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14. ABSTRACT
This report describes a research project which was directed at the synthesis and self-assembly of diluted magnetic semiconducting nanocrystals using high temperature organic solution techniques in an effort to advance the fundamental understanding of spin-related phenomena in nanometer-scale diluted magnetic semiconductors. This project was in support of a DARPA sponsored project entitled "Spin Transport and Dynamics in Nano-Scaled Quantum Structures" (Grant No. MDA972-02-1-0001), which began January 1, 2002 and continued through June 30, 2004.

15. SUBJECT TERMS
diluted magnetic semiconductors (DMS); magnetic nanocrystals, spin transport and dynamics phenomena, nano-meter scaled materials

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Title of Research	Synthesis and Spin Dynamics/Coherence Properties of Nanometer-Sized Diluted Magnetic Semiconductors (DMS)
Principal Investigator	Dr. Charles J. O'Connor
Organization	University of New Orleans

Final Technical Report – September 30, 2004

Technical Objectives

The primary objective of this research was to advance the fundamental understanding of spin-related phenomena in nanometer-scale diluted magnetic semiconductors.

Progress Summary (06/01/2002 – 12/31/2002)

The synthesis and analysis of diluted nanoscale magnetic semiconductor (DMS) materials have attracted great interest due to their potential application in the new field of spintronics. $Mn_{(x)}Pb_{(1-x)}Se$ nanocrystals (NCs) are typical diluted magnetic semiconducting nanomaterials and possess very promising properties. However, there has no report concerning the investigation of this kind of material. We initialized this project from the synthesis and characterization of $Mn_{(x)}Pb_{(1-x)}Se$ NCs. The main challenge in the synthesis of such doped nanocrystals has been to introduce the magnetic impurity in the core of the nanocrystals without compromising the quality of the nanocrystals. We have, for the first time, demonstrated the successful synthesis of these Mn-doped PbSe nanocrystals (NCs) by employing a high temperature organic solution approach with proper capping agent, and have characterized them using a number of techniques including TEM, EPR, SQUID, XANES, EDS and NIR. This investigation has exhibited the presence of doped-Mn ions in PbSe NCs and has also shown that the enhancement of intra-nanocrystals spin-spin exchange interactions is greatly dependent on the amount and the location of the dopant (Mn-ions). These accomplishments have been summarized into an academic paper J. Am. Chem. Soc., 125(28), 8448 - 8449 (2003), and later on been highlighted by the ACS web portal on September 1, 2003.

Technical Progress (06/01/2002 – 12/31/2002)

Our initial work under this project focuses on the synthesis and characterization of $Pb_{(1-x)}Mn_xSe$ nanocrystals using an organic colloidal solution approach at high temperature. $Pb_{(1-x)}Mn_xSe$ nanocrystals were prepared by decomposition of Pb-, Mn-Se-precursors and Se-TOP complex in TOP/TOPO solution at high temperature. We employed lead (II) (2-ethylhexanate) [$Pb(EH)_2$] and $Mn_2(\mu\text{-}SeMe)_2(CO)_8$(Mn-Se*) as the precursors of Pb- and (Mn-Se*), respectively. The preparation procedure is briefly described as follows:

$Pb(EH)_2$, Mn-Se* and TOP-Se solution were pre-mixed in a glovebox with Ar, and the mixture was quickly injected into a solution of TOPO at 260 - 300 °C under Ar atmosphere.

Once the reaction mixture turns to dark brown color, the heating source was removed and the stirring was kept continuously until the temperature dropping down to room temperature. The as-prepared $Pb_{(1-x)}Mn_xSe$ NCs were then purified through a size-selection post treatment by repeatedly precipitating particles with a pair solvents of ethanol-toluene and re-dispersing them into toluene. The content of $Pb_{(1-x)}Mn_xSe$ NCs can be changed in certain range by varying the precursor ratio between $Mn_2(\mu\text{-}SeMe)_2(CO)_8$(Mn-Se*) and other reagents. Through this process, we are able to obtained monodisperse spherical $Pb_{(1-x)}Mn_xSe$ NCs ranging around ~10 nm in diameter with monodistribution ($\delta \leq 8\%$) (see Figure 1). We also varied the Mn-concentration in the samples and conducted various chemical analyses for verifying the existence and real concentration of Mn^{2+} in these NCs. (see Figure 2).

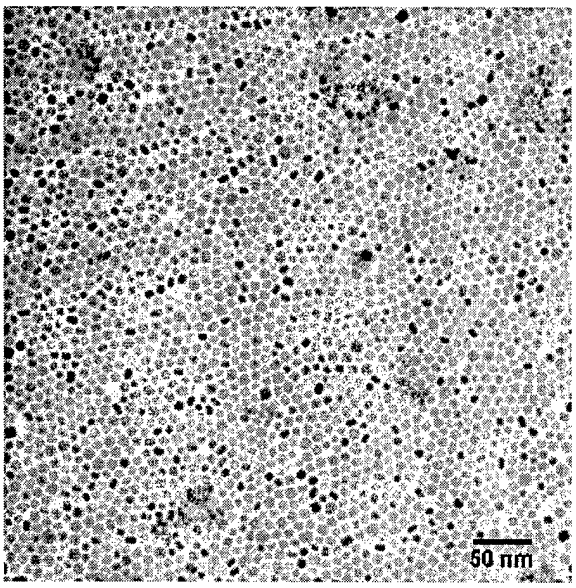

Figure 1. TEM image showing the morphology of $Pb_{(1-x)}Mn_xSe$ NCs.

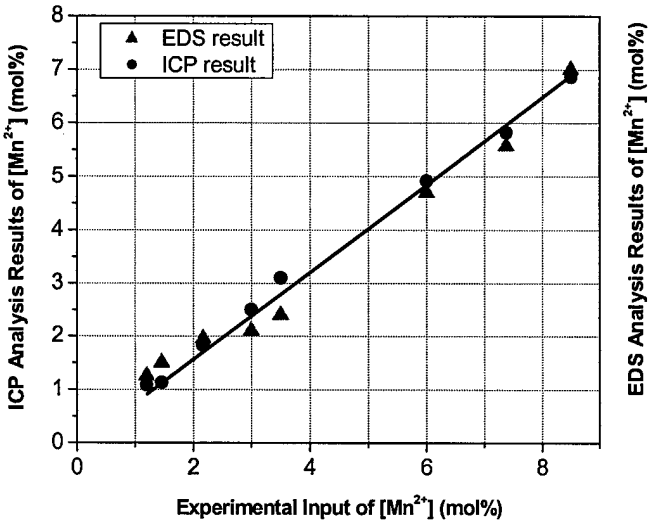

Figure 2. A verification of Mn^{2+} concentration by EDS and ICP analyses.

Progress Summary (01/01/2003 – 12/31/2003)

During this period, three parts of progress have been achieved at AMRI. **(1)** We have further characterized various $Pb_{(1-x)}Mn_xSe$ NCs using XRD, SQUID and EPR methods, demonstrating that (a) the Mn^{2+} has been really doped into the PbSe lattice rather than on the surface; (b) below the Mn^{2+} concentration of 4%, increase of Mn^{2+} results in a lattice contraction; (c) the intensity of the spin-nucleus interactions decreases with an increase of Mn-concentration. **(2)** We have also synthesized Mn-doped CdSe NCs using high temperature organometallic method and observed that the size effects enhanced dynamic magnetic interactions in electron spins from the Mn^{2+} ions from EPR results. The Mn-Mn interaction is enhanced and the transverse relaxation time (T^2) is increased due to quantum confinement effects. The quantized electrons enhance spin phase coherence and Mn-Mn interactions in the nanocrystalline quantum dots, indicating that electron spins from doped magnetic ions in NCs may provide long spin lifetime and may benefit the application in quantum computing. **(3)** We have preliminarily measured the Faraday rotation as a function of the wavelength of Mn-doped CdSe samples,

determining a peak at ~640 nm due to the existence of Mn^{2+}; We have also continuously further studied the static magnetic interactions between electron spins of Mn^{2+} ions introduced in CdSe NCs, and observed the difference of magnetic behaviors at low and high magnetic fields.

Technical Progress (01/01/2003 – 12/31/2003)

Progress 1:

To differentiate whether the doped Mn ions are on the surface or in the PbSe NCs, we measured the lattice constant using XRD. A plot of the lattice parameter (a) vs molar percentage of Mn^{2+} together with a typical TEM diffraction pattern is shown in Figure 3. The recorded XRD patterns are provided in Figure 4. All the detectable peaks are indexed to almost the same positions as those from a standard bulk and from cubic PbSe NCs, but the measured lattice constant is different. We applied Cohen's method to estimate the average lattice constant (a) and found a lattice contraction after introduction of Mn ions. We therefore believe that the Mn ions are distributed inside the NCs rather than on the surface, since the substitution of Pb^{2+} by Mn^{2+} ions may cause some stress and strain effects to shrink the lattice structure. Based on the line broadening at half maximum of the peaks according to Scherrer equation, the average crystalline size estimated is close to that from our TEM observation.

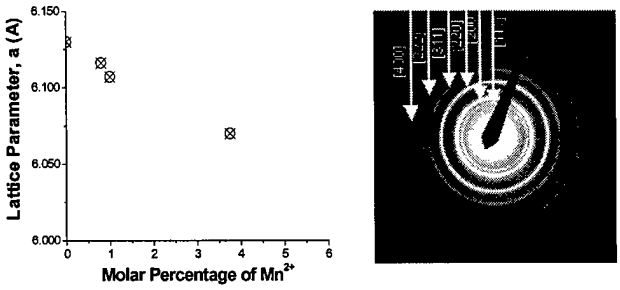

Fig. 3. (left) A plot of the lattice parameter, a, vs molar percentage of Mn^{2+}; (right) Diffraction patterns of $Pb_{0.985}Mn_{0.015}Se$ (NC size:10.5 nm).

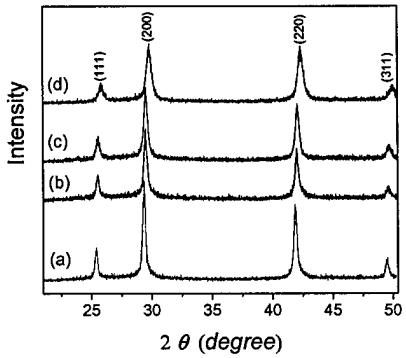

Fig. 4. XRD traces of $Pb_{1-x}Mn_xSe$ NCs measured at RT: pattern (a) is for pure PbSe (10 nm); (b) for $Pb_{0.992}Mn_{0.008}Se$; (c)for $Pb_{0.990}Mn_{0.010}Se$; and (d) for $Pb_{0.962}Mn_{0.038}Se$.

To ensure that the Mn ions are really introduced into the PbSe NCs, we further performed magnetic measurements by using a SQUID. The PbSe semiconductor is a diamagnetic material confirmed by SQUID result of our pure PbSe NCs (10 nm). When we doped the NCs with Mn^{2+} ions, their magnetic properties dramatically changed from diamagnetic (negative magnetization) into paramagnetic (positive magnetization) behavior. The paramagnetic property displaying a linear dependence between inverse susceptibility ($1/\chi$) and temperature (T) is shown in Figure 5 (a). The slope of each line, which is relative to magnetic moments per NC, does change with increasing of the Mn-concentration. We fitted it with the Curie-Weiss law to confirm the Mn-concentration inside the PbSe NCs. The fitted concentration of our $Pb_{0.953}Mn_{0.047}Se$ sample is 0.0481 and the fitted Weiss temperature is 4.36 K. The Weiss temperatures for all of our samples are in the range of 3 ~ 5 K which agrees well with a previous result of bulk samples. In Figure 5 (b) we show the intensity dependence of hyperfine splitting on Mn-concentration carried out by

EPR. Each EPR spectrum displays a superposition of a broad curve and six lines of sharp splittings (Figure 6). The broad curve is due to electron's spin-spin interactions (1/2→-1/2) of isolated Mn ions in the $Pb_{1-x}Mn_xSe$ NCs whereas the sharp splitting lines (hyperfine splittings) are due to spin-nucleus interactions. A symmetric Lorentzian line of the broad curve is observed since the sample size is smaller than the skin depth. The nuclear spin of Mn ions is 5/2, therefore six hyperfine splittings are observed in the EPR spectra. Hyperfine splitting spectra of various Mn-concentrations were obtained by a subtraction of spin-spin interaction after it was normalized (as shown in Fig. 5 (b)). This hyperfine structure shows the electron spin - nuclear spin interactions in an isolated Mn ion. As the Mn ion is affected by other Mn ions that are randomly distributed around it, the Mn-Mn interactions should result in a reduction of the electron spin-nuclear spin interactions in an individual Mn ion. We did observe that the intensity of the spin-nucleus interactions decreases with an increase of Mn-concentration. The result confirms again that the Mn ions are embedded in the lattice of our PbSe NC samples. Part of above results has been published in ***IEEE Trans. Mag.***, 39(5), 2791-2793 (2003).

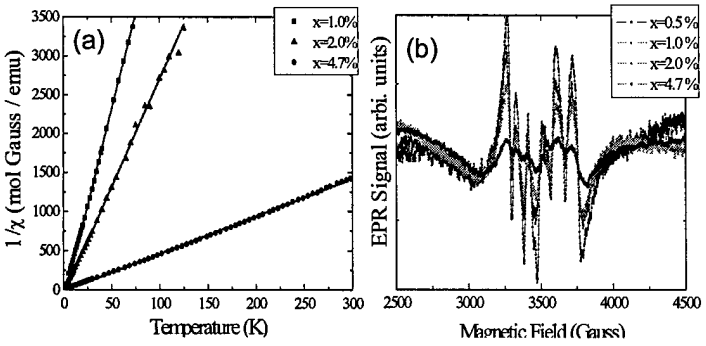

Fig. 5. (a) The inverse susceptibility of $Pb_{1-x}Mn_xSe$ NCs measured by SQUID at a field of 10k Gauss displaying linear dependence on temperature. The slope of each line is inversely depending on Mn concentration; (b) The EPR spectra carried out in an x-band at RT showing that the intensity of hyperfine splittings is variable depending

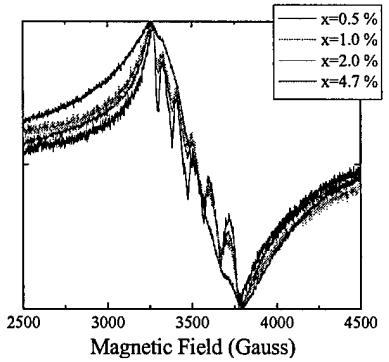

Fig. 6. EPR spectra of four $Pb_{1-x}Mn_xSe$ NC samples with different Mn concen-tration measured at room temperature ,using the x-band frequency of 9.87 GHz. The intensity of hyperfine splitting decreases when increasing the concentration of Mn

Progress 2:

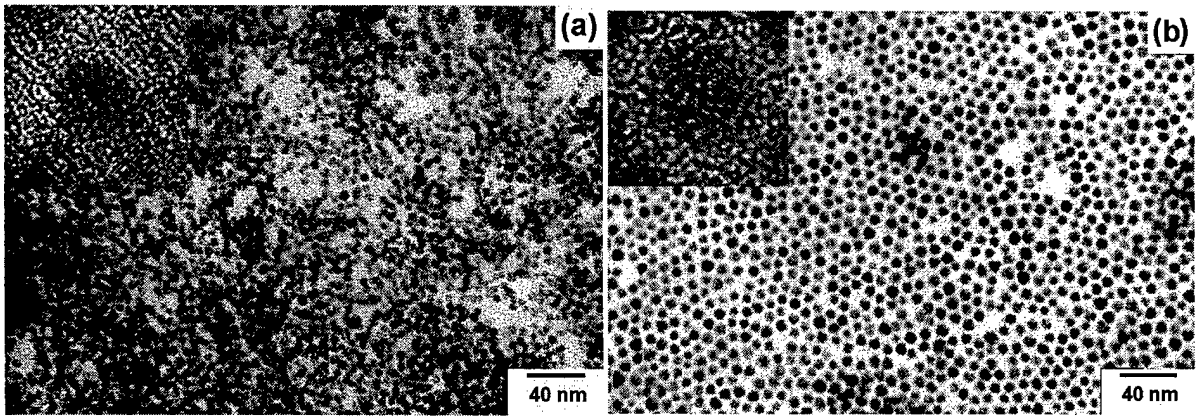

Fig. 7: TEM images of $Cd_{1-x}Mn_xSe$ NCs with sizes of (a) 5 nm and (b) 8 nm. Dark gray and light gray spheres indicate the same concentration but different rotation of lattice direction among NCs. Insets: high resolution TEM images showing nanocrystalline structure.

We have further synthesized Mn-doped CdSe NCs using a high-temperature organic solution approach and have observed the size-effect-enhanced dynamic magnetic interactions in electron spins from the Mn^{2+} ions from EPR results. $Cd_{1-x}Mn_xSe$ NCs with monodisperse sizes of 5 and 8 nm were successfully prepared by employing the same Mn-precursors mentioned above. The nanocrystalline structure of NCs with diameter of 5 and 8 nm is shown in Figs. 7. The EPR spectra recorded at 9.86 GHz and at room temperature for $Cd_{1-x}Mn_xSe$ NCs with sizes of 5 and 8 nm are shown in Figs.8. Hyperfine structures can be observed for all samples with Mn-concentration lower than 1.5 at %. Fig. 9 reveals the electron spin-nuclear spin interactions after a normalization and a subtraction of spin-spin interactions from EPR spectra for $Cd_{1-x}Mn_xSe$ QDs with a size of 8 nm. A linewidth analysis from Fig. 8 for the two sizes of NCs with a serious variation of Mn-concentartion indicates that, (1) for the same concentration of Mn-ions, the line-width is broader in large-size QDs than that in small-size ones; (2) the Mn-Mn interactions can be also estimated from the inverse of peak to peak intensities of hyperfine splittings [Fig. 9]. It is therefore concluded that the Mn-Mn interaction is enhanced and the transverse relaxation time (T^2) is increased due to quantum confinement effects. The quantized electrons enhance spin phase coherence and Mn-Mn interactions in the nanocrystalline NCs, indicating that electron spins from doped magnetic ions in NCs may provide long spin lifetime and may benefit the application in quantum computing. This investigation was published in ***Appl. Phys. Lett.***, 83(16), 3377-3379 (2003).

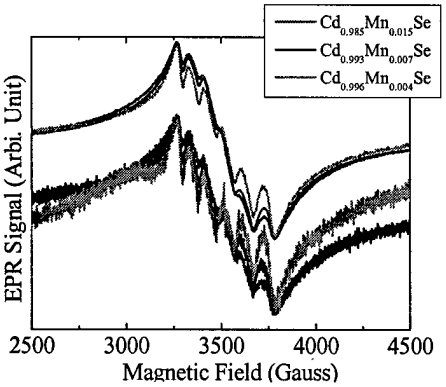

Fig. 8. EPR spectra recorded at 9.86 GHz and at room temperature for $Cd_{1-x}Mn_xSe$ NCs with sizes of 5 (top) and 8 (bottom) nm. Hyperfine structures can be observed for all samples with Mn-concentration lower than 1.5 at %.

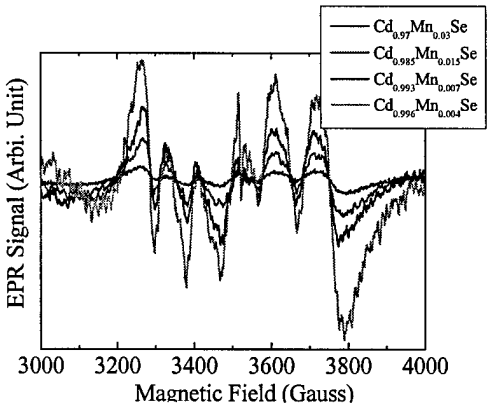

Fig. 9: Electron spin-nuclear spin interactions after a normalization and a subtraction of spin-spin interactions from EPR spectra for $Cd_{1-x}Mn_xSe$ NCs with a size of 8 nm.

Progress 3:

By using a SQUID magnetometer, we have further studied the static magnetic interactions between electron spins of Mn^{2+} ions introduced in CdSe NC with different crystalline sizes. The NC with a series variation of low Mn-concentration all behave paramagnetic. The temperature dependent dc magnetic susceptibility was studied under several different fields. We have observed that the paramagnetic materials in the nanophase do not obey the Curie-Weiss law

under a high magnetic field and at high temperature. In addition, the novel effect disappears when the sizes of the quantum dots as well as their Mn-concentrations are increased. One of the typical results is illustrated in Figure 10. We found that the inverse susceptibility is linearly dependent on temperature in the low temperature range (T < 20 K).

At a high temperature, the temperature dependence of inverse susceptibility is either a convex curve (low fields) or a concave curve (high fields). The higher the external field is raised, the larger the inverse susceptibility becomes, indicating that the magnetization is reduced under a high magnetic field at a high temperature. This is confirmed by the measurement of field dependent magnetization. These results were drafted and submitted for publish in ***J. Magn. Magn. Mater.*** (2003). We have also concentrating on the Faraday rotation measurement on these $Cd_{1-x}Mn_xSe$ samples using a Magneto-optical characterization equipment (Oxford Instruments) at UNO. As illustrated in Figure 12, a rotation peak at ~640 nm can be clearly detected from those Mn-doped samples but not pure CdSe NCs. A manuscript concerning these results is under drafting.

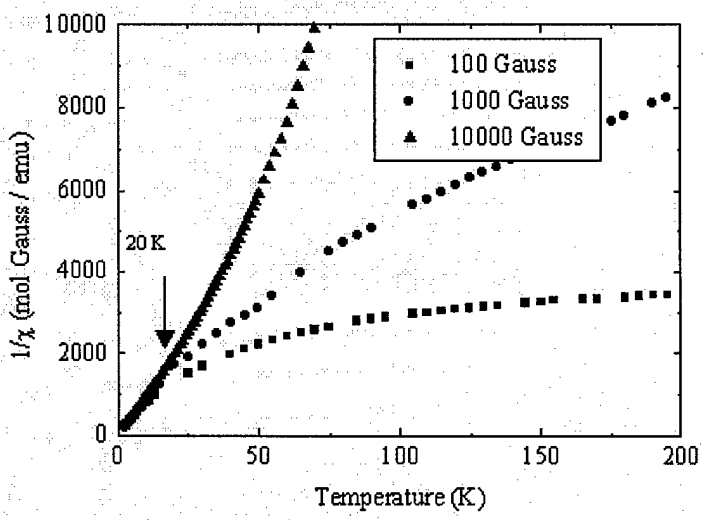

Fig. 10 The inverse susceptibility of $Cd_{0.996}Mn_{0.004}Se$ NCs as a function of temperature under magnetic fields of 100 (closed triangles), 1000 (closed circles) and 10000 (closed squares) gauss. The mean size of NCs is 5 nm in diameter.

Progress Summary (01/01/2004 – 09/30/2004)

During this period, progress can be broken down to two parts. **(1)** We developed a *in-situ* self-assembly approach and prepared Mn-doped PbSe nanowires from high temperature organic solution; **(2)** We have for the first time synthesized PbTe NCs and achieved their cubic self-assembly.

Technical Progress (01/01/2004 – 09/30/2004)

Progress 1.

At the beginning of this year, we have also achieved a solution-phase processing for nanoarrays (NAs) of Mn-doped PbSe. In this work, we demonstrated a novel approach for large-scale, controlled synthesis of one-dimensional (1D) corrugated Nas (nanowires) through an *in-situ* self-assembly without using either capping polymer or ionic surfactant. This is a clear departure from the traditional methods of synthesizing nanowires. (2) The one-step-prepared 1D NAs (Fig. 11-left) exhibit a well-defined morphology, single-crystal-orientation and clean surface without amorphous characteristics. (3) The mechanism was clearly understood as

discussed in our submitted manuscript. Four growth models were suggested to explain the *in-situ* self-assembly processes based on the fundamental building blocks of octahedral nanocrystals by sharing {111} facets.

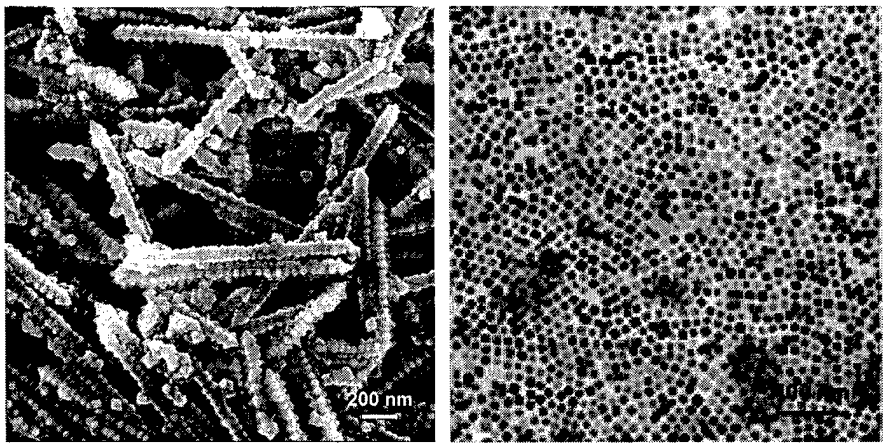

Fig. 11. Images of (left) *in-situ* self-assembled Mn-PbSe nanoarrays (SEM, bottom-left) and (right) monolayer PbTe cubic self-assembled NCs (TEM, bottom-right).

Progress 2.

We have, for the first time, achieved the synthesis and self-assembly of perfect cubic PbTe NCs with well-defined fcc structure using the high-temperature organic solution approach as well. As revealed by the XRD determination (Fig.12), such self-assembled quantum dots (SAQDs) (nanocubes) exhibit {200}-perfect-orientated alignment which is different from that of the spherical SAQDs. We have also investigated the mechanism of the nanocrystalline evolution from spherical to cubic structure. This demonstration creates an additional avenue to employ these highly orientated PbTe cubic NCs as building blocks to achieve thickness-controlled films for a further manipulation of devices. In next step, we will dope Mn^{2+} into this type of NCs if we have chance to be supported.

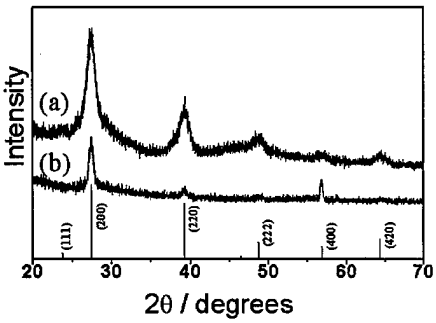

Fig. 12. XRD patterns of PbTe SAQDs on Si wafer: (a) spherical QDs (8 nm in diameter) (b) cubic QDs (14 nm in size)

Summary of Publications Derived from this Project (2002-2004):

Tianhao Ji, Wen-Bin Jian and Jiye Fang, "The First Synthesis of $Pb_{1-x}Mn_xSe$ Nanocrystals," ***J. Am. Chem. Soc.***, 125(28), 8448-8449 (2003).

Tianhao Ji, Wen-Bin Jian, Jiye Fang, Jinke Tang, Volodymyr Golub and Leonard Spinu, "Magnetic Properties of Nanocrystalline $Pb_{1-x}Mn_xSe$," ***IEEE Trans. Mag.***, 39(5), 2791-2793 (2003).

W.B. Jian, Jiye Fang, Tianhao Ji and Jibao He, Quantum-Size-Effect-Enhanced Dynamic Magnetic Interactions among Doped Spins in $Cd_{1-x}Mn_xSe$ Nanocrystals," ***Appl. Phys. Lett.***, 83(16), 3377-3379 (2003).

Weigang Lu, Puxian Gao, Wen Bin Jian, Zhong Lin Wang and Jiye Fang, "Perfect Orientation Ordered *in-situ* One-Dimensional-Self-Assembly of Mn-doped PbSe Nanocrystals," ***J. Am. Chem. Soc.,*** 126(45), 14816-14821 (2004).

Weigang Lu, Jiye Fang, Kevin L. Stokes and Jun Lin, "Shape-Evolution and Self-Assembly of Monodisperse PbTe Nanocrystals," ***J. Am. Chem. Soc.***, 126 (38), 11798–11799 (2004).

Kiko Sunderland, Philip Brunetti, Leonard Spinu, Jiye Fang, Zhenjun Wang and Weigang Lu, "Synthesis of γ-Fe_2O_3/Polypyrrole Nanocomposite Materials, ***Mater. Lett.,*** 58(25), 3136-3140 (2004).

W.B. Jian, Weigang Lu, Jiye Fang, S.J. Chiang, M.D. Lan and C.Y. Wu, Diamagnetic Properties of PbSe Spherical Quantum Dots," submitted to ***Appl. Phys. Lett.*** (2004).

W.B. Jian and Jiye Fang, "Magnetic Interaction in Mn-CdSe Quantum Dots," submitted to ***J. Magn. Magn. Mater.,*** (2004).

Zuqin Liu, Daihua Zhang, Song Han, Chao Li, Bo Lei, Weigang Lu, Jiye Fang and Chongwu Zhou, "Single Crystalline Magnetite Nanotubes," ***J. Am. Chem. Soc.,*** 127, xxx - xxx (2005). (accepted, ASAP article will be released on web in Dec. 2004)

Summary of Presentations Derived from this Project (2002-2004):

"Nanophase Diluted Magnetic Semiconductor Particles," Charles J. O'Connor and Jiye Fang, Presented on the Spins/Spintronics PI Review, September 30 - October 4, 2002; Delray Beach, FL, USA.

"Synthesis and Self-assembly of Semimetallic and Semiconducting nanocrystals with Control of Pattern Shape" Jiye Fang, Charles O'Connor, Kevin Stokes and Jibao He, Presented on the 1st International Conference and School: Nanoscale / Molecular Mechanic (N/M2), May 12-17, 2002, Outrgger Wailea Resort, Maui, Hawaii.

"Synthesis and Novel Properties of DMS NCs," Tianhao Ji, Wen-Bin Jian, Volodymyr Golub and Jiye Fang*, Presented on the DARPA/UNO.AMRI PI REview, February 26-28, 2003; Sheraton New Orleans Hotel, Louisiana.

"Preparation and Characterization of Mn-doped PbSe Nanocrystals with Interesting Shapes," Tianhao Ji, Jiye Fang* and Charles J. O'Connor, Abstracts of Papers, 225th ACS National Meeting, New Orleans, LA, United States, March 23-27, 2003 (2003), INOR-235.

"Simple Preparation and Optical Properties of CdSe and CdTe Nanocrystals", Tianhao Ji and Jiye Fang,* Abstracts of Papers, 225th ACS National Meeting, New Orleans, LA, United States, March 23-27, 2003 (2003), INOR-241.

"Self-assembly of PbSe Nanoparticles into Nanorings," Weilie L. Zhou, Jibao He, Jiye Fang, Tuyet-Anh Huynh, Trevor J. Kennedy, Kevin L. Stokes and Charles J. O'Connor, Presented on MRS 2003 Spring Meeting, Paper P10-5, San Francisco Marriott and Argent Hotels, San Francisco, CA, April 21- 25, 2003.

"Chemical Synthesis and Magnetic Interactions among Doped Spins in Mn-PbSe and Mn-CdSe NC's", Jiye Fang, Wen-Bin Jian, Tianhao Ji and Charles J. O'Connor, Presented on Annual DARPA DSO SpinS Spintronics PI Review, The Fairmont Miramar Hotel, Santa Monica, CA, October 13-16, 2003.

"Synthesis of γ-Fe_2O_3/Polypyrrole Nanocomposite Materials with Magnetic and Electrical Conductive Properties," Kiko Sunderland, Philip Brunetti, Leonard Spinu, Jiye Fang, Zhenjun Wang, Weilie L. Zhou, Weigang Lu, Volodymyr Golub, Hongguo Zhang and Eun Young Shin, 9^{th} Joint-MMM Intermag Conference, Anaheim, 2004.

"DARPA SPINS: Investigation of Spintronic NanoMaterials at AMRI," Jiye Fang, Leszek Malkinski and Weilie Zhou (co-presented) on DARPA Spins/Spintronics PI Review meeting, JW Marriott New Orleans, Feb. 19-20, 2004.