(Sr$_3$La$_2$O$_5$)(Zn$_{1-x}$Mn$_x$)$_2$As$_2$: A bulk form diluted magnetic semiconductor isostructural to the “32522” Fe-based superconductors

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Abstract - A new diluted magnetic semiconductor (DMS) system, $(Sr_3La_2O_5)(Zn_{1-x}Mn_x)_2As_2$, has been synthesized and characterized. 10% Mn substitution for Zn in bulk form $(Sr_3La_2O_5)Zn_2As_2$ results in a ferromagnetic ordering below the Curie temperature, $T_C \sim 40$ K. $(Sr_3La_2O_5)(Zn_{1-x}Mn_x)_2As_2$ has a layered crystal structure identical to that of 32522-type Fe-based superconductors, and represents the fifth DMS family that has a direct counterpart among the FeAs high-temperature superconductor families.

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Introduction. – The research of DMS (diluted magnetic semiconductors) has been explosive following the successful fabrication of III-V (Ga,Mn)As ferromagnetic thin films by Ohno et al. in the 1990s [1]. Over the past two decades, much progress has been made in the fabrication of DMS materials and in the understanding of the ferromagnetism [2–6]. On the other hand, most extensively studied DMS materials are thin films that are grown under non-equilibrium condition, which encounters some inherent difficulties. For example in (Ga,Mn)As, some Mn impurities enter the interstitial sites, and makes it difficult to precisely determine the amount of Mn that substitutes ionic Ga, which donates a hole and acts as a local moment [6]. The thin films also prohibit the utilization of powerful magnetic probes such as neutron scattering and nuclear magnetic resonance (NMR) that are based on bulk form specimens, to provide complementary information for understanding the ferromagnetism at a microscopic level. Seeking a bulk form DMS system grown in thermally equilibrium condition will be helpful to understand ferromagnetism.

Recently, through doping Mn into the I-II-V semiconductors LiZnAs and LiZnP, Deng et al. successfully synthesized two bulk DMS systems, Li(Zn,Mn)As [7] and Li(Zn,Mn)P [8], with $T_C \sim 50$ K. The I-II-V DMSs have advantages of decoupling spins and carriers, where spins are introduced by Mn atoms and carriers are created by off-stoichiometry of Li concentrations. This advantage makes it possible to precisely control the amount of spins and carriers, and investigate their individual effects on the ferromagnetic ordering. More recently, several more bulk DMS systems have been reported. Firstly, Ding et al. reported the ferromagnetic ordering below $T_C \sim 40$ K in a “1111”-type (La,Ba)(Zn,Mn)AsO system [9]; Han et al. reported the ferromagnetism in (La,Ca)(Zn,Mn)SbO semiconductor [10] and Yang et al. reported the fabrication of (La,Sr)(Cu,Mn)SO DMS with $T_C \sim 210$ K [11]. Secondly, Zhao et al. reported the “122”-type DMS system, (Ba,K)(Zn,Mn)$_2$As$_2$, which has $T_C$ as high as 180 K [12], and Yang et al. observed the ferromagnetic transition below $T_C \sim 17$ K and a large negative magnetoresistance in (Ba,K)(Cd,Mn)$_2$As$_2$ [13]. The Curie temperature of (La,Sr)(Cu,Mn)SO and (Ba,K)(Zn,Mn)$_2$As$_2$ polycrystals is already comparable to the record $T_C$ of (Ga,Mn)As thin films [14,15].

The availability of bulk form DMS specimens readily enables the microscopic investigation by $\mu$SR (muon spin relaxation) and NMR techniques. $\mu$SR has demonstrated that the exchange interaction supporting ferromagnetic coupling in Li(Zn,Mn)As, (La,Ba)(Zn,Mn)AsO,

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Table 1: The transition temperature $T_c$ for a superconductor (SC), Curie temperature $T_C$ for a diluted magnetic semiconductor (DMS) and Neel temperature $T_N$ for an antiferromagnet (AFM) of “11”, “111”, “111” “122”, “32522” and “42622” type compounds. The type of crystal structure and lattice constants are also listed for available compounds.

| SC                  | DMS                  | AFM                  |
|---------------------|----------------------|----------------------|
|                     | FeSe $(T_c \approx 8$ K) [20] | DMS $(Zn,Mn)Se$ $(T_f \approx 24$ K) [21] | MnSe $(T_N \approx 197$ K) [22] |
| Tetragonal          | Cubic                | Cubic                |
| $a = 3.7676$ Å      | $a = 5.669$ Å        | $a = 5.646$ Å        |
| $c = 5.4847$ Å      |                      |                      |
| “11”               |                      |                      |
|                     | LiFeAs $(T_c \approx 18$ K) [23] | Li$(Zn,Mn)As$ $(T_C \approx 50$ K) [7] | LiMnAs $(T_N \approx 393$ K) [24] |
| Tetragonal          | Cubic                | Tetragonal           |
| $a = 3.77$ Å        | $a = 5.94$ Å         | $a = 4.273$ Å        |
| $c = 6.36$ Å        |                      | $c = 12.370$ Å       |
| “111”              |                      |                      |
|                     | LaFeAs(O,F) $(T_c \approx 26$ K) [18] | La$(Ba)(Zn,Mn)As$O $(T_C \approx 40$ K) [9] | LaMnAsO $(T_N \approx 317$ K) [19] |
| Tetragonal          | Tetragonal           | Tetragonal           |
| $a = 4.0320$ Å      | $a = 4.116$ Å        | $a = 4.1398$ Å       |
| $c = 8.7263$ Å      | $c = 9.11$ Å         | $c = 9.03044$ Å      |
| “111”              |                      |                      |
|                     | (Ba,K)Fe$_2$As$_2$ $(T_c \approx 38$ K) [25] | (Ba,K)$(Zn,Mn)$_2$As$_2$ $(T_C \approx 180$ K) [12] | BaMn$_2$As$_2$ $(T_N \approx 625$ K) [26] |
| Tetragonal          | Tetragonal           | Tetragonal           |
| $a = 3.917$ Å       | $a = 4.131$ Å        | $a = 4.1684$ Å       |
| $c = 13.2968$ Å     | $c = 13.481$ Å       | $c = 13.4681$ Å      |
| “32522”            |                      |                      |
|                     | Ca$_3$Al$_2$O$_{6-x}$Fe$_2$As$_2$ $(T_c \approx 30.2$ K) [27] | Sr$_3$La$_2$O$_5$(Zn,Mn)$_2$As$_2$ $(T_C \approx 40$ K, this work) | hypothetical $(Sr_3La_2O_5Mn_2As_2)$ |
| Tetragonal          | Tetragonal           | Tetragonal           |
| $a = 3.742$ Å       | $a = 4.2612$ Å       |                      |
| $c = 26.078$ Å      | $c = 27.675$ Å       |                      |
| “42622”            |                      |                      |
|                     | Sr$_4$V$_2$O$_6$Fe$_2$As$_2$ $(T_c \approx 37.2$ K) [28] | Sr$_4$Ti$_2$O$_6$(Zn,Mn)$_2$As$_2$ $(T_C \approx 25$ K) (Unpublished) | hypothetical $(Sr_4Ti_2O_6Mn_2As_2)$ |
| Tetragonal          |                      |                      |
| $a = 3.9296$ Å      |                      |                      |
| $c = 15.6732$ Å     |                      |                      |

$(Ba,K)(Zn,Mn)$_2As$_2$ and $(Ga,Mn)As$ has a common origin and comparable magnitude for a given spatial density of ordered moments, no matter the specimens are thin films or bulk forms [7,9,12,16]. Moreover, Ding et al. has conducted $^7$Li NMR measurement of Li(Zn,Mn)P, and successfully detected the fast relaxed Li sites that have Mn at nearest-neighbor sites. They found that the Mn spin-spin interactions extend over many unit cells, which explains why DMSs could exhibit a relatively high $T_C$ with such a low density of Mn [17].

More interestingly, each of above “111”, “1111” and “122” DMSs families has a direct counterpart among the FeAs-based high-temperature superconductor families. For example, “1111”-type $(La,Ba)(Zn,Mn)As$ DMS has a ZrCuSiAs-type tetragonal structure, identical to FeAs-based “1111”-type LaFeAsO$_{1-x}$Fe$_x$ high-temperature superconductor $(T_c = 26$ K) [18] and the antiferromagnetic LaMnAsO $(T_N = 317$ K) [19]. The excellent lattice matching (lattice constants are within 5% difference) between ferromagnetic, antiferromagnetic and superconducting systems opens the possibilities to make junctions between these systems through the As layer. The parameters for all these compounds are listed in table 1. We also mention that for the “11”-type Fe-based superconductor FeSe [20], the counterpart is $(Zn,Mn)Se$, which has been extensively studied as one of the prototypical II-VI DMSs.

In this letter, we report a successful synthesis and characterization of a new bulk DMS system $(Sr_3La_2O_5)(Zn_{1-x}Mn_x)$_2As$_2$, which is iso-structural to the “32522” FeAs-based superconductor (Ca$_3$Al$_2$O$_3$)Fe$_2$As$_2$ [27], 5% Mn substitution for Zn in the parent semiconductor $(Sr_3La_2O_5)Zn$_2$As$_2$ results in ferromagnetic ordering, as indicated by the strong enhancement of magnetization below $T_C \approx 36$ K. The bifurcation of ZFC and FC curves below $T_f = 12.5$ K and a parallelogram-shaped hysteresis loop are also observed. $T_C$ increases to 40 K with the doping level increasing to 10%, but starts to decrease at the doping level of 20%. The saturation moment is suppressed from 0.5 $\mu_B$/Mn for $x = 0.10$ to 0.2 $\mu_B$/Mn for

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x = 0.20. More Mn doping also suppresses the coercive field from 324 mT for x = 0.05 to 111 mT for x = 0.20. No ferromagnetic ordering is observed for the doping level of 30\% Mn.

**Experimental methods.** – We synthesized (Sr$_3$La$_2$O$_5$)(Zn$_{1-x}$Mn$_x$)$_2$As$_2$ (x = 0.00, 0.05, 0.10, 0.20, 0.30) polycrystalline specimens by the solid-state reaction method. High-purity elements of La, Zn, Mn and As were mixed and heated to 900 °C in an evacuated silica tube to produce intermediate products LaAs, ZnAs and MnAs. They were then mixed with La$_2$O$_3$, SrO with nominal concentrations and slowly heated up to 1150 °C, and held for 50 hours before cooling to room temperature with turning off the furnace. The polycrystals were characterized by X-ray diffraction at room temperature and dc magnetization by the Quantum Design SQUID. The electrical resistance was measured on sintered pellets with the typical four-probe method.

**Results and discussion.** – We show the crystal structure of (Sr$_3$La$_2$O$_5$)(Zn$_{1-x}$Mn$_x$)$_2$As$_2$ and the X-ray diffraction patterns in fig. 1. Appreciable Bragg peaks from the parent compound (Sr$_3$La$_2$O$_5$)(Zn$_2$As$_2$) can be indexed by a layered tetragonal crystal structure (I$_4$/mmm), with a = 4.2612 Å and c = 27.675 Å. These lattice constants are close to a = 4.069 Å and c = 26.876 Å of a “32522” compound (Sr$_3$Sc$_2$O$_5$)(Fe$_2$As$_2$) [29], as well as a = 3.742 Å and c = 26.078 Å of the superconducting sample (Ca$_2$Al$_2$O$_5$)Fe$_2$As$_2$ [27]. We observed secondary phases of Zn$_3$As$_2$ and Sr$_3$As for x ≤ 0.10, as marked by the stars and the arrows in fig. 1. These impurities are non-magnetic, which will not affect the magnetic properties discussed in the following section.

In fig. 2, we show the electrical resistivity measured for (Sr$_3$La$_2$O$_5$)(Zn$_{1-x}$Mn$_x$)$_2$As$_2$ with x = 0.05, 0.10, 0.20, 0.30. The resistivity of all samples monotonically increases toward lower temperature. This type of behavior in Mn-doped specimens has been observed in the heavily doped region of (Ga$_{1-x}$Mn$_x$)$_2$As [6], as well as (La,Ba)(Zn$_{1-x}$Mn$_x$)AsO [9] and (Ba,K)(Zn$_{1-x}$Mn$_x$)$_2$As$_2$ [12] DMS polycrystals. It has been ascribed to the scattering of carriers by the magnetic fluctuations through exchange interactions in (Ga$_{1-x}$Mn$_x$)$_2$As [6]. We have also conducted the Hall effect measurements for the sample of (Sr$_3$La$_2$O$_5$)(Zn$_{0.05}$Mn$_{0.10}$)$_2$As$_2$. The large resistivity forbids us to accurately determine the carriers type, and a preliminary measurement indicates that the carrier density is in the order of 10$^{16}$ cm$^{-3}$. This carrier density is comparable to that of Li$_{1.1}$Zn$_{1-x}$Mn$_x$P [8] but it is 4 orders smaller than that of Li$_{1.1}$Zn$_{1-x}$Mn$_x$As [7].

In fig. 3, we show the zero-field-cooled (ZFC) and field-cooled (FC) measurements of the dc magnetization M of (Sr$_3$La$_2$O$_5$)(Zn$_{1-x}$Mn$_x$)$_2$As$_2$ for B$_{ext}$ = 0.1 T. For the doping of x = 0.05, we observe a strong increase of M at $T_C$ = 36 K, and the bifurcation of ZFC and FC curves below the temperature $T_J = 12.5$ K, where $T_J$ stands for the freezing temperature of individual spins or domain wall motion. The saturation moment at 2 K is 0.4 $\mu_B$/Mn. With the doping level increasing to x = 0.10, $T_C$ increases to ~ 40 K, and the saturation moment increases to 0.5 $\mu_B$/Mn. This indicates that additional Mn atoms raise the ferromagnetic ordering temperature. Both $T_C$ and the moment size start to decrease with further doping to x = 0.20. The ferromagnetic ordering disappears for the doping level of x = 0.30. This is primarily due to the competition of antiferromagnetic exchange interaction between spins from nearest-neighbor Mn sites. For 100% Mn substitution for Zn in “11”, “111”, “1111” and “122” compounds, the ending product is always an antiferromagnet with Neel temperature $T_N$ ~ 200–600 K, as displayed in table 1. Following this trend, we would expect a hypothetical antiferromagnet with “32522” structure,
in the low field of 1 mT (not shown), a higher magnetic field defined as the temperature where \( T_C \) shows a sharp upturn in the low field of 1 mT (not shown), a higher magnetic field suppresses the sharp upturn feature.

\( (\text{Sr}_3\text{La}_2\text{O}_5)\text{Mn}_2\text{As}_2 \). We fit the temperature dependence of \( M \) above \( T_C \) to a Curie-Weiss law. The effective paramagnetic moment is determined to be 5–6 \( \mu_B / \text{Mn} \), indicating that the valence of Mn ions is +2, and it is in a high spin state, as observed in other Mn-doped DMSs [5–7].

In fig. 4, we show the isothermal magnetization of \( (\text{Sr}_3\text{La}_2\text{O}_5)(\text{Zn}_{1-x}\text{Mn}_x)\text{As}_2 \). For \( x = 0.05 \), a parallelogram-shaped hysteresis loop with a coercive field of 324 mT is observed at 5 K. The coercive field continuously decreases to 111 mT with the doping level increasing to \( x = 0.20 \). The coercive fields are larger than \( \sim 5–10 \) mT of the cubic structural \( (\text{Ga}_{0.965}\text{Mn}_{0.035})\text{As} \) [1], \( \text{Li}_1(\text{Zn}_{0.97}\text{Mn}_{0.03})\text{As} \) [7], and \( \text{Li}_1(\text{Zn}_{0.97}\text{Mn}_{0.03})\text{P} \) [8], but smaller than \( \sim 1000 \) mT of the two-dimensional \((\text{La},\text{Ba})(\text{Zn,Mn})\text{AsO} \) [9] and \((\text{Ba},\text{K})(\text{Zn,Mn})_2\text{As}_2 \) [12]. We show the temperature dependence of the hysteresis loop for \( x = 0.10 \) in fig. 4(d). The coercive field decreases from \( 203 \) mT to \( 42 \) mT at 10 K and becomes zero at 60 K.

As we have discussed in ref. [9], the bifurcation of ZFC and FC curves and the hysteresis loops can be found not only in regular ferromagnets [30] but also in spin glasses [31]. The neutron scattering technique can resolve spatial spin correlations and decisively distinguish the two cases. Our neutron diffraction experiments on polycrystals \((\text{Ba},\text{K})_{0.3}(\text{Zn}_{0.9}\text{Mn}_{0.1})_2\text{As}_2 \) [12] and \((\text{La},\text{Sr})_{0.1}(\text{Zn}_{0.9}\text{Mn}_{0.1})\text{AsO} \) [32] were not able to decouple the magnetic and structural Bragg peaks since they superpose to each other. Single crystals are expected for high-resolution neutron scattering experiments. Nonetheless, the moment size is usually small for typical spin glasses, \( i.e. \), \( \sim 0.01 \mu_B / \text{Mn} \) for the II-VI \((\text{Zn,Mn})\text{Se} \) or other typical dilute alloy spin glasses [33–35]. Considering that the saturation moment size of our \((\text{Sr}_3\text{La}_2\text{O}_5)(\text{Zn}_{1-x}\text{Mn}_x)\text{As}_2 \) is as large as 0.5 \( \mu_B \), we tentatively assign it to a ferromagnetic ordering rather than a spin glass.

The ferromagnetism in various diluted magnetic conductors and oxides has been explained by several theoretical models, such as Zener’s model [36], percolation of bound magnetic polarons (BMPs) [37–39], and \( d-d \) double exchange due to hopping between transition metal \( d \) states [40]. In the case of \((\text{Sr}_3\text{La}_2\text{O}_5)(\text{Zn}_{1-x}\text{Mn}_x)\text{As}_2 \), the resistivity is large \( \sim 10^4 \Omega \text{mm} \) at 2 K and the carrier density is \( \sim 10^{19} \) cm\(^{-3} \). The low carrier density and the relative low \( T_C \) seems more amenable to the BMPs model.

Summary. – In summary, we report the synthesis and characterization of a bulk form diluted magnetic semiconductor \((\text{Sr}_3\text{La}_2\text{O}_5)(\text{Zn}_{1-x}\text{Mn}_x)\text{As}_2 \) with the Curie temperature \( \sim 40 \) K. The physical properties of this layered polycrystal are very similar to the recently discovered bulk form DMSs, \((\text{La},\text{Ba})(\text{Zn,Mn})\text{AsO} \) [9] and \((\text{Ba},\text{K})(\text{Zn,Mn})_2\text{As}_2 \) [12]. Summing up the previously reported “11”-type \((\text{Zn,Mn})\text{Se} \) (II-VI), “111”-type \( \text{Li}(\text{Zn,Mn})\text{As} \), “1111”-type \((\text{La},\text{Ba})(\text{Zn,Mn})\text{AsO} \) and “122”-type \((\text{Ba},\text{K})(\text{Zn,Mn})_2\text{As}_2 \), the “32522”-type \((\text{Sr}_3\text{La}_2\text{O}_5)(\text{Zn}_{1-x}\text{Mn}_x)\text{As}_2 \) system in the current study represents the fifth DMS system that has a direct counterpart with identical/similar structure in the Fe-based superconductors. As we stated in earlier papers [7,9,12], the common structure and excellent lattice matching between ferromagnetic, antiferromagnetic and superconducting systems make it possible to make junctions between these systems through the As layer.
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