Metamagnetic transitions and anomalous magnetoresistance in EuAg$_4$As$_2$ single crystal

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In this paper, the magnetic and transport properties were systematically studied for EuAg$_4$As$_2$ single crystals, crystallizing in a centrosymmetric trigonal CaCu$_4$P$_2$ type structure. It was confirmed that two magnetic transitions occur at $T_{N1} = 10$ K and $T_{N2} = 15$ K, respectively. With the increasing field, the two transitions are noticeably driven to lower temperature. At low temperatures, applying a magnetic field in the ab plane induces two successive metamagnetic transitions. For both $H \parallel ab$ and $H \parallel c$, EuAg$_4$As$_2$ shows a positive, unexpected large magnetoresistance (up to 202%) at low fields below 10 K, and a large negative magnetoresistance (up to -78%) at high fields/intermediate temperatures. Such anomalous field dependence of magnetoresistance may have potential application in the future magnetic sensors. Finally, the magnetic phase diagrams of EuAg$_4$As$_2$ were constructed for both $H \parallel ab$ and $H \parallel c$.

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I. INTRODUCTION

Responses of Eu-based compounds to external fields have generated immense interest due to exhibiting many exotic properties, such as valence transition$^9$, Kondo behavior$^{10}$, quantum hall effect$^{11}$, novel magnetoresistance (MR)$^{12}$, resulted from their complicated, tunable magnetic ground states with large local moments. In those modulated systems with an incommensurate magnetic structure induced by the strong competing interactions involving the magnetic moments, the lattice, and/or the conduction electrons, like the long-range Ruderman-Kittel-Kasuya- Yosida (RKKY) type coupling, one or several successive transitions have been observed at low temperatures when a magnetic field is applied, leading to complex magnetic phase diagrams$^{13}$.

The Eu-based ternary pnictide EuAg$_4$As$_2$, crystallizing in a centrosymmetric trigonal CaCu$_4$P$_2$ type structure (space group $R3m$, No. 166), was first reported by Stoyko et al.$^8$. The structure can be considered as the derivative from the trigonal CaAl$_2$Si$_2$-type structure, by inserting an additional itinerant Ag$_2$ layer between the close-packed Ag$_2$As$_2$ layers. Unlike in CaCu$_4$P$_2$, where the Cu$_2$ sites are fully occupied, the Ag$_2$ sites are split into three isotropic and equally partially occupied sites in EuAg$_4$As$_2$ [see Fig. 1(b)]. To the authors’ knowledge, a few studies on the physical properties of the ternary CaCu$_4$P$_2$ type pnictides AAg$_4$P$_n$$_2$ (A = Sr, Eu; Pn = As, Sb) have been reported. For SrAg$_4$As$_2$, the quantum oscillation measurements reveal small Fermi pockets with light effective masses and unexpected high mobilities, in contrast with the prediction of the first-principle calculation$^6$. For the magnetic EuAg$_4$As$_2$, based on the measurements of magnetization$^8$, neutron diffraction$^{16}$, Mössbauer spectroscopy$^{17,18}$ and pressure effect$^{19}$, it was found that a structural distortion occurs at about 120 K, and two magnetic transitions emerge at 15 K and 9K, respectively, below 9 K, an incommensurate, non-collinear long-range antiferromagnetic (AFM) state; between 9 - 15 K, a long-range magnetic order with the Eu$^{2+}$ 4$f^7$ spin moments having an incommensurate sine modulated structure. However, there are no reports about the magnetotransports and the phase diagram for this low-dimensional system with so rich magnetic structures.

In this article, we report the magnetization and MR measurements on EuAg$_4$As$_2$ single crystal. It is confirmed that two magnetic transitions occur at $T_{N1} = 10$ K and $T_{N2} = 15$ K, respectively, with the magnetic moments being aligned in the $ab$ plane. We further observed that the magnetic transition temperatures, $T_{N1}$ and $T_{N2}$, decrease with increasing magnetic field and two successive metamagnetic (MM) transitions occurring at 0.5 T and 0.95 T applied in the $ab$ plane at 2 K. Interestingly, it was found that an anomalous MR emerges for both $H \parallel ab$ and $H \parallel c$ orientations, with different magnetic field dependence at various temperature range, which is related to the magnetic ground states. Finally, we constructed the phase diagrams based on the data of the magnetization and resistivity measurements.

II. EXPERIMENTAL METHODS

EuAg$_4$As$_2$ crystals were grown using a self-flux method. First, Eu chunks, and Ag, As powders were
mixed with a ratio of 1:4:2 and were put into an alumina crucible and sealed in an evacuated silica tube. The mixture was heated up to 1100 °C and kept for 24 hours, then cooled down to 700 °C at a rate of 3 °C/h. Finally the furnace was cooled to room temperature after shutting down the power. Single crystals with a typical dimension of 0.8 mm were mechanically exfoliated from the flux. The crystalline composition was determined by Energy-dispersive X-ray spectroscopy (EDX) in a Zeiss Supra 55 scanning electron microscope to be the stoichiometric EuAg₄As₂.

The lattice structure was confirmed by X-ray diffraction (XRD) performed at room temperature on a Rigaku X-ray diffractometer with Cu Kα1 radiation [see Fig. 1(a)]. All XRD peaks are indexed to be (00l) planes, and the cell parameter c is yielded to be about 23.65 Å, in consistent with the previous result[10]. The magnetization was measured using the Quantum Design Magnetic Properties Measurement System (MPMS-VSM-7T). The resistivity measurements were carried out by the standard four-probe technique on the Quantum Design Physical Properties Measurement System (PPMS-9T).

### III. RESULTS AND DISCUSSIONS

Figure 2 shows the temperature dependence of magnetic susceptibility, $\chi_{ab}(T)$ (H $\parallel$ ab plane) and $\chi_c$ (H $\parallel$ c axis), respectively, measured at magnetic field of 1 kOe with a zero field cooling (ZFC) process for a EuAg₄As₂ single crystal. The higher temperature ($> 50$ K) $\chi_{ab}(T)$ data can be well fitted by the Curie-Weiss law, $\chi = \chi_0 + \frac{C}{T - \theta}$, where $\chi_0$ and $C$ are the temperature-independent constants, $T_0$ is the Curie temperature. The fitting result yields an effective moment $\mu_{eff} = 7.95 \mu_B$, close to the theoretical value of Eu$^{2+}$ moments ($g\sqrt{S(S + 1)} = 7.94 \mu_B$, $S = 7/2$ and $g = 2$), implying that Eu$^{2+}$ 4f$^7$ electrons are localized, and a positive Curie temperature $T_0 = 14.1$ K, indicating ferromagnetic (FM) interactions in the paramagnetic (PM) regions (see Table I). At low temperatures, $\chi_{ab}(T)$ exhibits a sharp peak at about 15K ($T_{N2}$) and a kink at 10K ($T_{N1}$), where the transition temperature $T_{N1}$ is a little bit larger than that reported previously[11] due to the smaller field applied in our measurement, as discussed below. The result confirms that the triangle Eu$^{2+}$ spin sublattice undergoes a transition from a PM state to an incommensurate sine modulated AFM state (referred to as AFM-II) at $T_{N2}$ and then to an incommensurate, non-collinear AFM state (referred to as AFM-I) at $T_{N1}$[11][11]. The $\chi_c(T)$ has a similar behavior to that in $\chi_{ab}(T)$ at higher temperatures, but $\chi_c(T)$ remains almost unchanged and is larger than that of $\chi_{ab}(T)$ below $T_{N1}$, suggesting that the Eu$^{2+}$ moments align within the ab plane.

Figure 3(a) shows $\chi_{ab}(T)$ measured at several selected fields. At $\mu_0 H = 0.1$ T, the AFM-II transition occurs at $T_{2}^{ab} = 15$ K and the AFM-I transition occurs at $T_{1}^{ab} = 10$ K, as discussed above. It can be seen that with increasing field, both $T_{1}^{ab}$ and $T_{2}^{ab}$ are noticeably shifted to lower temperature. Meanwhile, an additional transition is observed at $T_{3}^{ab}$ under external field, characterized by a tiny kink in the $\chi_{ab}(T)$ curves. For $0.3 \leq \mu_0 H \leq 0.6$ T, we observe a clearly deviation between ZFC and FC curves at low temperatures, which may be due to the MM transitions occurring in this field range as discussed.
FIG. 3. (Color online) Temperature dependence of magnetic susceptibility of the EuAg₄As₂ single crystal under several selected magnetic fields for $H \parallel ab$ (a) and $H \parallel c$ (d), below $T = 50$ K. For some selected magnetic fields along the $ab$ plane, both field-cooled (FC, hollow circles) and zero-field-cooled (ZFC, solid circles) data are shown. For most other cases, only the ZFC data are shown. In the case of $H \parallel ab$, the data have been shifted for clarity except for the one taken in the field of 0.1 T. Field dependence of magnetization of the EuAg₄As₂ single crystal at various temperatures, with the field parallel to the $ab$ plane (b) and $c$ axis (e). The data taken below $T = 14$ K have been shifted for clarity. (c) The field dependence of $M_{ab}$ (top) and its first derivative (bottom) taken in the field increasing process at $T = 2$ K. (f) The field dependence of $M_c$ (top) and its first derivative (bottom) at $T = 2$ K. The dashed lines are guides to the eyes.

below. From the $\chi_c(T)$, it can also be seen that both $T_1^c$ and $T_2^c$ are shifted to lower temperature with increasing magnetic field as shown in Fig. 3(d).

In order to understand these peculiar behaviors of $\chi(T)$, we carried carefully out the isothermal magnetization measurements. Figure 3(b) shows the field dependence of magnetization, $M_{ab}(H)$, for $H \parallel ab$ up to 5 T. At $T = 50$ K, the $M_{ab}$ increases near linearly with increasing field, indicating a typical paramagnetic behavior. When $14$ K $< T \leq 30$ K, the $M(H)$ curves exhibit an apparent nonlinear behavior due to the magnetic fluctuation close to the $T_{N2}$. When $T \leq 14$ K, we note that the $M_{ab}$ undergoes a steep jump with increasing field, and is split into two jumps in the field of $H_{1}^{ab}$ and $H_{2}^{ab}$ at the lower temperatures. With the decreasing temperature, the critical field $H_{2}^{ab}$ shifts to a higher value, while the $H_{1}^{ab}$ remains almost unchanged and coincides with the critical field of AFM-I below $\sim 7$ K. To get more information on the jumps, we present the $M_{ab}$ data taken at $T = 2$ K in Fig. 3(c). With the increasing field, the $M_{ab}$ undergoes two successive steep magnetization jumps at $\mu_0 H_{1}^{ab} = 0.53$ T /0.44 T and $\mu_0 H_{2}^{ab} = 0.95$ T, when ramping the field up/down, and finally saturates to 7.05 $\mu_0$ at $\mu_0 H_{S}^{ab} = 2.53$ T. The critical fields are more clear from the derivative plot of $M_{ab}$ as shown in Fig. 3(c). We note that a hysteresis emerges at the first jump, i.e., $M_{ab}$ values measured in the field increasing and decreasing process are not the same, indicating that this tran-
position is the first order. The similar behavior has been reported in the polycrystalline EuAg$_4$As$_2$ samples before, which is explained by a MM transition of the Eu$^{3+}$ moments. However, only one MM transition is derived in that work, much different from our results. In addition, we note that the $M_{ab}(H)$ curves for $T > 2$ K still have a small slope in the high field region, which implies a FM fluctuation rather than a FM ordered state.

For comparison, no MM transition was detected up to 7T in the case of $H \parallel c$ [see Fig. 3(e)]. At $T = 2$K, the $M_c$ increases slowly with the increasing field compared with $M_{ab}$, and displays two kinks around $\mu_0 H_c^{ab} = 2.06$T, and $\mu_0 H_c^c = 4.14$T, respectively. A small slop is also observed in the high field region for $M_c$, which is not saturated until the highest measured field. With the increasing temperature, the low-field kink shifts to zero gradually at $T_{N1} = 10$K, suggesting that it characterizes a magnetic phase transition from AFM-I to AFM-II.

The two successive MM transitions occurring in EuAg$_4$As$_2$ resemble to that in CaCo$_2$As$_2$, which exhibits two successive spin-flop transitions at low temperatures. However, the authors suggest that the rapid increase in $M_{ab}$ is unlikely due to the spin-flop transition, because the spins tend to align in $ab$ plane in EuAg$_4$As$_2$, as discussed above. This is also supported by the magnetic hysteresis behavior, which indicates a first order transition. We suggest that the origination may be ascribed to a spin-reorientation or spin-reversal transition, which is particularly common in rare-earth intermetallic compounds, and the exact magnetic structure is needed to be determined by the neutron diffraction experiments in the future.

Figure 4 shows the electrical resistivity in the $ab$-plane, $\rho_{ab}(T)$, and along $c$ axis, $\rho_c(T)$, as a function of temperature for a EuAg$_4$As$_2$ crystal. The $\rho_{ab}$ and $\rho_c$ at room temperature (300 K) are about 65 $\mu$Ω cm and 370 $\mu$Ω cm, respectively, thus the resistivity anisotropy $\rho_c/\rho_{ab}$ at $T = 5.7$, which is not so large for this layered compound. With decreasing temperature from 300 K, the $\rho_{ab}$ decreases monotonically at first, then drops sharply near $T_s = 120$ K due to the structural transition, and goes through a hump around 15K, which can be ascribed to the magnetic transitions. The $\rho_c(T)$ exhibits a similar behavior. In the following, we will focus on the in-plane resistivity.

Next, we discuss the magnetic responses of $\rho_{ab}(T)$ measured at low temperatures ($\leq 40$ K) with applied $H \parallel ab$ plane and $H \parallel c$, axis, respectively. Under zero field, the $\rho_{ab}(T)$ exhibits a hump feature starting at $T_{N2} = 15$ K, and a rapid drop around $T_{N1} = 10$ K, which is consistent with the magnetic transitions. For both $H \parallel ab$ and $H \parallel c$, the two transitions shifts to lower temperature with increasing field, and the resistivity hump is suppressed, resulting in a large MR. Meanwhile, an additional transition is observed at $T_s^{ab}$ in the case of $H \parallel ab$, characterized by a peak (or kink) in the $\rho_{ab}(T)$ curves, consistent with the $\chi_{ab}(T)$ data as discussed above. In the highest measured field of 9 T, the $\rho_{ab}(T)$ deceases monotonically with decreasing temperature, and no phase transition is observed for both $H \parallel ab$ and $H \parallel c$.  

FIG. 4. (Color online) Temperature dependence of the in-plane (left) and out-plane (right) resistivity of EuAg$_4$As$_2$ single crystal.

FIG. 5. (Color online) Temperature dependence of in-plane resistivity $\rho_{ab}$ of EuAg$_4$As$_2$ single crystal under several selected fields for $H \parallel ab$ (a) and $H \parallel c$ (b), below $T = 40$ K. The dashed lines are guides to the eyes.
FIG. 6. (Color online) Field dependence of the MR at several selected temperatures for $H \parallel ab$ (a) and $H \parallel c$ (d). The applied current is parallel to the field in the case of $H \parallel ab$. (b) The field dependence of MR$^{ab}$ (left) and its first derivative (right) at $T = 2$ K. (c) The first derivative of the $M_{ab}$ as a function of field at $T = 2$ K. (e) The field dependence of MR$^{c}$ (left) and its first derivative (right) at $T = 2$ K. (f) The first derivative of the $M_{c}$ as a function of field at $T = 2$ K. The dashed lines are guides to the eyes.

Figure 6(a) shows the field dependence of MR for $H \parallel ab$ measured at several selected temperatures. In order to eliminate the hysteresis effect in the first MM transition region, all the data are collected in a field increasing process. At $T = 2$ K, the MR$^{ab}$, defined as $\rho_{ab}(H,T) - \rho_{ab}(0,T)$, increases slowly with increasing field at first, then displays a quick rise around $\mu_0H_1^{ab} = 0.5$ T, and reaches a maximum value of 202% at 0.7 T, then decreases rapidly until $\mu_0H = 1$ T, exhibiting a peak-like feature. With increasing field further, the MR$^{ab}$ decreases gradually to negative values and remains almost unchanged under higher fields, consistent with the behavior of the strong FM fluctuation state. The critical fields at $T = 2$ K are clearly shown in the first derivative of MR$^{ab}$, and are also consistent with that of $dM_{ab}/dH$ [see Fig. 6(b) and 6(c)].

With the increasing temperature, the positive MR$^{ab}$ at low fields is significantly suppressed, and disappears for
$T \geq 10$ K. Instead, a large negative MR$^{ab}$ emerges for $T \geq 10$ K in the whole measuring field range, which is probably contributed to the reduction of spin disorder scattering. At $T = 10$ K, the MR$^{ab}$ can even reach -78% at 9 T. With increasing temperature further, the magnitude of the negative MR$^{ab}$ decreases, exhibiting a maximum at $T_{N1}$. This behavior is similar to that observed in the well-known perovskites-based manganites (CMR systems). It’s interesting that the negative MR$^{ab}$ is also observed at higher temperatures far above $T_{N2}$ in EuAg$_4$As$_2$, such as, the MR$^{ab}$ reaches as large as -21% at 9 T for $T = 40$ K, as shown in Fig. 6(a), which usually occurs in the ferromagnetically ordered state. We also note that there is a sign crossover around 65 K, beyond which MR$^{ab}$ is a negligibly small, but positive value (not shown here). So we suggest that the large negative MR above $T_{N2}$ may origin from the precursor effect of Eu$^{2+}$ 4$f$ moment long-range magnetic ordering, as discussed in Eu$_2$CuSi$_3$ and Eu$_3$Ni$_4$Ga$_7$.[11]

As shown in Fig. 6(d), the field dependence of out-plane MR$^c$ ($H \parallel c$) is quite similar with the behavior in MR$^{ab}(H)$. At $T = 2$ K, with increasing field, the MR$^c$ increases gradually at first, then increases sharply at $\mu_0 H^c = 2.1$ T, goes through a maximum (over 160% at 2 K at a field of 2.5 T), and decreases steeply until $\mu_0 H^c = 4.1$ T, reaches a minimum, finally increases a little until the highest measuring field (9 T). The MR$^c$ behavior is consistent with $M_{ab}(H)$, as shown in Fig. 6(f). Compared with $dM_{ab}/dB$, several additional peaks were observed in the first derivative of MR$^c$, which may be ascribed to the movement of magnetic domain walls with the external field. However, we can’t exclude the possible magnetic transitions. With increasing temperature, the positive MR$^c$ is dramatically suppressed and disappears above $T_{N1} = 10$ K. On the other hand, a large negative MR$^c$ (up to -70% at 9 T) is also occurs and remains up to higher temperature far above $T_{N2}$.

As discussed above, such complicated behaviors exhibiting in $\rho_{ab}(T)$ and $\rho_{c}(T)$ in EuAg$_4$As$_2$ crystal are related to the magnetic ground states at different magnetic fields ($H$) and various temperatures ($T$). In order to clarify the relationship between the transport and magnetic order, we construct the $H(T)$ phase diagram based on the resistivity and magnetization data measured at various ($H$, $T$), as shown in Fig. 7(a) ($H \parallel ab$) and Fig. 7(b) ($H \parallel c$). For $H \parallel ab$ plane, the phase diagram can divided into six regions, AFM-I, AFM-II, MM-I, MM-II, PM and FM fluctuation region. At $T = 2$ K, the AFM-I ground state goes through MM-I and MM-II phase successively with increasing field and enters finally the strong FM fluctuation region, with the critical field of 0.5 T, 0.95 T and 2.5 T, respectively. It should be pointed out that the boundary between FM fluctuation region and PM state can’t be precisely determined. For $H \parallel c$ axis, the phase diagram can divided into four regions, AFM-I, AFM-II, PM and FM fluctuation region, two phase boundaries are clearly distinguished. At $T = 2$ K, EuAg$_4$As$_2$ crystal undergoes magnetic phase transitions from the AFM-I phase to the intermediate AFM-II phase, then to the strong FM fluctuation region with increasing magnetic field, with the critical field of 2.1 T, and 4.2 T, respectively. Another, as shown in Fig. 7(a) and Fig. 7(b), the phase boundaries deduced from the data of magnetization and resistivity measurements, respectively, are well consistent with each other, indicating that the complicated magnetotransport properties are related to the magnetic orders, especially the large positive MR occurring in a narrow magnetic range provides a chance for application on magnetic sensors.

In summary, we studied systematically the magnetic and transport properties of EuAg$_4$As$_2$ single crystals by using magnetization and resistivity measurements. Under zero field, it was confirmed that two magnetic transitions occur at $T_{N1} = 10$ K and $T_{N2} = 15$ K, respectively, with the magnetic moments being aligned in the $ab$ plane. With increasing field, the two magnetic transitions are driven noticeably to lower temperature, indi-
cating that they are tunable ground states. At $T = 2$ K, two successive MM transitions were observed at 0.5 T and 0.95 T, respectively, when applying magnetic field in the $ab$ plane. On the other hand, no MM transition was detected below 7 T for $H \parallel c$. For both $H \parallel ab$ and $H \parallel c$, an anomalous field dependence of MR was observed, which shows a positive, unexpected large value at low fields below 10 K, and a large negative value at high fields/intermediate temperatures, indicating a rather disordered spin-alignment state in the intermediate phases. Such anomalous field dependence of MR is rather rare, and may have potential application in the future magnetic sensors. Interestingly, the large negative MR is seen even at 40 K, which is far above the magnetic transition temperature. According to these results, we established the phase diagrams of EuAg$_4$As$_2$ for both $H \parallel ab$ and $H \parallel c$.

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