Spin reorientation and Ce-Mn coupling in antiferromagnetic oxypnictide CeMnAsO

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Structure and magnetic properties of high-quality polycrystalline CeMnAsO, a parent compound of the “1111”-type oxypnictides, have been investigated using neutron powder diffraction and magnetization measurements. We find that CeMnAsO undergoes a C-type antiferromagnetic order with Mn2+(S = 5/2) moments pointing along the c-axis below a relatively high Néel temperature of TN = 345 K. Below TNR = 35 K, two instantaneous transitions occur where the Mn moments reorient from the c-axis to the ab-plane preserving the C-type magnetic order, and Ce moments undergo long-range AFM ordering with antiparallel moments pointing in the ab-plane. Another transition to a noncollinear magnetic structure occurs below 7 K. The ordered moments of Mn and Ce at 2 K are 3.32(4)μB and 0.81(4)μB, respectively. We find that CeMnAsO primarily falls into the category of a local-moment antiferromagnetic insulator in which the nearest-neighbor interaction (J1) is dominant with J2 < J1/2 in the context of the J1 − J2 − J3 model. The spin reorientation transition driven by the coupling between rare earth Ce and transition metal seems to be common to Mn, Fe and Cr ions, but not to Co and Ni ions in the iso-structural oxypnictides. A schematic illustration of magnetic structures in Mn and Ce sublattices in CeMnAsO is proposed.

INTRODUCTION

The discovery of superconductivity (SC) in LaFeAsO1−yFy [1] with square lattice of Fe2+ has triggered renewed interest in superconductivity and also in itinerant magnetism in general. In the Fe-based “1111” and “122” pnictides, the emergence of superconductivity is accompanied by the suppression of the stripe-like antiferromagnetic (AFM) ordering of Fe2+ ions and a tetragonal(T)-orthorhombic(O) structural transition[2]. It has also been suggested that AFM/structural fluctuations may be the driving forces for superconductivity[3]. In view of the prominent role of magnetism in driving superconductivity in the Fe-based pnictides, in particular by doping with transition metal ions, systematic investigations of the magnetic and structural properties of the iso-structural “1111” and “122” parent pnictides involving the square lattice of other transition metals are called for[4]. It fact, the various transition metals influence subtle and specific structural, magnetic, electronic properties that are pivotal to understanding how the magnetism is related to the SC state[5]. Furthermore, compared with the parent “122” compounds, the parent “1111” oxypnictides RTAsO (R = magnetic and non-magnetic rare earths, T = transition metals Fe, Co, Ni, Mn, etc) are more intriguing as they offer the possibility to tweak an additional interplay of coupling between the rare-earth R and the transition-metal ions.

CeFeAsO is an itinerant poor metal with a T-O structural transition at TS = 151 K followed by stripe-like AFM order of Fe at TN = 145 K[6]. Recent μSR studies[7] have indicated relatively strong coupling between the rare earth Ce and Fe, and further neutron and X-ray scattering studies have shown the coupling leads to a gradual Fe spin-reorientation at low temperatures[8]. By contrast, in the heavy-fermion metal CeNiAsO[9], Ni does not order magnetically but two successive AFM transitions associated with Ce ions are observed[10]. In CeCoAsO, a ferromagnetic ordering is found below ~75 K with no indication for Ce ordering at lower temperatures[11]. To date, little attention has been paid to CeMnAsO for which magnetization and heat capacity measurements indicate that Mn moments order above room temperature and a first-order magnetic transition emerges at ~35 K, possibly related to a Mn spin reorientation[12]. It has also been proposed that the Ce spins do not undergo long range order but are parasitically induced to order below ~35 K[12]. However, the AFM Néel temperature TN, actual magnetic structures, the values of the ordered moments, and the interplay between Ce and Mn in CeMnAsO have not been determined. Here, we report neutron diffraction, and magnetization results on CeMnAsO to answer these questions and also to critically compare the structure and magnetism with related pnictides.

EXPERIMENTAL DETAILS

Previous reports on the synthesis of CeMnAsO used CeAs and Mn2O3 [12] and added excess Ti as an oxygen getter which resulted in the formation of a secondary phase. In the present study, MnO and CeAs as starting materials were mixed thoroughly in stoichiometric proportions. (CeAs was prepared firstly by reacting Ce and As powders at 600 °C for 35 h and then at 950 °C for 8 h).
The mixed powder was sealed in an evacuated tantalum tube and sintered at 1150 °C for 40 h. A single-phase polycrystalline CeMnAsO powder was then obtained and characterized by x-ray and neutron diffraction methods.

Neutron powder diffraction (NPD) measurements on \( \approx 4 \) g CeMnAsO sample were conducted on the HB1A triple-axis spectrometer with a fixed-incident-energy 14.6 meV (located at the high flux isotope reactor, HFIR, at the Oak Ridge National Laboratory, USA). The data between \( 2 < T < 300 \) K were collected using an orange cryostat and a high temperature furnace was used for the measurements between \( 300 < T < 420 \) K. All the neutron diffraction data were analyzed using Rietveld refinement program Fullprof suite[13].

The temperature/field dependence of the magnetization was carried out in a superconducting quantum interference device (Quantum Design MPMS-7S, SQUID) magnetometer.

RESULTS AND DISCUSSION

A. Crystalline structure

Neutron powder diffraction pattern at 420 K is shown in Fig. 1 indicating the purity of the material with no indication of a secondary phase in CeMnAsO, consistent with our x-ray diffraction measurement at room temperature (not shown here). Rietveld analysis confirms the tetragonal ZrCuSiAs-type structure with space group \( P4/nmm \), as illustrated in the right panel of Fig. 1. Similar to the tetragonal structure in \( RFeAsO \) (\( R \) is rare earth element)[8] or LaMnAsO[14], the structure of CeMnAsO consists of MnAs and CeO layers where the Mn\(^{2+}\) ions form a square lattice. Our neutron diffraction results show no change in the crystal structure of this compound down to 2 K. The refined atomic positions, lattice constants, and volume of CeMnAsO at 420 K and ground temperature 2 K are summarized in Table 1.

B. Multiple magnetic transitions and field-induced metamagnetic transition revealed from magnetization measurements

The temperature dependence of the zero-field-cooled (ZFC) and field-cooled (FC) magnetization in Fig. 2 (a) shows a clear magnetic transition at 35 K emphasized by a single peak in the first derivative of magnetization with no indication of additional anomaly up to 370 K. The anomaly at 35 K has been previously attributed to a spin reorientation (SR) transition of Mn.[12] Our susceptibility measurements show that this magnetic transition is not shifted by external magnetic fields up to 5 T in accordance with typical behavior of a spin reorientation transition (the transition temperature at 35 K is labeled \( T_{SR} \) hereafter). The thermal hysteresis of the magnetization below \( T_{SR} \) is indicative of the first-order nature of the transition. Below 7 K, both ZFC and FC magnetization decrease implying the emergence of another magnetic transition. We point out that such anomalous decrease below 7 K was not observed by Tsukamoto et al. because of the influence of a secondary phase as mentioned in Ref. 12. Interestingly, we do not observe a clear anomaly in the magnetization in the temperature range 35 – 370 K. However, the neutron measurements of the (100) and (101) magnetic Bragg peaks shown in Fig. 3 exhibit a sharp increase in the integrated intensity below 345 K, which we identify as the AFM transition temperature \( T_{N} \) of the Mn sublattice. We note that a weak and broader (100) Bragg peak persists above \( T_{N} \) with a linewidth that increases with temperature indicating the presence of short-range ordered Mn spins above \( T_{N} \). This suggests the existence of strong spin fluctuations above \( T_{N} \) that tend to wash out any anomaly in the susceptibility at \( T_{N} \) even in its first derivative with respect to temperature. The absence or weak peak in the derivative of the susceptibility is characteristic of the two-dimensional nature of the Mn magnetic system with a strong inplane coupling that gives rise to short range fluctuating magnetic order, as has been found in other systems[15,16].

Figure 2 (b) shows magnetic field dependence of the magnetization at different temperatures. Whereas the magnetization at room-temperature (RT) shows a very weak hysteresis, typical of AFM behavior, i.e., linear \( M \) versus \( H \) curve is observed in \( T_{SR} < T < T_{N} \). Below \( T_{SR} \), the magnetization first rises linearly at low fields indicative of an AFM behavior, with a weak jump in the slope at approximately 1.5 T, indicating a possible field-induced meta-magnetic transition. However, the magnetization is far from saturation even at 5 T, indicating that the magnetic structure under magnetic field is mostly preserved, and that the external magnetic field may induce a transformation to canted AFM structures below \( T_{SR} \).

| T (K) | Atom          | Atomic position | c(Å)   | V(Å\(^3\)) |
|-------|---------------|-----------------|--------|----------|
| 420   | Ce            | \( z = 0.1263(6) \) | 4.1032(2) | 9.0038(3) | 151.594(8) |
| 2     | Ce            | \( z = 0.6696(4) \) | 4.0837(3) | 8.9567(4) | 149.370(6) |
| As    | \( z = 0.6720(5) \) | | | |
Figure 1: (color online) Rietveld refinement fit to neutron diffraction pattern at 420 K (left) and a graphic representation of the crystal structure (right) for CeMnAsO using the best fit parameters listed in Table I. The observed data and the fit are indicated by the open circles and solid lines, respectively. The difference curve is shown at the bottom. The vertical bars mark the positions of Bragg reflections for the phases of CeMnAsO (up) and Al sample holder (below).

C. Evidence of Mn spin reorientation and long-range Ce spin orderings determined by NPD measurements

A comparison of the neutron diffraction patterns at different temperature windows \( T > T_N \) (420 K), \( T_{SR} < T < T_N \) (RT), \( 7K < T < T_{SR} \) (10K) is shown in Fig. 3 (a) (nuclear and magnetic Bragg reflections are labeled N and M, respectively). At RT, whereas the (100) Bragg peak is purely magnetic, the (101) and (102) peaks have nuclear and magnetic contributions due to the Mn ordering. At 10 K (below \( T_{SR} \)), the intensity of (100) peak decreases dramatically whereas the intensities of the (101) and (102) increase, evidence for a change in magnetic structure. The magnetic contribution to (002) nuclear peak below \( T_N \) is negligible, however it increases below \( T_{SR} \) and peaks at \( \approx 7 \) K (see inset of Fig. 3 (c)). As shown below by our analysis, the intensities of (100) and (002) peaks reflect the order parameters of Mn and Ce moments, respectively. All the magnetic reflections can be indexed on the high temperature nuclear (chemical) unit cell with a magnetic propagation vector \( k = (0,0,0) \). SARAH representational analysis program \[17\] is used to derive the symmetry allowed magnetic structures. The decomposition of the magnetic representation (\( \Gamma_{Mag} \)) into the irreducible representations is \( \Gamma_1 + \Gamma_6 + \Gamma_9 + \Gamma_{10} \) and \( \Gamma_1 + \Gamma_2 + \Gamma_9 + \Gamma_{10} \) for Mn sites and Ce sites, respectively. The symmetry allowed basis vectors are summarized in Table \[11\] There are two FM \( (\Gamma_1^2) \) and three AFM \( (\Gamma_1^1, \Gamma_6^1 \text{ and } \Gamma_{10}^1) \) solutions. But the two FM solutions can be discarded at all temperatures as there is no FM contribution to the nuclear Bragg reflections in our neutron diffraction patterns consistent with the magnetization measurements below \( T_N \). Thus, only the three AFM solutions are considered for the data refinement to obtain the magnetic structures at different temperature windows.

Rietveld refinement fits to neutron diffraction patterns and the graphic representation of the determined magnetic structures of CeMnAsO at different temperatures are shown in Fig. 4. In \( T_{SR} < T < T_N \), there is no evidence for Ce moment ordering and the neutron diffraction pattern is best fitted using \( \Gamma_1^1 \) model, i.e., the Mn spins are antiparallel at Mn1 and Mn2 sites, forming a nearest-neighbor antiferromagnetic alignment in \( ab \) plane and ferromagnetic stacking along \( c \)-axis (C-type AFM order) with the Mn moments along \( c \)-axis, as shown in Fig. 4 (a) at 45 K, which indicates that the preferred orientation of Mn\(^{2+}\) moments (\( S = 5/2, L = 0 \)) is along the \( c \)-axis. As the temperature decreases to \( T_{SR} \), the magnetic structure is preserved but the Mn magnetic moment gradually increases. We find that the magnitude of the Mn moment at RT has a relatively large value of 2.29(3) \( \mu_B \) and further increases to 2.78(2) \( \mu_B \) at 45 K.

In the temperature range \( 7K < T < T_{SR} \), the refinement of the neutron diffraction patterns are not satisfactory with the assumption of Mn ordering only and the ordering of Ce magnetic moments is required to obtain good agreement with the data. Trial refinements assuming a linear combination of the \( \Gamma_1^1 \) of Mn sites and \( \Gamma_1^2 \) of
Figure 3: (color online) (a) Comparison of the neutron diffraction patterns at 420 K, RT and 10 K. The inset shows the temperature dependence of the intensities of the (101) and (110) peaks. (b) Temperature dependence of the integrated intensity of (100) peak. The inset shows the temperature dependence of the linewidth of (100) peak. (c) Temperature dependence of the integrated intensity of (002) peak. The inset shows a zoomed-in view at low temperatures below 80 K with a peak at $T = 7$ K that we attribute to additional magnetic transition in the Ce sublattice.

Table II: The symmetry-allowed basis vectors $[m_x, m_y, m_z]$ for the space group $P4/nmm$ with $k=(0,0,0)$ in CeMnAsO. Mn1: (0.75, 0.25, 0.5), Mn2: (0.25, 0.75, 0.5), Ce1: (0.25, 0.25, 0.126) and Ce2: (0.75, 0.75, 0.874).

| Atom | $\Gamma_2$ | $\Gamma_3^\dagger$ | $\Gamma_6^\dagger$ | $\Gamma_9$ | $\Gamma_{10}^\dagger$ |
|------|------------|----------------|----------------|----------|----------------|
| Mn1  | [0 0 m_z]  | [0 0 m_z]  | $[m_x, m_y, 0]$ | $[m_x, m_y, 0]$ | $[m_x, m_y, 0]$ |
| Mn2  | [0 0 m_z]  | [0 0 m_z]  | $[m_x, m_y, 0]$ | $[-m_x, -m_y, 0]$ | $[m_x, m_y, 0]$ |
| Ce1  | [0 0 m_z]  | [0 0 m_z]  | $[m_x, m_y, 0]$ | $[m_x, m_y, 0]$ | $[-m_x, -m_y, 0]$ |
| Ce2  | [0 0 m_z]  | [0 0 m_z]  | $[m_x, m_y, 0]$ | $[-m_x, -m_y, 0]$ | $[m_x, m_y, 0]$ |

Ce sites, $\Gamma_{10}^\dagger$ of Mn sites and $\Gamma_3^\dagger$ of Ce sites, or $\Gamma_6^\dagger$ of Mn sites and $\Gamma_{10}^\dagger$ of Ce sites did not fit the data well. A satisfactory fit to the neutron diffraction patterns below $T_{SR}$ is obtained by using $\Gamma_{10}^\dagger$, i.e., antiparallel Ce spins at Ce1 and Ce2 sites and antiparallel Mn spins at Mn1 and Mn2 sites with ordered Mn and Ce moments in the $ab$ plane. Thus, nearest-neighbor Mn spins align antiferromagnetically in the $ab$ plane and ferromagnetically along $c$ (still C-type) but the ordered Mn magnetic moments reorient to the $ab$ plane, whereas the Ce spins align antiferromagnetically along $c$, similar to the magnetic structure in PrMnSbO [18] and NdMnAsO [19] below its SR transition. It is impossible to determine the absolute angle between Mn (or Ce) moments with respect to $a$ axis in $ab$ plane due to the tetragonal nature of the system, nevertheless we show both Mn and Ce moments along $a$ axis in Fig. 4 (b). Note that there is no significant broadening of the (002) peak below/above $T_{SR}$, as shown in the temperature dependence of its linewidth (see the inset of Fig. 4 (c)) indicating long-range ordered Ce below $T < T_{SR}$.

Below $T_{SR}$, the intensity of the (002) reflection increases slightly peaking at $\sim 7$ K, consistent with the peak in the magnetization shown in Fig. 2 (a) confirming there is another magnetic transition in the Ce sublattice. Furthermore, the good refinement of the neutron diffraction pattern at 2 K is still obtained by using $\Gamma_{10}^\dagger$ model within uncertainties. Since the refinement using $\Gamma_{10}^\dagger$ model of Ce1 and Ce2 spins requires antiparallel and confined spins to the $ab$ plane, it is very likely that the transition observed at 7 K is due to a finite angle between Ce spins with respect to the Mn spins, forming a noncollinear magnetic structure between Ce and Mn moments as theoretically predicted [20]. However, we emphasize that our powder data is not sufficiently sensitive to determine the accurate relative angle between Ce and Mn moments. The quality of the refinement to the 2 K data has the tendency to be slightly improved when this angle is increased from 0° to $\sim 20°$. The best re-

Figure 4: (color online) Rietveld refinement fits to neutron diffraction patterns and the graphic representations of the determined magnetic structures of CeMnAsO at (a) 45 K, (b) 10 K and (c) 2 K. The observed data and the fit are indicated by the open circles and solid lines, respectively. The difference curve is shown at the bottom. The vertical bars mark the positions of Bragg reflections for the nuclear phase (up) and magnetic phase (down) in CeMnAsO. The middle vertical bars in (c) mark the positions of the phase of Al sample holder.
The ordered Mn moments 3.32(4) $\mu_B$ at 2 K compared to the 5 $\mu_B$ expected from a localized moment indicate that in the spectrum of itinerant versus local-moment AFM, CeMnAsO tends to be the latter (i.e., local-moment AFM)\cite{20}. This is different from the itinerant CeFeAsO with a much lower ordered Fe moment of $\sim 0.9\mu_B$ compared to the 4 $\mu_B$ expected from a localized moment\cite{21}. The inplane checker-board-like AFM structure of the C-type order in CeMnAsO in $T < T_N$ suggests that the NN interaction $J_1$ plays a dominant role whereas inplane next-nearest-neighbor (NNN) interaction $J_2$ is very weak or negligible. Thus, in the context of $J_3 - J_2 - J_x$ model\cite{22}, we conclude that $J_1 > 0$, $J_2 < J_1/2$ with negligible spin frustration. This is in sharp contrast to CeFeAsO for which the effective NNN interaction $J_2 > J_1/2$ is necessary to stabilize the stripe-like AFM ordering with the ordered moment in the $ab$ plane. Note that the preferred orientation of Mn is along the $c$ axis in CeMnAsO, in contrast to the preferred orientation of Fe in $ab$ plane in CeFeAsO.

Another significant difference between CeMnAsO and CeFeAsO is that the T-O structural transition observed above $T_N$ in CeFeAsO is absent in CeMnAsO. It is generally accepted that the T-O structural transition in CeFeAsO and other “1111” Fe-based pnictides has the magnetic origin and driven by the magnetic interactions or nematic phase\cite{24, 25, 26}. The stripe-like AFM structure in “1111” Fe-based pnictides can be separated into two Néel sublattices and each of them is defined by NNN Fe spins within the basal plane.\cite{27} Due to $J_2 > J_1/2$, there is a strong frustration between these two sublattices and the orthorhombic distortion is induced to reduce the frustration and therefore lower the total energy (magnetic and elastic energy).\cite{24, 25, 27} The absence of magnetic frustration due to $J_2 < J_1/2$ in CeMnAsO may explain the absence of the T-O structural transition here. This is further supported by the absence of the T-O transition in the isostructural BaMnAsF\cite{21}, and in the Mn-based “122” system BaMn$_2$As$_2$\cite{24}. The AFM structure in both systems is G-type with the NN Mn spins antiparallel along all the directions and Mn moments are along the $c$-axis. Thus, the dominant magnetic interaction in BaMnAsF and BaMn$_2$As$_2$ is also $J_1$ with $J_2 < J_1/2$ without the significant spin frustration as in CeMnAsO. It is very interesting to point out that the T-O structural transition at $\approx 35$ K was found in PrMnSbO below its $T_N$ ($= 230$K), which does not originate from the transition-metal magnetism as that of the “1111” Fe-based pnictides and but most likely driven by the local $f$-electron degrees of freedom of rare-earth Pr$^{3+}$. The absence of any T-O structural transition below $T_N$ in CeMnAsO indicates that the rare-earth Ce$^{3+}$ cannot drive such T-O structural transition, the mechanism of which deserves further investigations.

As compared to BaMn$_2$As$_2$ with the antiparallel Mn spins along $c$-axis, the parallel Mn spins along $c$-axis in CeMnAsO suggests that the interlayered magnetic interaction $J_c > 0$ in BaMn$_2$As$_2$ but $J_c < 0$ in CeMnAsO. The Néel temperature of 625 K in BaMn$_2$As$_2$ with three-dimensional magnetism is much higher than 345 K in CeMnAsO, implying a much weaker magnetic interlayer interaction $J_c$ in CeMnAsO. The much weaker interlayer magnetic interaction due to longer distance between adjacent MnAs layers gives CeMnAsO a quasi-two-dimensional AFM character similar to other “1111” systems\cite{28}.

The Mn SR transition found in CeMnAsO is not observed in LaMnAsO\cite{19}, BaMnAsF\cite{21} or BaMn$_2$As$_2$\cite{24} where there is no magnetic rare earth ion but is found in CeMnAsO and NdMnAsO\cite{19} involving magnetic rare-earth ions, which strongly indicates that such SR transition in CeMnAsO is driven by the coupling between rare earth Ce and Mn. The Mn$^{2+}$ moment, com-
monly displaying very weak single-ion anisotropy as expected for the $L = 0$ of Mn$^{2+}$, favors orientation along the c-axis. As soon as Ce$^{3+}$ spins ($S = 1/2$ and $L = 3$) order below 35 K, the Ce-Mn coupling exerts an effective field that induces a flop of Mn$^{2+}$ spins to the basal plane. It is of interest to point out that there is also a spin reorientation in the transition metal ions in CeFeAsO [8] and CeCrAsO [12] but not in CeCoAsO [10] or CeNiAsO [10]. In CeFeAsO, the stripe-like Fe$^{2+}$ spins rotate uniformly and gradually in the ab plane below ~14 K and a SR of Cr occurs at ~36K in CeCrAsO. Recently we performed neutron diffraction measurements on CeCoAsO and confirmed the FM behavior of Co without evidence for a Co SR transition or Ce ordering in agreement with previously reported studies. [11] SR of transition metal ions Mn, Fe, Cr, has also been observed in other systems involving coupling between magnetic rare earth $f$ and transition metal $d$ moments, such as in $R$FeO$_3$ ($R$=Ce and Nd) [29], $R$Fe$_2$ ($R$=Ce and Nd) [30], $R$$_2$$Fe$_4$B ($R$=Nd and Er) [31], hexagonal HoMnO$_3$ [32], RCrO$_3$ ($R$=Ce, Nd and Sm) [33].

CONCLUSION

In summary, we report on the structure and magnetic properties in CeMnAsO. Whereas no structural transition is observed above/below Neél temperature in tetragonal CeMnAsO, it exhibits a set of complex magnetic transitions. We find a two-dimensional short-range ordered Mn above $T_N = 345$ K with a C-type AFM structure of Mn moments along the c-axis. A rapid spin reorientation of the Mn moments from the c-axis to the ab plane while keeping the C-type order occurs below $T_{SR} = 35$ K, which is induced by a long-range ordering of Ce via Ce-Mn coupling. Below 7 K, the collinear magnetic structure transforms to a noncollinear one with an angle between the Ce and Mn moments. A possible field-induced metamagnetic transition is observed below $T_{SR}$. The local-moment antiferromagnetism with dominant NN interaction and negligible NNN interaction in CeMnAsO contrasts with the itinerant antiferromagnetism in CeFeAsO where the NNN interaction is significant. We also point out that the spin reorientation transition may be common not only to Mn, but also to Fe or Cr ions in the oxypnictides and other oxides or intermetallics once there exists strong coupling between rare-earth $R$ and the transition metal ions.

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