Metamagnetic transitions and anomalous magnetoresistance in EuAg$_4$As$_2$ crystals

QinQing Zhu$^{1}$, Liang Li$^{1}$*, ZhiHua Yang$^{1}$, ZheFeng Lou$^{2}$, JianHua Du$^{3}$, JinHu Yang$^{1}$, Bin Chen$^{1}$, HangDong Wang$^{1}$*, and MingHu Fang$^{2,4}$*

$^{1}$Hangzhou Key Laboratory of Quantum Matter, Department of Physics, Hangzhou Normal University, Hangzhou 311121, China; $^{2}$Department of Physics, Zhejiang University, Hangzhou 310027, China; $^{3}$Department of Physics, China Jiliang University, Hangzhou 310018, China; $^{4}$Collaborative Innovation Center of Advanced Microstructures, Nanjing University, Nanjing 210093, China

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Introduction

The responses of Eu-based compounds to external fields have generated immense interest because they may exhibit many exotic properties, such as valence transition [1], Kondo behavior [2], Quantum Hall effect [3], and novel magnetoresistance (MR) [4, 5], resulting from their complicated, tunable magnetic ground states with large local moments. In those modulated systems with an incommensurate magnetic structure induced by strong competing interactions involving the magnetic moments, lattice, and/or conduction electrons, such as the long-range Ruderman-Kittel-Kasuya-Yosida (RKKY) type coupling, one or several successive transitions may be observed at low temperatures when a magnetic field is applied, leading to complex magnetic phase diagrams [6].

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. [7]. The structure can be considered as a derivative of 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 Cu2 sites are fully occupied, the Ag2 sites are split into three isotropic and equally partially...
occupied sites in EuAg₄As₂ (Figure 1 (b)). To the authors’ knowledge, only a few studies on the physical properties of the ternary CaCu₄P₂-type pnictides AAg₄Pn₂ (A=Sr, Eu; Pn=As, Sb) have been reported. For SrAg₄As₂, the quantum oscillation measurements revealed small Fermi pockets with light effective masses and unexpectedly high mobilities, in contrast with the prediction of the first-principles calculations [8]. For magnetic EuAg₄As₂, based on the measurements of magnetisation [9], neutron diffraction [10], Eu Mössbauer spectroscopy [9,11] and pressure effects [12], it was found that a structural distortion occurred at approximately 120 K, and two magnetic transitions emerged at 15 and 9 K, respectively: below 9 K, an incommensurate, non-collinear long-range antiferromagnetic (AFM) state; between 9 and 15 K, a long-range magnetic order with Eu²⁺ 4f⁷ spin moments with an incommensurate sine-modulated structure. However, there are no reports on the magnetotransports or phase diagram for this low-dimensional system with such rich magnetic structures.

In this paper, we report the magnetisation and MR measurements of EuAg₄As₂ single crystal. It was confirmed that two antiferromagnetic transitions occurred at \( T_{N1} = 10 \text{ K} \) and \( T_{N2} = 15 \text{ K} \), with the magnetic moments almost lying in the \( ab \)-plane. We further observed that the \( T_{N1} \) and \( T_{N2} \) decreased with increasing magnetic field, and that two successive metamagnetic (MM) transitions occurred at 0.5 and 0.95 T fields applied in the \( ab \)-plane at 2 K. Interestingly, it was found that an anomalous MR emerged for both \( H//ab \) and \( H//c \) orientations, with different magnetic field dependences at various temperature ranges, related to the various magnetic ground states. Finally, we constructed the phase diagrams of EuAg₄As₂ based on the magnetisation and resistivity data.

2 Experimental methods

EuAg₄As₂ crystals were grown using a self-flux method. First, Eu chunks and Ag and As powders were mixed carefully at a ratio of 1:4:2, placed in an alumina crucible, and sealed in an evacuated silica tube. The mixture was heated to 1100°C at a rate of 60°C/h and kept for 24 h in a muffle furnace, before being cooled slowly to 700°C at a rate of 3°C/h. Finally, the power was shut down to allow the furnace to cool quickly to room temperature. Single crystals with typical dimensions of 0.8×0.8×0.2 mm³ (the inset of Figure 1(a)) were mechanically exfoliated from the flux. The stoichiometric EuAg₄As₂ was confirmed by Energy-dispersive X-ray spectroscopy (EDX) using a Zeiss Supra 55 scanning electron microscope. X-ray diffraction (XRD) was performed on a Rigaku X-ray diffractometer with Cu Kα1 radiation at room temperature (Figure 1(a)). All XRD peaks were indexed to be (00l) planes, and the cell parameter \( c \) was found to be approximately 23.65 Å, in agreement with previous results [10]. The magnetisation measurements were carried out using a Quantum Design Magnetic Properties Measurement System (MPMS-SQUID-VSM-7T). The MR was measured using the standard four-probe technique on a Quantum Design Physical Properties Measurement System (PPMS-9T).

3 Results and discussion

Figure 1(c) shows the temperature dependence of the magnetic susceptibility, \( \chi_{ab} \) (\( H//ab \)-plane) and \( \chi_c \) (\( H//c \)-axis), measured at a magnetic field of 1 kOe with a zero-field cooling (ZFC) process for a EuAg₄As₂ crystal. At low temperatures, \( \chi_{ab}(T) \) exhibits a sharp peak at approximately 15 K (\( T_{N2} \)) and a kink at 10 K (\( T_{N1} \)), where the transition temperature \( T_{N1} \) is slightly higher than reported previously [10]. This result confirms that the triangular Eu²⁺ spin sublattices undergo a transition from a paramagnetic (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 \( T_{N1} \) [10, 11]. The \( \chi_c(T) \)
remains almost unchanged and is larger than that of $\chi_{ab}(T)$ below $T_{N1}$, suggesting that the Eu$^{3+}$ moments lie almost in the $ab$-plane. Moreover, it should be noted that the large residual magnetic moment at $T=2$ K is consistent with the incommensurate, non-collinear AFM ordered state.

Figure 2(a) shows $\chi_{ab}(T)$ measured at several magnetic fields. At $H=0.1$ T, the AFM-II and AFM-I transitions occur at $T_{2ab}=15$ K and $T_{1ab}=10$ K, respectively, as discussed above. With increasing field, both $T_{1ab}$ and $T_{2ab}$ are noticeably shifted to lower temperatures. Meanwhile, an additional transition is observed at $T_{3ab}$ under an external field, characterized by a tiny kink in the $\chi_{ab}(T)$ curves. For $0.3 \leq H \leq 0.6$ T, we observe a clear deviation between the ZFC and FC curves in the low temperature region, which may be due to the MM transitions occurring in this field range, as discussed below. From the $\chi_{c}(T)$, it can also be seen that both $T_{1c}$ and $T_{2c}$ are shifted to lower temperatures with increasing magnetic field, as shown in Figure 2(d).

To understand these novel behaviors of $\chi(T)$, we carefully performed isothermal magnetisation measurements. Figure 2(b) shows the field dependence of the magnetisation, $M_{ab}(H)$, for $H//ab$ up to $5$ T. At $T=50$ K, $M_{ab}$ increases almost linearly with increasing field, indicating typical PM behavior. When $14 \leq T \leq 30$ K, the $M_{ab}(H)$ curves exhibit an apparent nonlinear behavior owing to the magnetic fluctuation close to $T_{N2}$. At $T \leq 10$ K, $M_{ab}(H)$ exhibits two jumps at $H_{1ab}^{c}$ and $H_{2ab}^{c}$, respectively, and a tiny kink at $H_{3ab}^{c}$, indicating three transitions emerging, which will be discussed in detail below. With decreasing temperature, the critical field $H_{2ab}^{c}$ shifts to a higher value, whereas $H_{1ab}^{c}$ remains almost unchanged. To obtain more information on the abnormal jumps, we present the $M_{ab}$ data measured at $T=2$ K in the upper panel of Figure 2(c). With increasing field, $M_{ab}$ initially increases linearly, consistent with the expectation of the AFM ground state. Subsequently, it changes sharply at $H=0.53$ T/0.44 T, when increasing/decreasing the magnetic field, respectively, i.e., a hysteresis emerging at this field that corresponds to the first MM transition (MM-I). After the transition, $M_{ab}(H)$ displays a linear dependence. As the field increases further, $M_{ab}(H)$ increases again sharply at $H_{2ab}^{c}=...$
0.95 T without hysteresis, corresponding to a second MM transition (MM-II), and then returns to a linear field dependence again. Finally, it saturates to 7.05 \( \mu_B \) at \( \mu_0H_{3ab} = 2.53 \) T. The critical fields are displayed more clearly in the derivative plot of \( M_{ab}(H) \), as shown in the lower panel of Figure 2(c). A hysteresis emerges at the MM-I transition, i.e., the \( M_{ab} \) values measured in the increasing and decreasing field processes are not the same, indicating that this transition is the first order. Similar behavior has been reported in polycrystalline EuAg\(_4\)As\(_2\) samples, which was explained as an MM transition of the Eu\(^{2+}\) moments. However, only one MM transition was observed in this work [9], which is different from our results. In addition, we note that the \( M_{ab}(H) \) curves for \( T > 2 \) K retain a small slope in the high field region, which implies an FM-like alignment state rather than an FM ordered state. For comparison, we also measured the isothermal magnetisation when a magnetic field was applied along the c-axis, as shown in Figure 2(e), but no MM transition was observed up to 7 T. At \( T = 2 \) K, \( M_c(H) \) increases slowly with increasing field compared with \( M_{ab}(H) \), and displays two kinks around \( \mu_0H_{1c} = 2.06 \) T and \( \mu_0H_{2c} = 4.14 \) T. A small slope is also observed in the high field region for \( M_c(H) \), which does not saturate until the highest measured field. At \( T_{N1} = 10 \) K, the kink in \( M_c(H) \) disappears, which may be related to the difference between AFM-I and AFM-II.

Next, we discuss the two MM transitions observed in the \( M_{ab}(H) \) curves. According to the neutron diffraction results reported in ref. [10], below \( T_{N1} \), EuAg\(_4\)As\(_2\) is an incommensurate antiferromagnet (AFM-I), i.e., the spins of Eu\(^{2+}\) rotate around the c-axis in a helical arrangement, and around the b-axis in a cycloidal arrangement, with a small propagation vector of \( \mathbf{K}_\text{n} = (0, -0.1, 0.12) \). Such a complex non-collinear magnetic structure was considered to result from the RKKY interaction in EuAg\(_4\)As\(_2\) [10]. As discussed above, with an increasing field applied in the \( ab \)-plane, \( M_{ab} \) undergoes two steep jumps at \( H_{1ab} \) and \( H_{2ab} \), the first jump exhibiting a notable hysteresis and the second without hysteresis, before finally saturating to 7.05 \( \mu_B \) at \( H_{3ab} \). However, no MM transition was observed as \( H \) was applied along the c-axis. These results indicate that the spin alignment of EuAg\(_4\)As\(_2\) for \( H/ab \) may transform into two metastable states (MSS-I and MSS-II) successively with increasing field. Although the exact arrangement of spins in these metastable states can not be described by our static magnetisation measurements alone, and needs to be determined by neutron diffraction experiments in the future, the corresponding MM transitions are different from those due to the spin-flop transition (in which the spins are driven perpendicular to the applied magnetic field), which are usually observed in a uniaxial antiferromagnet with low anisotropy, such as in CaCo\(_2\)As\(_2\) [13]. In EuAg\(_4\)As\(_2\), the spin alignment below \( T_{N1} \) may have four states: AFM-I (discussed above), MSS-I (\( H_{1ab} < H < H_{2ab} \)), MSS-II (\( H_{2ab} < H < H_{3ab} \)), and FM-like alignment (\( H > H_{3ab} \)) under an applied magnetic field in the \( ab \)-plane. Finally, the balance between Zeeman energy, magnetic coupling energy, and magnetocrystalline anisotropy energy leads the moments to rotating to the direction of \( H \) (in the \( ab \)-plane). Similar phenomena can also be found in other rare-earth intermetallic compounds [14-17].

Figure 3 shows the electrical resistivity in the \( ab \)-plane, \( \rho_{ab}(T) \), and along the c-axis, \( \rho_c(T) \), as a function of temperature for a EuAg\(_4\)As\(_2\) crystal. The values of \( \rho_{ab} \) and \( \rho_c \) at room temperature (300 K) are 65 and 370 \( \mu\Omega \) cm, respectively; thus, the resistivity anisotropy \( \rho_c/\rho_{ab} = 5.7 \), which is not so large for this layered compound. With decreasing temperature from 300 K, \( \rho_{ab} \) initially decreases monotonically, then drops sharply near \( T_s = 120 \) K due to a structural transition [10], and exhibits a hump at approximately 15 K, which can be ascribed to the magnetic transitions. \( \rho_c(T) \) exhibits a similar behavior.

Then, we discuss the magnetic responses of \( \rho_{ab}(T) \) measured at low temperatures (\( \leq 40 \) K) with the applied \( H/ab \)-plane and \( H//c \)-axis, respectively (Figure 4(a) and (b)). Under zero field, \( \rho_{ab}(T) \) 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 \( H/ab \) and \( H//c \), the two transitions are shifted to lower temperatures with increasing field, and the resistivity hump is suppressed, resulting in a large MR. Meanwhile, an additional transition is observed at \( T_{3ab} \) in the case of \( H/ab \), 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, \( \rho_{ab}(T) \) decreases monotonically with decreasing temperature, and no phase transition is observed for either \( H/ab \) or \( H//c \).

Figure 5(a) shows the field dependence of the MR for
**Figure 4** (Color online) Temperature dependence of in-plane resistivity $\rho_{ab}$ of EuAg$_4$As$_2$ single crystal under several selected fields for $H//ab$ (a) and $H//c$ (b), below $T=40$ K. The dashed lines are visual guides.

**Figure 5** (Color online) Field dependence of the MR at several temperatures for $H//ab$ (a) and $H//c$ (d). The applied current is parallel to the field in the case of $H//ab$. (b) Field dependence of MR$^{ab}$ (left) and its first derivative (right) at $T=2$ K. (c) First derivative of $M_{ab}$ as a function of field at $T=2$ K. (e) Field dependence of MR$^c$ (left) and its first derivative (right) at $T=2$ K. (f) First derivative of $M_c$ as a function of field at $T=2$ K. The dashed lines are visual guides.

$H//ab$ measured at several temperatures. 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 $\rho_{ab}(H,T)-\rho_{ab}(0,T)$ $\rho_{ab}(0,T)$, increases slowly with increasing field at first, then exhibits a rapid rise around $\mu_0H_1^{ab}$=0.5 T, and reaches a maximum value of 202% at 0.7 T, before decreasing rapidly until $\mu_0H=1$ T, exhibiting a peak-like feature. With further increasing field, the MR$^{ab}$ decreases
gradually to negative values, reaches a minimum, and then increases slightly, consistent with the behavior of the FM-like state. The critical fields at $T=2$ K are clearly shown in the first derivative of $\text{MR}^{ab}$, and are also consistent with those of $dM_{ab}/dH$ (Figure 5(b) and (c)), indicating that the spin alignment of MSS-I has the strongest scattering of electrons, resulting in a large positive MR. With increasing temperature, the positive $\text{MR}^{ab}$ at low fields is significantly suppressed, and disappears for $T \geq 10$ K. Instead, a large negative $\text{MR}^{ab}$ emerges for $T > 10$ K in the whole measuring field range, which is probably attributed to the reduction of spin disorder scattering. At $T=10$ K, the $\text{MR}^{ab}$ can even reach $\sim 78\%$ at 9 T. With further increasing temperature, the magnitude of the negative $\text{MR}^{ab}$ decreases, exhibiting a maximum at $T_{N1}$. This behavior is similar to that observed in the well-known perovskite-based manganites (CMR systems) [18]. It is interesting that negative $\text{MR}^{ab}$ is also observed at temperatures far above $T_{N2}$ in EuAg$_2$As$_2$. For example, $\text{MR}^{ab}$ reaches as much as $-21\%$ at 9 T for $T=40$ K, as shown in Figure 5(a), which usually occurs in the ferromagnetically ordered state. We also note that there is a sign crossover around 65 K, beyond which $\text{MR}^{ab}$ is a negligibly small but positive value (not shown here). Therefore, we suggest that the large negative MR above $T_{N2}$ may originate from the precursor effect of Eu$^{2+}$ 4$f^7$ moment long-range magnetic ordering, as discussed in Eu$_2$CuSi$_3$ and Eu$_3$Ni$_4$Ga$_4$ [4, 14].

As shown in Figure 5(d), the field dependence of $\text{MR}^c$ ($H//c$) is quite similar to the behavior in $\text{MR}^{ab}(H)$. At $T=2$ K, with increasing field, $\text{MR}^c$ initially increases gradually and then sharply at $\mu_0H_1^c=2.1$ T, exhibits a maximum (over 160% at 2 K and 2.5 T), subsequently decreases steeply until $\mu_0H_2^c=4.1$ T, reaches a minimum, and finally increases slightly until the highest measured field (9 T). The $\text{MR}^c$ behavior is consistent with that of $M_c(H)$, as shown in Figure 5(f). Compared with $dM_c/dB$, several additional peaks were observed in the first derivative of $\text{MR}^c$, which may be ascribed to the movement of magnetic domain walls with the external field. These results imply that when a magnetic field is applied along the $c$-axis, the spin alignment in AFM-II has the strongest scattering of electrons flowing in the $ab$-plane (corresponding to $\rho_{ab}$), resulting in a large positive MR. With increasing temperature, $H_1^c$ and $H_2^c$, and the positive MR decrease. At $T=10$ K, the peak of positive MR disappears, and the largest negative MR (up to $\sim 70\%$ at 9 T) emerges. Up to 40 K, far above $T_{N2}$, a large negative MR remains ($\sim 20\%$, 9 T). With the exception of 2 K, the MR decreases monotonically with increasing magnetic field after the positive peak at all temperatures, which may be related to the spin dynamics.

As discussed above, the complicated behaviors exhibited 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 ($T$). To clarify the relationship between the transport and magnetic order, we constructed the $H(T)$ phase diagrams based on the resistivity and magnetisation data measured at various ($H$, $T$), as shown in Figure 6(a) ($H//ab$ and (b) $H//c$). For $H//ab$-plane, the phase diagram can be divided into six regions: AFM-I, AFM-II, PM, the FM-like alignment region, and two metastable states (MSS-I and MSS-II). At $T=2$ K, the compound undergoes two MM transitions from AFM-I with increasing field and finally enters the FM-like alignment region, with critical fields of 0.5, 0.95, and 2.5 T, respectively. It should be noted that the boundary between the FM-like alignment region and PM state can not be precisely determined. For $H//c$-axis, the phase diagram can be divided into four regions: AFM-I, AFM-II, PM, and the FM-like alignment region; two phase boundaries are clearly distinguished. At $T=2$ K, the EuAg$_4$As$_2$ crystal undergoes magnetic phase transitions from the AFM-I phase to the intermediate AFM-II phase, then to the FM-like alignment region with increasing
magnetic field, with critical fields of 2.1 and 4.2 T, respectively. As shown in Figure 6(a) and (b), the phase boundaries, deduced from the data of magnetisation and resistivity measurements, are well consistent with each other, indicating that the complicated magnetotransport properties are related to the magnetic orders. In particular, the large positive MR that occurs in a narrow magnetic range provides the opportunity for application in magnetic sensors.

4 Conclusions

In summary, we systematically studied the magnetic and transport properties of EuAg$_4$As$_2$ single crystals using magnetisation and resistivity measurements. Under zero field, it was confirmed that two magnetic transitions occurred at 0.5 and 0.95 T, when applying a magnetic field in the $ab$-plane. With increasing field, the two magnetic transitions were driven noticeably to lower temperatures, indicating that they are tunable ground states. At $T=2$ K, two successive MM transitions were observed at 0.5 and 0.95 T, when applying a magnetic field in the $ab$-plane. In contrast, no MM transition was detected below 7 T for EuAg$_4$As$_2$. This anomalous field dependence of MR is rare indicating a rather disordered spin alignment state in the intermediate phases. This anomalous field dependence of MR is rare and may have potential applications in future magnetic sensors. Interestingly, a large negative MR was observed even at 40 K, which is far above the magnetic transition temperature. Based on these results, we established the phase diagrams of EuAg$_4$As$_2$ for both $H//ab$ and $H//c$.

Note added: While the paper was being reviewed, we learned of new results on EuAg$_4$As$_2$ that were updated and published by Bing Shen et al. [arXiv: 1809.07317, ref. [10]].

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