigations, we suggest that there are two uncoupled phases in our sample, one is the top Mn layer, the other one is the unknown ferromagnetic phase, which displays a \(T_c\) around 300 K. At this stage, we propose that the formation of a Mn-doped SiC within the 3C–SiC lattice can be considered as the most likely origin of the observed room-temperature ferromagnetism.

Within the above scenario, an interesting question arises as to where the Mn atoms can reside in the 3C–SiC lattice. To address this issue, we have performed fluorescence EXAFS
measurements, exploiting the relationship between the local structures around Mn atoms and the magnetic properties. Figure 6(a) shows the experimental Mn K-edge EXAFS oscillation functions $k^2 \chi(k)$ spectra of the synthesized sample and a pure $\alpha$-Mn foil. The latter serves here as a reference. The experimental spectrum of the synthesized sample shows similar behavior to the $\alpha$-Mn foil. This coincidence corroborates that the dominant phase is $\alpha$-Mn in the sample, which is indeed consistent with our previous XRD results. Figure 6(b) shows the theoretical Mn K-edge EXAFS oscillation functions $k^2 \chi(k)$ spectra for the substitutional Mn atom on the C- and Si-sites in the 3C–SiC lattice. At first glance, the EXAFS spectrum of the synthesized sample is quite different from the two theoretical spectra. Generally, the Fourier transform (FT) of a EXAFS spectrum allows a separation of the different configuration shells versus radial distance from the absorbing atom, thus providing a direct way to investigate the local structures around Mn atoms in the 3C–SiC lattice. In figure 7(a), we show the FT Mn K-edge EXAFS spectra for the synthesized sample and the pure $\alpha$-Mn foil. Unlike the pure $\alpha$-Mn that exhibits a single peak in the radial distance range of 2.2 Å, we found that the synthesized sample shows multi-peak character in the radial distance range of 1.0–3.0 Å. A main peak due to the first coordination shell around the Mn atoms was observed in the radial distance range of $\sim$2.2 Å. The main peak position is consistent with that of the pure $\alpha$-Mn foil. In addition, two shoulder peaks were also observed in the radial distance range of $\sim$1.4 and 2.6 Å, which are consistent with the main peak of the theoretical FT Mn K-edge EXAFS spectrum for the substitutional Mn atom on the C- and Si-sites, respectively, in the 3C–SiC lattice as shown in figure 7(b). As a result of the simple decomposition, we can draw a conclusion as follows: apart from metallic $\alpha$-Mn formed on the surface of 3C–SiC, there are some Mn atoms that may randomly substitute the Si and C atoms within the 3C–SiC lattice. Nevertheless, we should mention that a quantitative analysis based on the curve fitting of the experimental EXAFS data would be useful to further support our argument, and is now in progress.

Thus, it is clear that, unlike previous Mn-doped 4H–SiC systems that indicated the majority Mn atoms are incorporated on the interstitial site in the 4H–SiC lattice [19], we proposed that most of Mn atoms may randomly incorporate on the Si- and C-sites in the 3C–SiC lattice. Qualitatively, this is in line with a recent theoretical model in which ferromagnetic behavior is found in the case of Mn atoms substituting for the Si atoms in the 3C–SiC lattice [16].

Figure 5. Magnetic properties of the sample after etching: (a) Magnetic hysteresis loop measured at 2 K. The inset shows the loop at small magnetic fields. (b) Temperature dependence of ZFC magnetization under a magnetic field of 100 Oe. The magnetic fields were applied parallel to the sample plane.
Figure 6. (a) Experimental Mn K-edge EXAFS oscillation functions $k^2 \chi(k)$ spectra for the synthesized sample and Mn foil, respectively; (b) theoretical Mn K-edge EXAFS oscillation functions $k^2 \chi(k)$ spectra for the substitutional Mn atom on the C- and Si-sites in the 3C–SiC lattice. The Debye–Waller factor was assumed as 0.075 Å for a bond length below 4.0 and 0.100 Å for a bond length above 4.0 Å. The theoretical EXAFS spectrum was generated by FEFF8 [28].

Since the origin of ferromagnetism in Mn-doped SiC compounds is considered to be different from the carrier induced mechanism in (III, Mn)As [29], another explanation is needed for the doping effect. According to the theoretical calculation by Miao and Lambrecht [16], in the case of Mn-doping on the Si-site in the 3C–SiC lattice, the strong energetic preference for ferromagnetic over antiferromagnetic coupling is found when the lattice relaxation is induced. Here, an interesting question arises: how many initially incorporated Mn atoms in SiC are needed in order to enable a ferromagnetic coupling? It is clear that the ferromagnetic coupling cannot be explained simply in terms of direct, double, or superexchange interaction between Mn atoms since the average Mn–Mn distance is too large for such a coupling to exist. Therefore, an actual understanding of the origin of the observed ferromagnetism will require detailed \textit{ab initio} studies. Recently, on the basis of density functional theory (DFT), Cui \textit{et al} [30] have demonstrated that in Cr-doped GaN system the Cr atoms prefer to occupy Ga sites and have a strong tendency to form ‘embedded clusters’ while maintaining the wurtzite structure of GaN. Indeed, their preliminary calculations in Mn-doped SiC system show very similar behavior, i.e. the Mn atoms in the SiC lattice have a tendency to form ‘embedded clusters’ while maintaining the SiC structure: when two Mn atoms get close to each other, the ferromagnetic coupling is favored over the antiferromagnetic one, for the three Mn clustering configurations, the partially ferromagnetic or ferrimagnetic, not the antiferromagnetic state, is the ground state [31]. Nevertheless, we do not completely rule out any other alternative explanation of
Figure 7. (a) Fourier transform of Mn K-edge EXAFS oscillation functions $k^2 \chi(k)$ spectra for the synthesized sample and Mn foil, respectively; (b) Theoretical EXAFS spectrum for Mn and the substitutional Mn atom on the C- and Si-sites in the 3C–SiC lattice. The FT was performed in the $k$ range of 3.0–12.0 Å$^{-1}$. The Debye–Waller factor was assumed as 0.075 Å for bond length below 4.0 Å, and 0.100 Å for bond length above 4.0 Å. The theoretical EXAFS spectra were generated by FEFF8.

the origin of the observed ferromagnetism in this system. Further experimental studies based on microscopic investigations are required to validate any such claims of the ferromagnetic ordering in Mn-doped SiC.

4. Conclusions

We have synthesized a Mn-doped SiC film having a $T_c$ of 300 K on the 3C–SiC(001) homoepitaxial wafer using the annealing method. By a combination of SQUID, MCD and EXAFS results, we strongly propose that the observed room-temperature ferromagnetism in the sample is of intrinsic origin, arising from the ‘embedded Mn atoms’ in the 3C–SiC lattice, rather than other magnetic clusters. This is consistent with recent theoretical models of Mn-doped SiC DMS [16]–[27], [29, 30]. Another important issue that is addressed in our results is that it may eventually be possible to use 3C–SiC as a host material for achieving a high-$T_c$ ferromagnetic DMS. Although the results presented here, in general, are encouraging from a fundamental perspective, potential device application will likely demand continued progress in understanding such as how the magnetic properties and electronic structures are influenced by...
varying Mn concentration in SiC and these areas are still in their infancy. Finally, we hope that our observations can contribute to an increased investigation of SiC-based DMS materials.

Acknowledgments

We thank Drs X Y Cui and A Sinsarp for valuable discussions and suggestions. This work was partially supported by the Photon Factory Program Advisory Committee (project no 2006G069). One of us (WHW) thanks the Japan Society for the Promotion of Science (JSPS) for financial support.

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