The synthesis and characterization of 1111-type diluted magnetic semiconductors \((\text{La}_{1-x}\text{Sr}_x)(\text{Zn}_{1-x}\text{TM}_x)\text{AsO})\) (TM = Mn, Fe, Co)

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Abstract – The doping effect of Sr and transition metals Mn, Fe, Co into the direct-gap semiconductor \((\text{LaZnAsO})\) has been investigated. Our results indicate that the single phase \((\text{ZrCuSiAs})\)-type tetragonal crystal structure is preserved in \((\text{La}_{1-x}\text{Sr}_x)(\text{Zn}_{1-x}\text{TM}_x)\text{AsO})\) (TM = Mn, Fe, Co) with the doping level up to \(x = 0.1\). While the system remains semiconducting, doping with Sr and Mn results in ferromagnetic order with \(T_C \approx 30\,\text{K}\), and doping with Sr and Fe results in a spin-glass-like state below \(\approx 6\,\text{K}\) with a saturation moment of \(\approx 0.02\,\mu_B/\text{Fe}\), an order of magnitude smaller than the \(\approx 0.4\,\mu_B/\text{Mn}\) of Sr- and Mn-doped samples. The same type of magnetic state is observed neither for \((\text{ZnFe})\) substitution without carrier doping, nor for Sr- and Co-doped specimens.

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Introduction. – The successful fabrication of III-V (Ga,Mn)As ferromagnetic semiconductors has generated great interests in the research into diluted magnetic semiconductors (DMSs) [1]. Tremendous effort has been paid to improve the Curie temperature \(T_C\), and to understand the mechanism of the ferromagnetic ordering [2–4]. While much progress has been made, some inherent difficulties are encountered. For instance, the synthesis of bulk (Ga,Mn)As specimens is difficult, because the mismatch valences of Mn\(^{2+}\) atoms and Ga\(^{3+}\) atoms results in severely limited chemical solubility, i.e., Mn \(\leq 1\%\) [4]. The thin-film form specimens fabricated by non-equilibrium molecular-beam epitaxy (MBE), on the other hand, suffer from the difficulty to determine precisely the amount of Mn that substitutes ionic Ga, which donates a hole and act as a local moment, since some Mn impurities enter the interstitial sites where they act as double donors [4]. In diluted magnetic oxides (DMOs) such as Co-doped ZnO and TiO\(_2\), the ferromagnetism has been observed in (Zn,Co)O and (Ti,Co)O\(_2\) thin films, but not in bulk specimens, leaving behind controversial issues about the origin of ferromagnetic ordering [3].

In the family of II-VI semiconductors with the form of \(A^{II}\) \(B^{VI}\), where \(A^{II} = \text{Cd}^{2+}, \text{Zn}^{2+}, \text{Hg}^{2+}\), and \(B^{VI} = \text{Se}^{2-}, \text{Te}^{2-}, \text{S}^{2-}\), the substitution of magnetic Mn atoms for \(A^{II}\) can be very high in bulk specimens (for example, 86\% in (Zn,Mn)Te [5,6]) because Mn\(^{2+}\) is isovalent to A\(^{II}\), and the chemical solubility is much higher. The carrier density is usually low, i.e., \(\sim 10^{17}/\text{cm}^3\) [7,8], and difficult to be enhanced. The magnetic state in (A\(^{II}\),Mn) \(B^{VI}\) DMSs is usually a spin glass with the magnetic moment size in the order of 0.01 \(\mu_B\) per Mn atom or even smaller [6,9].

Seeking for bulk DMS or DMO materials that have higher chemical solubility of magnetic atoms and whose carrier density can be controlled may be helpful to improve \(T_C\) and understand the mechanism of the ferromagnetic ordering. Recently, through the substitution of Mn for Zn and introducing extra Li atoms in the II-VI direct-gap semiconductor LiZnAs, Deng et al. successfully...
synthesized a bulk Li(Zn$_{1-x}$Mn$_x$)As DMS with $T_C$ as high as $\sim$50 K [10]. 1-II-V Li(Zn,Mn)As DMS has many similarities and differences to both III-V (Ga,Mn)As and II-VI (A$^{II}$,Mn) B$^{VI}$ DMSs. The parent semiconductor LiZnAs has a direct band gap of $\sim$1.6 eV [11–14], which is comparable to that of GaAs (1.42 eV) but much smaller than 2.80 eV of ZnSe [6]; LiZnAs has a cubic structure, analogous to the Zincblende structure of GaAs. Furthermore, Mn$^{2+}$ atoms are expected to replace the isovalent Zn$^{2+}$, which could overcome the chemical solubility limit encountered in III-V GaAs and InAs systems. Another advantage is that in Li(Zn,Mn)As, the concentration of Li can be used to precisely control the doping characteristics, allowing the possibility of either p-type or n-type doping. This contrasts with (Ga,Mn)As which only permits p-type carriers, and (A$^{II}$,Mn) B$^{VI}$ DMSs whose carrier density is difficult to control.

More recently, two types of bulk DMSs with a two-dimensional crystal structure have been successfully fabricated. Firstly, Zhao et al. doped Mn and K into a “122”-type semiconductor BaZn$_2$As$_2$, and fabricated a DMS with $T_C$ $\sim$ 180 K [15], which is already comparable to the record $T_C$ of 190 K of (Ga,Mn)As [16]. Secondly, Ding et al. doped Ba and Mn atoms into a “1111”-type semiconductor LaZnAsO with a direct gap of $\sim$1.5 eV, and synthesized another DMS with $T_C$ $\sim$ 40 K [17]. The saturation moment size in (La,Sr)(Zn,Mn)AsO DMS is as large as 1 $\mu_B$/Mn, indicating the strong ferromagnetic correlation. In A$^{II}$ B$^{VI}$, the substitution of Fe$^{2+}$ and Co$^{2+}$ for A$^{II}$ has also induced the same type of spin glass state as that of Mn$^{2+}$ [6,18]. It will be interesting to investigate whether Fe$^{2+}$ and Co$^{2+}$ substitution for Mn$^{2+}$ in the two-dimensional LaZnAsO is similar or different to the case of Mn$^{2+}$ doping.

In this paper, we report the synthesis and characterization of (La$_{1-x}$Sr$_x$)(Zn$_{1-x}$TM$_x$)AsO (TM = Mn, Fe, Co) by the solid-state reaction method. The intermediate products LaAs and TMAs (TM = Mn, Fe, Co) were prior synthesized by the solid-state reaction method with La, TM, As at 900°C in an evacuated silica tube for 10 hours. The mixture of LaAs, TMAs, ZnO and SrO with nominal concentration was heated up to 1150°C slowly and held for 40 hours in an evacuated silica tube before cooling down at a rate 10°C/h to room temperature. The polycrystals were characterized by X-ray diffraction carried out at room temperature and dc magnetization by Quantum Design SQUID. The electrical resistance was measured on sintered pellets with the typical four-probe method.

**Results and discussions.**

Results of (La$_{1-x}$Sr$_x$)(Zn$_{1-x}$Fe$_x$)AsO. We show the crystal structure and X-ray diffraction pattern of (La$_{1-x}$Sr$_x$)(Zn$_{1-x}$Fe$_x$)AsO specimens in fig. 1. The peaks of LaZnAsO can be well indexed by a ZrCuSiAs-type tetragonal crystal structure with a $P4/nnm$ space group. We reproduce the Rietveld analysis of the parent compound LaZnAsO from ref. [17] and show it in fig. 1(d). Sr and Fe doping does not change the crystal structure and the single phase is preserved with the concentration up to $x = 0.10$. The non-magnetic impurity phases of SrO$_2$ and Zn$_3$(AsO$_4$)$_2$ emerge at the level of $x = 0.12$ and 0.15, as marked by the stars in fig. 1(c). The a-axis lattice parameters remain constant and the c-axis lattice parameters monotonically increase with Sr and Fe doping up to $x = 0.15$, indicating the successful solid solution of (La,Sr) and (Zn,Fe).

In fig. 2, we show the electrical resistivity measurement for (La$_{1-x}$Sr$_x$)(Zn$_{1-x}$Fe$_x$)AsO. The resistivity increases with decreasing temperature down to 4 K for all doping levels, indicating the semiconducting behavior. The temperature dependence of resistivity are similar to the case of Sr- and Mn-doped (La$_{1-x}$Sr$_x$)(Cu$_{0.925}$Mn$_{0.075}$)SO specimens [19], but contrast to the case of Mn-doped CaNiGe and CaNiGeH where a Kondo-like behavior is observed [20]. The magnitude of resistivity decrease with higher doping levels, indicating more carriers are introduced. We do not observe the insulator-to-metal transition with the doping level up to 15%, as opposed to the observation in BaZn$_2$As$_2$ [15] and BaMn$_2$As$_2$ [21], where 5% potassium doping will induce a metal-insulator transition. In our case, the introduction of Mn atoms is to help the localization of the hole carriers through the disorder in the quasi-two-dimensional ZnAs layers. We also point out that the temperature dependence of resistivity for the $x = 0.10$ sample is apparently different to other doping levels, and the reasons are unknown. Effort to generate single crystals is underway to clarify this issue.

In fig. 3(a), we show the dc-magnetization of the La(Zn$_{0.9}$Fe$_{0.1}$)AsO specimen under zero-field-cooled (ZFC) and field-cooled (FC) condition for $B_{ext} = 1000$ Oe. We do not observe any anomaly or splitting of ZFC and FC curves in the measured temperature range. The curve
LaZnAsO with Rietveld analyses reproduced from fig. 1(d) of the reference [17].

LaZnAsO with Rietveld analyses reproduced from fig. 1(d) of the reference [17].

Ref. [17].

The situation is similar to the case of doping Mn atoms alone does not induce any type of magnetic ordering. We plot the concentration dependence of both coercive field and \( T_f \) in fig. 3(f). Both the coercive field and \( T_f \) show roughly the same concentration dependence. They increase with the doping level up to \( x = 0.10 \), and then decrease with more Sr and Fe doping. The decrease of the coercive field and \( T_f \) is likely due to the competition of antiferromagnetic exchange interaction with increasing

\[ x = 0.03, \] ZFC and FC curves are almost superposed to each other. Starting from \( x = 0.05 \), ZFC and FC curves display a clear splitting at \( T \sim 4.5 \text{ K} \). We show the low-temperature data in the range between 2 \( \text{K} \) and 15 \( \text{K} \) in fig. 3(d) to show the bifurcation of ZFC and FC curves clearly. We define the temperature where ZFC and FC curves split as \( T_f \). \( T_f \) increase to 6.5 \( \text{K} \) for \( x = 0.10 \) and then decrease to 5 \( \text{K} \) for \( x = 0.15 \). In constrast to the case of \((\text{La},\text{Ba})(\text{Zn},\text{Mn})\text{AsO}\), we do not observe a strong enhancement of \( M \) before the bifurcation of the ZFC and FC curves that corresponds to the appearance of a strong ferromagnetic correlation.

We also measured the isothermal magnetization for each doping level and show the results in fig. 3(c). S-shape hysteresis loops are observed for all doping levels. The magnetization is not saturated at an external field as high as 5 tesla. We show the hysteresis loops in the small-field region in fig. 3(e), and the loops with well-defined coercive fields of 10–220 Oe are clearly seen. The history-dependence behavior and the small moment size implies that a spin-glass–like state has developed. In general, the spin freezing temperature \( T_f \) of a typical spin glass has DC field dependence, and frequency dependence in the AC susceptibility measurement. In the case of \((\text{La},\text{Sr})(\text{Zn},\text{Fe})\text{AsO}\), the cusp shape of the ZFC magnetization is not clear, and the measurement of AC frequency and/or DC field dependence of \( T_f \) are difficult.

In fig. 3(b), we show the dc-magnetization curve of \( \text{La}(\text{Zn}_{0.9}\text{Mn}_{0.1})\text{AsO} \) in fig. 4(a) for convenience.

The magnitude of the saturation moment at 2 \( \text{K} \) is about an order of magnitude larger than that of \( \text{La}(\text{Zn}_{0.9}\text{Fe}_{0.1})\text{AsO} \), i.e., \( \sim 0.07 \mu_B/\text{Fe} \) for \( x = 0.03 \). For

Fig. 1: (Color online) (a) Crystal structures of LaZnAsO (\( P4/mnm \)). (b) Lattice constants for the a-axis (red filled circle) and c-axis (blue filled square) of \( (\text{La}_{1-x}\text{Sr}_x)(\text{Zn}_{1-x}\text{Fe}_x)\text{AsO} \) for various doping levels \( x \). (c) X-ray diffraction pattern of \( (\text{La}_{1-x}\text{Sr}_x)(\text{Zn}_{1-x}\text{Fe}_x)\text{AsO} \) with \( x = 0.00, 0.03, 0.05, 0.10, 0.12, 0.15 \). Traces of impurity SrO and \( \text{Zn}_3(\text{AsO}_4)_2 \) are marked by stars (*) for \( x \geq 0.10 \). (d) X-ray diffraction pattern of LaZnAsO with Rietveld analyses reproduced from fig. 1(d) of ref. [17].

can be well fitted by a Curie-Weiss function with \( C = 0.016 \mu_B K/\text{Fe} \) and \( \theta = 1.35 \text{ K} \), suggesting a paramagnetic ground state. This indicates that doping 10% Fe atoms alone does not induce any type of magnetic ordering. The situation is similar to the case of doping Mn atoms alone into LaZnAsO where a paramagnetic ground state is observed as well. We reproduce the dc-magnetization curve of \( \text{La}(\text{Zn}_{0.9}\text{Mn}_{0.1})\text{AsO} \) in fig. 4(a) for convenience.

In fig. 3(b), we show the dc-magnetization curve of \( (\text{La}_{1-x}\text{Sr}_x)(\text{Zn}_{1-x}\text{Fe}_x)\text{AsO} \) specimens with \( x = 0.03, 0.05, 0.10, 0.12, 0.15 \) under ZFC and FC condition for \( B_{zfc} = 1000 \text{ Oe} \). Once Sr atoms substitute for La and the carriers are introduced into the compound, the ground state becomes different. The magnitude of the saturation moment at 2 \( \text{K} \) is about an order of magnitude larger than that of \( \text{La}(\text{Zn}_{0.9}\text{Fe}_{0.1})\text{AsO}, \) i.e., \( \sim 0.07 \mu_B/\text{Fe} \) for \( x = 0.03 \). For

Fig. 2: (Color online) Electrical resistivity for \((\text{La}_{1-x}\text{Sr}_x)(\text{Zn}_{1-x}\text{Mn}_x)\text{AsO} \) with \( x = 0.00, 0.03, 0.05, 0.10, 0.12, 0.15 \).
a well-defined coercive field of 0.158 Tesla exists, as shown in fig. 4(c); 4) The hysteresis loop closes and saturates to ~1.4 µB/Mn at an external field of ~2 Tesla. The overall features are similar to the case of (La,Ba) (Zn,Mn)AsO DMS except that the ferromagnetic transition is much sharper and the coercive field is much smaller in the current case. A possible reason is that the ionic radius of Sr2+ (0.113 nm) is much closer to that of La3+ (0.106 nm), and its chemical solubility is better than in the case of Ba2+ (0.135 nm). The nature of T_f has been determined from the zero-field and longitudinal field µSR measurement of (La,Ba)(Zn,Mn)AsO. A peak of the muon spin relaxation rate 1/τ_f is observed at T_f ~ 15 K - 20 K. This indicates that the dynamic slowing-down of spin fluctuations approaches a “static freezing” of either individual spins or domain wall motion, as shown in fig. 3(f) of ref. [17]. The “static freezing” temperature T_f determined from the µSR data agrees well with that of the magnetic susceptibility data measured by SQUID.

We have also conducted neutron diffraction measurement of (La0.9Sr0.1)(Zn0.9Mn0.1)AsO to investigate the nature of the ferromagnetic ordering. Unfortunately the average moment size is only ~0.04 µB/Mn in this diluted magnetic system, and it is difficult to decouple the magnetic and structural Bragg peaks even at 6 K.
nonetheless, we find that the powder diffraction pattern agrees well with the structure found by X-ray shown in fig. 1. No structure phase transition is observed in the temperature range between 6 K and 300 K [26].

Results of (La$_{0.9}$Sr$_{0.1}$)(Zn$_{0.9}$Co$_{0.1}$)AsO. Since the saturation moment size of the Fe-doped sample is an order of magnitude smaller than that of Mn-doped samples, it will be interesting to see if Sr and Co doping can induce magnetic ordering in LaZnAsO. We show the demagnetization curve of the (La$_{0.9}$Sr$_{0.1}$)(Zn$_{0.9}$Co$_{0.1}$)AsO specimen in fig. 5. In the measured temperature range, we do not observe any anomaly or a spin-glass-like state as that of the (La$_{0.9}$Sr$_{0.1}$)(Zn$_{0.9}$Fe$_{0.1}$)AsO sample. The magnetization at 2 K in 1000 Oe is 0.0028 $\mu_B$/Co. We fit the data by a Curie-Weiss law and obtain $C = 0.01 \mu_B K$/Co and $\theta = 1.62 K$ that are determined from free parameter fitting. (b) log($M$) vs. log($T$) for (La$_{0.9}$Sr$_{0.1}$)(Zn$_{0.9}$Co$_{0.1}$)AsO in the temperature range between 2 K and 100 K; the solid line is a linear fit to the data with a slope of $-0.78$; the star marks the signal arising from the incident oxygen during the measurement.

Summary and discussions. – To summarize, we report the successful synthesis of polycrystalline semiconductors (La$_{1-x}$Sr$_x$)(Zn$_{1-x}$TM$_x$)AsO (TM = Mn, Fe, Co). We demonstrate that doping Sr and Mn results in a ferromagnetic order with $T_C \sim 30 K$, and doping Sr and Fe results in a spin-glass-like state below $T_F \sim 6 K$. The saturation moment decreases from $\sim 0.36 \mu_B$/Mn to $\sim 0.02 \mu_B$/Fe and to $\sim 0.003 \mu_B$/Co for the 10% doping level, along with the ground state from a ferromagnetic ordered state to a spin-glass-like state, and to a paramagnetic state. The concentration of dopant and the random spatial distribution of TM atoms are expected to be the same in each system, but the magnetization at 2 K decreases by two orders from Mn to Fe to Co.

In passing, ferromagnetism has been found in Fe-doped SnO$_2$ ($T_C \sim 610 K$) [29] and Co-doped SnO$_2$ ($T_C \sim 650 K$) thin films [30]. Mössbauer spectra show that the iron is all-high-spin state Fe$^{3+}$ with a net ferromagnetic moment 1.8 $\mu_B$/Fe in Sn$_{0.95}$Fe$_{0.05}$O$_2$. In II-VI DMS, both iron and cobalt are supposed to be in the valence of 2+ [6,31]. In the case of (La,Sr)(Zn,Fe)AsO, suppressing that Fe is in the valence of 3+ (or $\geq 2$), we would expect an electron doping once Fe$^{3+}$ substitutes for Zn$^{2+}$ in La(Zn$_{0.9}$Fe$_{0.1}$)AsO, but no ferromagnetic ordering is observed in La(Zn$_{0.9}$Fe$_{0.1}$)AsO, as shown in fig. 3(a). We fit the magnetization curve above $T_f$ and find that the effective moment is 2.37 $\mu_B$/Fe for 10% Sr- and Fe-doped samples. However, we cannot estimate either the valence of Fe ions or the spin state due to the magnetic frustration and/or the antiferromagnetic exchange interaction in (La,Sr)(Zn,Fe)AsO. A naive expectation is that the valence state of iron and cobalt is more likely the case of the (La,Sr)(Zn,Fe)AsO. We demonstrate that doping Sr and Mn results in a ferromagnetic–like state below $T_C \sim 6 K$.

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**Fig. 5:** (a) Temperature dependence of magnetization $M$ for (La$_{0.9}$Sr$_{0.1}$)(Zn$_{0.9}$Co$_{0.1}$)AsO, the solid line represents the Curie-Weiss law $M = C/(T - \theta)$ with $C = 0.01 \mu_B K$/Co and $\theta = 1.62 K$ that are determined from free parameter fitting. (b) log($M$) vs. log($T$) for (La$_{0.9}$Sr$_{0.1}$)(Zn$_{0.9}$Co$_{0.1}$)AsO in the temperature range between 2 K and 100 K; the solid line is a linear fit to the data with a slope of $-0.78$; the star marks the signal arising from the incident oxygen during the measurement.
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