Quest for high-Curie temperature $\text{Mn}_x\text{Ge}_{1-x}$ diluted magnetic semiconductors for room-temperature spintronics applications

Tianxiao Nie^{*}, Jianshi Tang, and Kang L. Wang^{*}
Device Research Laboratory, Department of Electrical Engineering,
University of California, Los Angeles, California 90095, USA

^{*}Address correspondence to. E-mail: wang@seas.ucla.edu; nietianxiao@gmail.com

Abstract:

In this paper, we report the non-equilibrium growth of various Mn-doped Ge dilute magnetic semiconductor nanostructures using molecular-beam epitaxy, including quantum dots, nanodisks and nanowires. Their detailed structural and magnetic properties are characterized. By comparing the results with those in $\text{Mn}_x\text{Ge}_{1-x}$ thin films, it is affirmed that the use of nanostructures helps eliminate crystalline defects and meanwhile enhance the carrier-mediate ferromagnetism from substantial quantum confinements. Our systematic studies provide a promising platform to build nonvolatile spinFET and other novel spintronic devices based upon dilute magnetic semiconductor nanostructures.

Keywords: A1. Quantum dots; A1. Nanodisks; A1. Nanowires; A3. Molecular beam epitaxy; B1. $\text{Mn}_x\text{Ge}_{1-x}$; B2. spinFET

1. Instruction:

The *International Technology Roadmap of Semiconductors* (ITRS) has highlighted the demands of novel materials and devices to satisfy the challenges in the continuous scaling of CMOS with low power dissipation and low variability [1-2]. Spintronics, which utilizes the spin of electrons as another degree of freedom for information processing, offers a promising pathway to meet such challenges [2]. As a unique material system, diluted magnetic semiconductor (DMS), by introducing magnetic dopants into a semiconductor lattice, plays an irreplaceable role in developing peculiar spintronic devices due to the advantage of simultaneously utilizing the charge and spin of electrons for information processing [3-4]. Among various DMS materials, Mn-doped Ge DMS [5] has attracted tremendous attention due to its potentially high Curie temperature and high compatibility with the mature Si technology. Especially, the discovery of electric-field controlled ferromagnetism in $\text{Mn}_x\text{Ge}_{1-x}$ systems [5-6] opens up great possibilities to realize low-power spintronic devices with increased functionalities.

For room-temperature spintronics applications, single-crystalline $\text{Mn}_x\text{Ge}_{1-x}$ with high Curie temperature ($T_c > 300\text{K}$) is highly desired. However, in practice it is very challenging. In the growth of $\text{Mn}_x\text{Ge}_{1-x}$ thin films, due to the extremely low Mn solubility in Ge, various intermetallic compounds (such as Mn_5Ge_3 and $\text{Mn}_{11}\text{Ge}_8$) are easily formed in a random and uncontrollable manner [7-8]. In fact, the acquisition of single-crystalline $\text{Mn}_x\text{Ge}_{1-x}$ DMS is in an extremely narrow growth window with poor reproducibility. In the pioneered work, Park et al. [5] successfully obtained such genuine $\text{Mn}_x\text{Ge}_{1-x}$ thin film and demonstrated the hold-mediated ferromagnetism, which greatly stimulated the research in the Mn-doped Ge system. However, the achieved T_c was only 116 K, far away from most practical applications. Later research aimed to further increase the T_c and electric-field controllability at room temperature. However, extensive studies in $\text{Mn}_x\text{Ge}_{1-x}$ thin films have proved the origin of room-temperature ferromagnetism is likely attributed to the presence of metallic phase Mn_5Ge_3 ($T_c \sim 296\text{K}$) [8-11], whose ferromagnetism cannot be controlled by external electric field. Recently, Jamet et al. [12] reported the formation of $\text{Mn}_x\text{Ge}_{1-x}$ thin film with T_c over 400 K, which was due to the coherent $\text{Mn}_x\text{Ge}_{1-x}$ nanocolumn with the new phase of MnGe_2 . The narrow growth window and poor reproducibility made it difficult to produce high-quality Ge-based DMS for reliable spintronic devices [13]. To date, although great progress has been made in growing $\text{Mn}_x\text{Ge}_{1-x}$ DMS, the biggest challenge is still how to avoid the formation of intermetallic precipitates and secondary phases.

To circumvent this problem, the employment of nanostructures might open up a possibility to generate high- T_c DMS with no precipitates. Two distinctive advantages could be introduced by employing nanostructures. 1) When it comes to the one or zero dimensions, the cluster formation may be possibly limited because the strain induced by Mn substitute could be easily accommodated by the nano-architecture [14]. 2)

When it comes to nanoscale structures, the quantum confinement effect emerges, which significantly enhances the exchange coupling between the confined holes and localized Mn [6, 15]. In fact, previous report has demonstrated that chemical vapor deposition (CVD)-grown $\text{Mn}_x\text{Ge}_{1-x}$ nanowires has a T_c over 300 K without any precipitation in the detection limitation of TEM [14]. Compared to CVD method, the non-equilibrium technique of molecular beam epitaxy (MBE) [3] growth allows us to incorporate much higher Mn-doping concentration into the Ge matrix beyond the solubility limit, which could facilitate to further increase the T_c .

In this work, we perform a systematic study on various MBE-grown $\text{Mn}_x\text{Ge}_{1-x}$ materials, and in particular, investigate the structural and magnetic properties of different nanostructures, including quantum dots, nanodisks and nanowires. By comparison with the $\text{Mn}_x\text{Ge}_{1-x}$ thin film, $\text{Mn}_x\text{Ge}_{1-x}$ nanostructures showed a much higher T_c over 400 K with minimized crystalline defects. Therefore, they provided a promising platform to build novel spintronic devices.

2. $\text{Mn}_x\text{Ge}_{1-x}$ thin film growth by MBE

As a control sample in our comparison study, $\text{Mn}_x\text{Ge}_{1-x}$ thin films were grown on Ge (111) substrate by a Perkin-Elmer solid source MBE with Ge and Mn Knudsen cells. Ge substrate was cleaned by immersing in acetone and isopropyl alcohol with ultrasonic agitation. After dipping in 1% diluted hydrofluoric acid (HF) solution, the substrate was directly transferred into the MBE chamber. The degassing was performed at 700 °C for 0.5 hour. After that, a 45 nm-thick $\text{Mn}_x\text{Ge}_{1-x}$ was directly deposited onto the Ge substrate at 200 °C with a Ge growth rate 0.2 Å/s. The microstructure and composition of the samples were comprehensively characterized by transmission electron microscopy (TEM) equipped with energy-dispersive spectroscopy (EDS). The magnetic properties were measured by superconducting quantum interference device (SQUID).

Figure 1(a) is a typical cross-sectional bright-field TEM image of the grown $\text{Mn}_x\text{Ge}_{1-x}$ film, in which some much dark regions are embedded in a relatively uniform contrast. It means other nanostructures were formed except for the relatively uniform doped MnGe as marked by red-dotted square. To understand the detailed structure, high-resolution TEM (HRTEM) was employed and typical [110] zone-axis TEM results were shown in Figures 1(b)-(d). In Figure 1(b), stacking faults (SFs) could be clearly observed in the Ge matrix, in which a triplet periodicity of $(111)_{\text{Ge}}$ lattice spacing in the SFs was explicitly featured [16]. The formation of SFs might come from the strain accumulation of Mn doping and lattice-mismatched precipitates. Figure 1(c) was collected from the relatively uniform doped region and clearly displayed a very good crystallinity, exhibiting a DMS feature. We must mention that noticeable precipitates could be observed in our thin film sample, as shown in Figure 1(d). It clearly shows another set of lattice structure different from the Ge matrix. Using the lattice spacing of the Ge as the reference, we calculated the observed lattice spaces of the cluster, which matched with the (002) and (010) atomic planes of the hexagonal Mn_5Ge_3 phase [17-18]. Furthermore, it was confirmed that the Mn_5Ge_3 (002) plane was parallel to the Ge (111) plane. To determine the Mn doping concentration, EDS was carried out and the result showed the average Mn concentration was ~4%, as shown in Figure 1(e).

The magnetic property of the grown $\text{Mn}_x\text{Ge}_{1-x}$ film was measured by SQUID, and the result is shown in Figure 1(f). Clear magnetic hysteresis can be observed between 10 K and 250 K, and it disappeared around 300 K. To further determine the T_c and detect any magnetic precipitates, zero-field cooled (ZFC) and field cooled (FC) magnetization measurements were performed under a small magnetic field of 200 Oe. As shown in Figure 1(g), the magnetization vanished near 300 K, indicating a $T_c \sim 300$ K, which further confirms the formation of Mn_5Ge_3 ($T_c \sim 296$ K) [12]. Two blocking temperatures coexisted in the ZFC curve, with the lower one at 20 K and the higher one at 200 K, which were attributed to Mn-rich coherent $\text{Mn}_x\text{Ge}_{1-x}$ nanostructures [13] and Mn_5Ge_3 precipitates [10], respectively. Both of them could be well resolved in the TEM characterization in Figure 1(a), as indicated by white arrows and red-dotted squares, respectively.

Based upon the above TEM characterization and magnetic property measurement, Mn-rich $\text{Mn}_x\text{Ge}_{1-x}$ lattice-coherent phases and Mn_5Ge_3 clusters were hard to be eliminated in the thin film growth, which hampered the formation of single-crystalline $\text{Mn}_x\text{Ge}_{1-x}$ DMS with high T_c . Therefore, it is of great interest to further investigate the growth of $\text{Mn}_x\text{Ge}_{1-x}$ nanostructures.

Figure 1. (a) A typical cross-sectional TEM image of the $\text{Mn}_x\text{Ge}_{1-x}$ thin film. (b)-(d) HRTEM images of stacking faults, $\text{Mn}_x\text{Ge}_{1-x}$ DMS region and Mn_5Ge_3 cluster in the film, respectively. (e) EDS spectrum confirming the $\sim 4\%$ Mn doping. (f) Temperature-dependent hysteresis loop of the $\text{Mn}_x\text{Ge}_{1-x}$ film. (g) ZFC and FC curves, showing a $T_c \sim 300$ K.

3. Self-assembled $\text{Mn}_x\text{Ge}_{1-x}$ quantum dots by MBE

In our earlier work, we have successfully demonstrated the growth of $\text{Mn}_x\text{Ge}_{1-x}$ quantum dots (QDs) on *p*-type Si (100) substrate by MBE [6]. The growth of $\text{Mn}_x\text{Ge}_{1-x}$ QDs were carried out at 450°C with Ge growth rate of $0.2 \text{ \AA/s}$, abiding by the Stranski-Krastanow (SK) [19] growth mode. Cross-sectional TEM was employed to determine the structural characteristics of the grown $\text{Mn}_x\text{Ge}_{1-x}$ QDs, as shown in Figure 2(a). It revealed a dome-shaped nanodot on top of the Si substrate with Mn diffusion underneath. Meanwhile, it shows a perfectly single crystallinity without any observable defects and precipitates in the QDs, implying a pure DMS system. The temperature-dependent hysteresis loops are shown in Figure 2(b), in which the coercivity and saturated magnetic moment decreased with increasing temperature. However, even at 400 K , the hysteresis loop still existed, indicating the T_c of $\text{Mn}_x\text{Ge}_{1-x}$ QDs was over 400 K . The fact of the high T_c in $\text{Mn}_x\text{Ge}_{1-x}$ QDs confirms that the use of nanostructures indeed helped enhance the T_c compared to thin films. Furthermore, using a metal-oxide-semiconductor (MOS) structure, the electric field control of ferromagnetism in the $\text{Mn}_x\text{Ge}_{1-x}$ QDs has been demonstrated, as shown in Figure 2(c). The experimental data suggest the itinerant holes play an important role in mediating the ferromagnetism.

Figure 2. (a) HRTEM image of a $\text{Mn}_x\text{Ge}_{1-x}$ QD. (b) Magnetic hysteresis loops from 5 K to 400 K . (c) Gate-dependent remnant moments and magnetic hysteresis loops (inset). Reproduced from Ref. [6].

4. Nanolithography-patterned $\text{Mn}_x\text{Ge}_{1-x}$ nanodisks by MBE

In addition to the effort on the growth of $\text{Mn}_x\text{Ge}_{1-x}$ QDs, another type of nanostructures – $\text{Mn}_x\text{Ge}_{1-x}$

nanodisks were investigated by MBE growth on a SiO_2 patterned Si substrate. The SiO_2 pattern was fabricated on a Si substrate by nanolithography [20], which utilized the nanospheres to self-assembly form hexagonal close-packed pattern. By series of etching process, the hexagonal close-packed pattern could be inversely transferred to the SiO_2 mask. Figure 3(a) is a typical AFM image of the SiO_2 mask, clearly showing a regular and repeatable nanohole pattern with a diameter of ~ 100 nm. After cleaned by standard RCA process [21], the patterned substrate was transferred to the MBE chamber for $\text{Mn}_x\text{Ge}_{1-x}$ nanodisks growth. After removing the native oxide layer at 950°C for 10 mins, the substrate was cooled down to 250°C for the $\text{Mn}_x\text{Ge}_{1-x}$ deposition with a Ge growth rate of $\sim 0.2 \text{ \AA/s}$. Figure 3(b) is a typical SEM image of the grown sample after removing the SiO_2 mask, which shows ordered $\text{Mn}_x\text{Ge}_{1-x}$ nanodisks with a diameter of 100 nm and uniform size distribution. The tilted SEM view in Figure 3(c) clearly demonstrates the ordered nanodisks are ~ 20 nm high. To understand the detailed microstructure, cross-sectional HRTEM was employed and a typical image is shown in Figure 3(d). A sharp interface between the Si substrate and the $\text{Mn}_x\text{Ge}_{1-x}$ nanodisks could be clearly observed with well-resolved lattice coherence. Careful examination could find there still exist some stacking faults in the $\text{Mn}_x\text{Ge}_{1-x}$ nanodisk, but without any noticeable precipitates, indicating a relatively good DSM system. Besides, EDS spectrum analysis in Figure 3(e) shows a Mn doping concentration of $\sim 4\%$. Magnetic properties of the grown $\text{Mn}_x\text{Ge}_{1-x}$ nanodisks were further characterized in SQUID as well. Figure 3(f) shows the temperature-dependent hysteresis loops with in-plane magnetic fields. The clear hysteresis loop at 300 K indicated that the T_c of the $\text{Mn}_x\text{Ge}_{1-x}$ nanodisks is above 300 K, affirming the enhancement of T_c in nanostructures. From the ZFC and FC curves as shown in Figure 3(g), the non-zero magnetization at 400 K further indicates that the T_c is beyond 400 K. The separation of the ZFC and FC curves possibly arises from strain-induced anisotropy because of a large lattice mismatch between Si and Ge [6].

Figure 3. (a) A typical AFM image of the ordered SiO_2 mask. (b) Top-view SEM image of the grown nanodisks with ~ 100 nm diameter. (c) Cross-sectional SEM image, showing the height is ~ 20 nm. (d) The corresponding HRTEM image, showing the coherent growth. (e) EDS spectrum showing a $\sim 4\%$ Mn doping. (f) Temperature-dependent hysteresis loops. (g) ZFC and FC magnetization of the nanodisks with $H = 100$ Oe.

5. Pattern-assisted MBE growth of $\text{Mn}_x\text{Ge}_{1-x}$ nanowires

Besides to the above quasi-zero-dimensional QDs and nanodisks, one-dimensional $\text{Mn}_x\text{Ge}_{1-x}$ DMS nanowires were also grown by MBE on a Ge substrate with SiO_2 nanotrench patterns as the mask [22]. After removal of the native oxide by annealing at 600°C for 30 mins, the substrate was cooled down to 180°C for the 60 nm-thick $\text{Mn}_x\text{Ge}_{1-x}$ nanowire growth with a Ge growth rate of $0.2 \text{ \AA/s}$. After the growth, cross-sectional

TEM was used to investigate its structural characteristics, and a typical image is shown in Figure 4(a). It shows that $\text{Mn}_x\text{Ge}_{1-x}$ nanowires are located on the Ge substrate and separated by the SiO_2 patterns. The inset HRTEM image clearly reveals a perfect crystallinity of the nanowire, in which no crystalline defects and MnGe precipitates were observed. Furthermore, magnetic properties of the grown $\text{Mn}_x\text{Ge}_{1-x}$ nanowires were measured by SQUID with in-plane magnetic fields. Figure 4(b) shows the temperature-dependent magnetic hysteresis loops with temperature range from 10 K to 400 K. Clear hysteresis loop can still be observed even at 400 K, which provides a direct evidence of T_c beyond 400 K. To investigate the electric-field controlled ferromagnetism, a MOS structure similar to the one in $\text{Mn}_x\text{Ge}_{1-x}$ QDs was fabricated using the $\text{Mn}_x\text{Ge}_{1-x}$ nanowires as channel. By sweeping the gate voltage from positive to negative, the carrier density in the $\text{Mn}_x\text{Ge}_{1-x}$ DMS nanowires was modulated. Figure 4(c) shows the gate-dependent coercivity, and the inset shows the magnified magnetic hysteresis loops recorded at various gate voltages. It is found that the coercivity decreased with increasing positive gate voltage, which depleted holes in the $\text{Mn}_x\text{Ge}_{1-x}$ nanowire. This result confirmed the ferromagnetism in the $\text{Mn}_x\text{Ge}_{1-x}$ nanowire was mediated by itinerant holes, similar to the scenario in the $\text{Mn}_x\text{Ge}_{1-x}$ QDs. Our results further confirmed that the use of DMS nanostructures could enhance the carrier-mediate ferromagnetism and meanwhile minimize crystal defects because of the quantum confinements.

Figure 4. (a) Cross-sectional and HRTEM images (inset) of the $\text{Mn}_x\text{Ge}_{1-x}$ nanowires. (b) Magnetic hysteresis loops from 10 K to 400 K. (c) Gate-dependent coercive fields and M-H loops (inset), indicating electric-field control of ferromagnetism in the $\text{Mn}_x\text{Ge}_{1-x}$ DMS nanowires.

6. Conclusion

In conclusion, we have demonstrated the MBE growth of various single-crystalline high- T_c $\text{Mn}_x\text{Ge}_{1-x}$ nanostructures, including QDs, nanodisks, and nanowires, in order to address the defects issue in the thin film growth. Detailed TEM structural analysis has excluded the presence of metallic precipitates or secondary phases in these nanostructures. Magnetic property characterizations show that the T_c is above 400 K. The enhancement in the carrier-mediated ferromagnetism and the single crystallinity is attributed to the substantial quantum confinement in nanostructures. Therefore, our findings provide great opportunities to build practical spintronic devices, such as DMS-based transpinor [22-23], using DMS nanostructures.

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