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# Evolution of magnetic properties in iron-based superconductor Eu-doped $\text{CaFe}_2\text{As}_2$

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This work presents the evolution of magnetic properties of  $\text{Eu}_x\text{Ca}_{1-x}\text{Fe}_2\text{As}_2$  ( $0 \leq x \leq 1$ , ECFA) samples. Unlike the resistivity data, that for magnetic susceptibility  $\chi$  (T) does not show any clear evidence of the spin density wave (SDW) transition. When the Curie-Weiss contribution is subtracted, a weak anomaly appears at a temperature close to the SDW transition temperature ( $T_{\text{SDW}}$ ) determined from the resistivity data. To understand the magnetic orders arising from Fe-moments and  $\text{Eu}^{2+}$  spins order, we have studied the doping dependence of  $T_{\text{SDW}}$  and  $\text{Eu}^{2+}$  antiferromagnetic order  $T_N$ . It is found that  $T_{\text{SDW}}$  increases almost linearly with increasing  $x$  and remains nearly unchanged above  $x \sim 0.4$ , whereas  $T_N$  first appears at  $x \sim 0.4$  and varies almost linearly with further increasing  $x$ . These observations suggest that magnetic orders due to two sublattices are coupled to each other. The results discussed here are helpful for understanding the magnetic properties of ECFA and other iron-based superconductors.

## KEYWORDS

iron-based superconductors, spin-density wave order, antiferromagnetic order, Curie-Weiss law, magnetic susceptibility

## Introduction

Iron-based superconductors (IBSs) have continuously served as a platform for exploring novel experimental tools and theoretical approaches since their discovery, especially in studies on the impact of magnetic fluctuations and quantum criticality (Chen et al., 2008a; Hsu et al., 2008; Kamihara et al., 2008; Wang et al., 2012; Deng et al., 2014; Lei et al., 2016; Komadera et al., 2018; Deng et al., 2021; Fernandes et al., 2022). The Fe-magnetic moments appear as a spin density wave (SDW) and, in general, superconductivity emerges when the SDW order is suppressed either by appropriate chemical doping or by applying external pressure (Chen et al., 2008b; Rotter et al., 2008; Takahashi et al., 2008). Thin-film FeSe/STO grown by molecular beam epitaxy (MBE) has exhibited interface-enhanced superconductivity with a  $T_c$  above 40 K (Wang et al., 2012; Deng et al., 2014). More recently, a  $T_c$  of 38 K was retained in FeSe single crystals at ambient pressure via pressure-quenching (Deng et al., 2021). Defects have also been reported to play an important role in the superconductivity of IBSs (Deng et al., 2016).

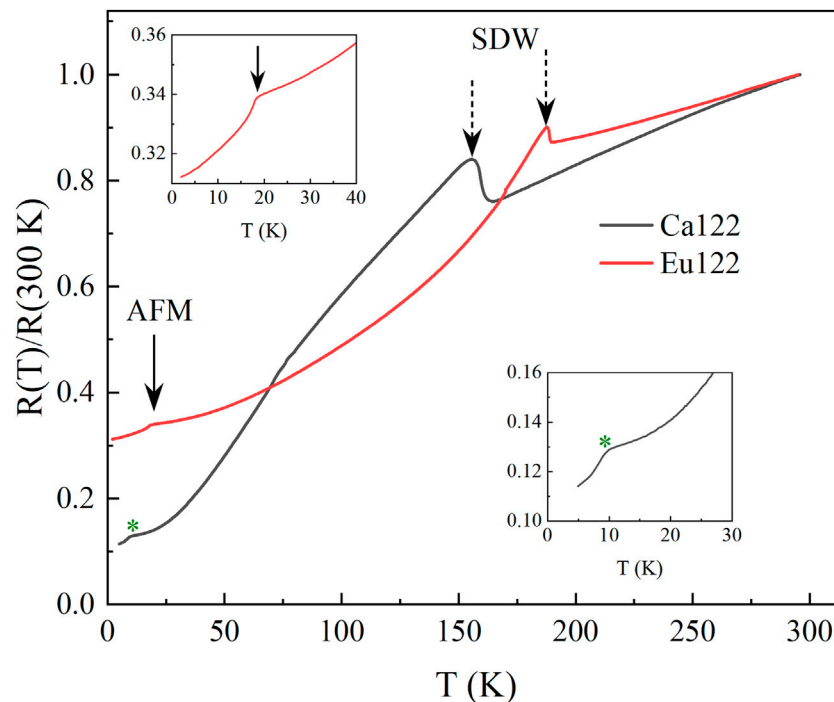
$\text{CaFe}_2\text{As}_2$  (Ca122) and  $\text{EuFe}_2\text{As}_2$  (Eu122) are two members of the “122” IBS family [(AE) $\text{Fe}_2\text{As}_2$  (AE = Ca, Ba, Sr, and Eu)] and show SDW transitions at  $T_{\text{SDW}} \sim 165$  K and 190 K, respectively (Park et al., 2008; Torikachvili et al., 2008; Lee et al., 2009). At the SDW transition, which appears as a sharp upturn in the resistivity data (Park et al., 2008; Lee et al., 2009; Lv et al., 2011; Shrestha et al., 2020), the material undergoes structural (from the tetragonal to the orthorhombic phase) and magnetic phase (antiferromagnetic ordering of Fe-moments) transitions simultaneously. In Ca122, there exists an additional structural transition near 100 K to the “collapsed-tetragonal” (cT) phase under a moderate pressure of  $\sim 0.4$  GPa before the superconductivity transition occurs. However, under truly hydrostatic pressure, no superconductivity is observed in Ca122 (Yu et al., 2009). A higher  $T_c$  up to 49 K was achieved in this system by chemical doping (Lv et al., 2011) with rare-earth elements. Our group recently reported (Zhao et al., 2016) that the  $T_c$  of Ca122 can be raised as high as 25 K using a proper annealing procedure. Another 122 IBS, Eu122, in addition to having a high-temperature SDW transition, exhibits an anomaly near  $T_N \sim 20$  K due to its  $\text{Eu}^{2+}$  antiferromagnetic (AFM) order (Ren et al., 2008). It does not show any signature of superconductivity at

ambient condition, but it can be turned into a superconductor either by chemical doping [with K (Jeevan et al., 2008) ( $T_c \sim 32$  K), with Na (Qi et al., 2008) ( $T_c \sim 35$  K), through Co or La substitutions (He et al., 2010; Ying et al., 2010; Zhang et al., 2012), etc.] or by the application of external high pressure (Terashima et al., 2009; Kurita et al., 2011).

Here, we explore the evolution of the electrical transport and magnetic properties of Eu-doped Ca122 [ $\text{Eu}_x\text{Ca}_{1-x}\text{Fe}_2\text{As}_2$  ( $0 \leq x \leq 1$ , ECFA)]. We recently reported (Shrestha et al., 2020) detailed electrical transport properties of ECFA at ambient and under high-pressure. Here we focus on how its magnetic properties evolve with varied Eu-doping. We observe that, unlike in the measurement of resistivity, the magnetic susceptibility data does not show a visible anomaly resulting from the SDW transition. We find that a tiny anomaly near the SDW transition appears only after subtracting the Curie-Weiss (CW) contribution of the  $\text{Eu}^{+2}$  moments.

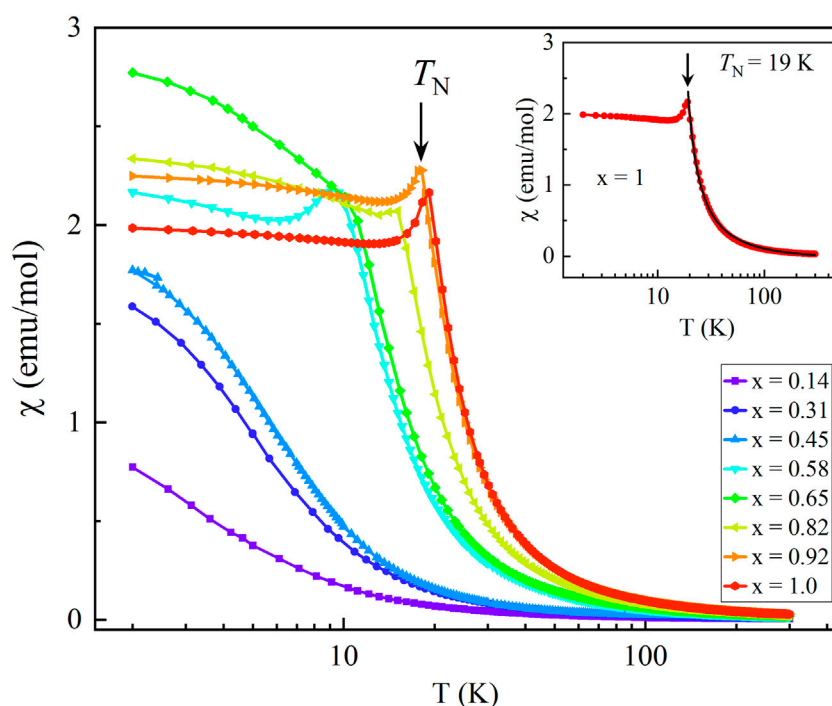
## Experimental details

Large, high-quality single crystals of ECFA were grown using the FeAs flux technique. Starting materials were placed in an



**FIGURE 1**

Electrical Resistance. Normalized electrical resistance vs. temperature for Ca122 and Eu122. Both samples show a clear anomaly, near 160 K and 190 K for Ca122 and Eu122, respectively, and indicated by dashed arrows, that arises due the SDW transition. The tiny kink near 19 K for Eu122, as indicated by the solid arrow, is due to the  $\text{Eu}^{2+}$  AFM order. A small drop in resistance for Ca122 near 10 K, as denoted by an asterisk, is likely due to the superconducting transition (see the text). Upper inset: magnified view of the  $\text{Eu}^{2+}$  AFM transition in Eu122. Lower inset: magnified view of the low-temperature drop in resistance for Ca122.



**FIGURE 2**

Magnetic susceptibility. Magnetic susceptibility ( $\chi$ ) vs.  $T$  for ECFA samples with different amounts of Eu doping ( $0.14 \leq x \leq 1$ ).  $\chi$  ( $T$ ) increases gradually with decreasing temperature. The low-temperature anomalies,  $T_N \sim 10$ – $19$  K, arise due to the  $\text{Eu}^{2+}$  AFM order, as indicated by the arrow. Inset:  $\chi$  ( $T$ ) data for  $\text{EuFe}_2\text{As}_2$  ( $x = 1$ ). The solid black curve is the best-fit curve using the Curie-Weiss model Eq. 1. The x-axis is in the logarithmic scale for better visibility of  $T_N$ .

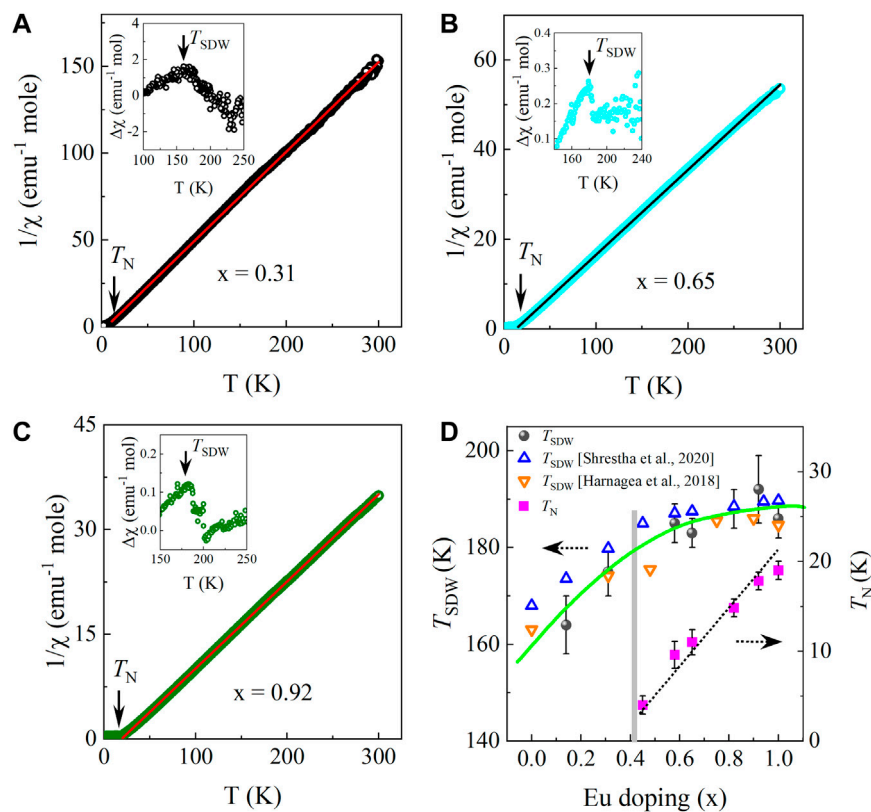
alumina crucible that was sealed inside a small silica tube under a reduced Ar atmosphere. The small silica tube was subsequently sealed inside a larger silica tube under vacuum. The assembly was first heated at a temperature of  $1,100^\circ\text{C}$  and then slowly cooled down at a rate of  $2^\circ\text{C}/\text{h}$ . The sample preparation and preliminary characterization details are provided in our previous report (Shrestha et al., 2020). Electrical transport measurements were carried out in a physical property measurement system (PPMS, Quantum Design). A shiny crystal was selected and four platinum wires were attached to its flat surface using silver paint. Magnetic measurements from room temperature down to 2 K were carried out using a magnetic property measurement system (MPMS, Quantum Design). A plate-like sample was placed inside a gelatin capsule that was subsequently attached to a plastic straw. A small magnetic field value of 0.1 T was applied along the  $c$ -axis to induce a magnetic moment in the sample.

## Results and discussion

Figure 1 shows the temperature-dependent electrical resistance of Ca122 and Eu122. The resistance is

normalized to the room-temperature value. Each sample shows a clear upturn anomaly near  $T_{\text{SDW}} \sim 160$  K (Ca122) or 190 K (Eu122) that arises due to the SDW transition, as indicated by the dashed arrows. These  $T_{\text{SDW}}$  values are consistent with previously reported data (Park et al., 2008; Torikachvili et al., 2008; Lee et al., 2009; Lv et al., 2011). Moreover, there is a tiny kink near  $T_N \sim 19$  K in the Eu122 results (indicated by the solid arrow), which can be more clearly observed in the magnified view shown in the upper inset to Figure 1. This anomaly arises due to the AFM ordering of  $\text{Eu}^{2+}$  spin moments (Shrestha et al., 2020). For Ca122, there is a small drop in the resistance value near 10 K, indicated by an asterisk and more clearly visible in the magnified view shown in the lower inset to Figure 1. Under application of external pressure, Ca122 enters into the superconducting state with a transition temperature  $T_c \sim 10$  K (Park et al., 2008; Torikachvili et al., 2008; Lee et al., 2009). Therefore, this tiny drop in resistance could be due to the onset of the superconducting transition.

Figure 2 shows the temperature-dependent magnetic susceptibility ( $\chi$ ) of ECFA samples with different amounts of Eu doping ( $x$ ).  $\chi$  ( $T$ ) increases gradually with decreasing temperature and exhibits a distinct anomaly near  $T_N \sim$



**FIGURE 3**

Curie-Weiss fit. Inverse susceptibility,  $1/\chi$ , vs.  $T$  for ECFA samples at (A)  $x = 0.31$ , (B)  $x = 0.65$ , and (C)  $x = 0.92$ .  $1/\chi$  increases linearly with  $T$ , consistent with Curie-Weiss behavior. The arrows show the AFM due to the  $\text{Eu}^{2+}$  spins. The solid lines are the best-fit lines using the Curie-Weiss law Eq. 1. The insets in (A)–(C) display the respective  $\Delta\chi$  vs.  $T$  plots showing the  $T_{SDW}$  transitions, as indicated by the arrow in each. (D)  $T_{SDW}$  (solid spheres) and  $T_N$  (solid squares) vs.  $x$  for ECFA samples. Open upright and inverted triangles are  $T_{SDW}$  data from Refs (Shrestha et al., 2020), and (Harnagea et al., 2018), respectively.  $T_{SDW}$  increases rapidly with increasing  $x$  and then slowly saturates to  $T_{SDW} \sim 190$  K above  $x \sim 0.4$ . The error bars are taken as the half-width of the transition. The vertical solid stripe and solid curve are guides to the eye.  $T_N$  first appears at  $x \sim 0.4$  and increases almost linearly with further increasing  $x$ , as indicated by the dotted line.

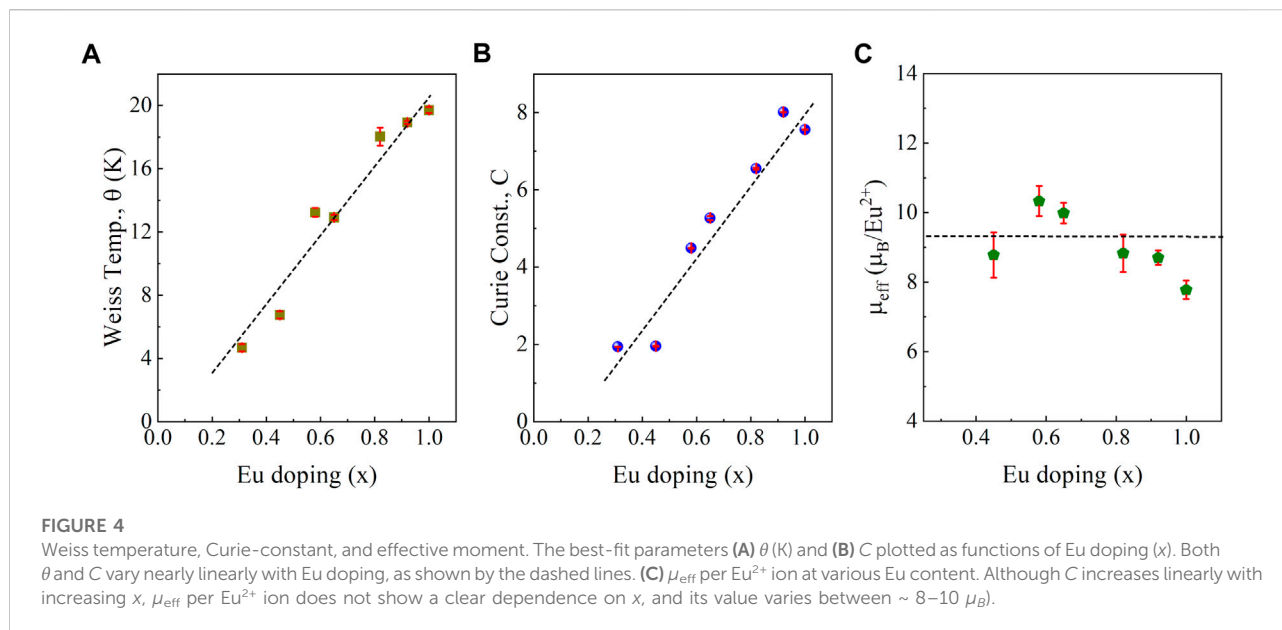
10–19 K, as indicated by the arrow. This transition arises due to the  $\text{Eu}^{2+}$  AFM order (Ren et al., 2008; Zhang et al., 2012; Maiwald and Gegenwart, 2017).  $T_N$  is prominent at higher  $x$ , and it shifts toward lower temperatures at lower  $x$ . For  $x = 1$ ,  $T_N \sim 19$  K, which is consistent with previously reported  $T_N$  values (Ren et al., 2008; Zhang et al., 2012; Maiwald and Gegenwart, 2017). Our recent rigorous electrical transport and magnetic studies (Shrestha et al., 2020) showed that  $T_N$  varies linearly with  $x$ . Here, the temperature dependence of  $\chi(T)$  can be described by the Curie-Weiss (CW) law Eq. 1 as shown in the inset to Figure 2 for the  $x = 1$  sample. The CW law (Ashcroft and Mermin, 1976; Kittel, 2006) is expressed as

$$\chi = \frac{C}{T - \theta} \quad (1)$$

where  $C$  and  $\theta$  are the Curie-Weiss constant and the Weiss temperature, respectively. Here,  $C$  is directly proportional to the effective magnetic moment ( $\mu_{\text{eff}}$ ) of the sample. Therefore, by

fitting the temperature-dependent  $\chi(T)$  data, we can estimate  $\mu_{\text{eff}}$  and  $\theta$  of the ECFA samples.

Figures 3A–C shows the  $1/\chi(T)$  plots for selected ECFA samples.  $1/\chi(T)$  varies linearly at high temperatures, but anomalies corresponding to the  $\text{Eu}^{2+}$  AFM order occur at low temperatures, as indicated by arrows. The solid lines represent the best-fit to the CW law using Eq. 1. As seen in Figures 3A–C, the temperature-dependent susceptibility data can be well explained by the CW law. It is important to note that there is no clear evidence of the SDW transition in any of the ECFA samples studied [neither in Figure 2 nor in Figures 3A–C] due to Fe-moments in our susceptibility data. In previous studies (Ren et al., 2008; Jiang et al., 2009), the same issue of being unable to observe a clear SDW transition in the magnetization data was reported, but it could be observed after subtracting the CW contribution of the  $\text{Eu}^{2+}$  moments. Therefore, we have followed the same approach and calculated  $\Delta\chi(T)$  by subtracting the CW contribution of the



$\text{Eu}^{2+}$  moments. As shown in the insets to Figures 3A–C, there is a tiny but distinguishable anomaly at  $T_{\text{SDW}} \sim 165$ – $185$  K. These  $T_{\text{SDW}}$  values are in close agreement with those previously reported based on resistivity and magnetization measurements (Jiang et al., 2009; Harnagea et al., 2018; Tran et al., 2018; Shrestha et al., 2020). We subsequently determined  $T_{\text{SDW}}$  for various ECFA samples and plotted it as a function of Eu doping. As shown in Figure 3D,  $T_{\text{SDW}}(x)$  initially increases rapidly with increasing  $x$ , gradually increases with further increasing in  $x$  above  $x \sim 0.4$ , and then saturates to the value of 190 K. As mentioned in the Introduction, ECFA serves as a special platform that has two magnetic orders, one from Fe-moments (SDW order) and another due to  $\text{Eu}^{2+}$  spins (AFM order). To confirm the interaction between these orders, we have also plotted  $T_N$  vs.  $x$  on the right  $y$ -axis of Figure 3D. As seen in Figure 3D, with decreasing  $x$ ,  $T_{\text{SDW}}$  remains almost unchanged until the  $\text{Eu}^{2+}$  moment order is destroyed at  $x \sim 0.4$ . This suggests that the magnetic orders of the two sublattices are coupled to one another.

From the CW-fitting above, we have obtained the best-fit parameters  $C = (7.94 \pm 0.001)$  emu K/mole and  $\theta = (19.36 \pm 0.13)$  K for  $x = 0.92$ . From the  $C$  value obtained, we have estimated the effective magnetic moment of  $\text{Eu}^{2+}$ ,  $\mu_{\text{eff}} = (7.97 \pm 0.10) \mu_B$ . These values are comparable with those previously reported (Jiang et al., 2009; Harnagea et al., 2018; Tran et al., 2018) for a similar Eu-doping level. The positive  $\theta$  value implies ferromagnetic (FM) interaction between  $\text{Eu}^{2+}$  moments. Resonant X-ray scattering (Herrero-Martín et al., 2009) and neutron diffraction data (Xiao et al., 2009; Xiao et al., 2010) have also shown that  $\text{Eu}^{2+}$  spins on the same plane have FM interaction, whereas they have weak AFM coupling across the plane. The Eu-doping-dependent best-fit

parameters were calculated for various ECFA samples and are presented in Figure 4.

Figure 4A shows the variation of the best-fit of parameter  $\theta$  with  $x$ . For  $x = 0.31$ ,  $\theta = (4.68 \pm 0.23)$  K, and it increases almost linearly with further increasing Eu doping. At  $x = 1$ ,  $\theta = (19.71 \pm 0.19)$ . This  $\theta$  value is comparable with those of 20.3 K reported by Harnagea et al. (2018) and 19.7 K reported by Jiang et al. (2009). Similarly, the Eu-doping-dependent best fit of  $C$  is shown in Figure 4B and has nearly linear dependence on  $x$ . Figure 4C displays  $\mu_{\text{eff}}$  per  $\text{Eu}^{2+}$  ion at various Eu content and shows that, although  $C$  increases linearly with  $x$ ,  $\mu_{\text{eff}}$  per  $\text{Eu}^{2+}$  ion does not exhibit any clear Eu-doping dependence. Its value changes between  $\sim 8$ – $10 \mu_B$ .

The doping effect on 122 IBSs has been studied extensively using both rare-earth metals ( $RE = \text{La}, \text{Pr}, \text{Nd}$ , etc.) (Qi et al., 2008; Gao et al., 2011; Lv et al., 2011; Saha et al., 2012; Ying et al., 2012) and alkali metals ( $A = \text{K}$  and  $\text{Na}$ ) (Rotter et al., 2008; Sasmal et al., 2008; Zhao et al., 2010). Replacing a divalent  $AE^{2+}$  ion with a trivalent  $RE^{3+}$  (or a univalent  $A^+$ ) in a 122 IBS serves as electron doping (or hole doping) and tunes the electronic properties of the system. In both cases (electron and hole doping), the SDW order is gradually suppressed, and the superconducting phase has been stabilized in the 122 system with a maximum  $T_c$  of 49 K (for electron doping) (Gao et al., 2011; Qi et al., 2012; Saha et al., 2012; Ying et al., 2012) and 38 K (for hole doping) (Rotter et al., 2008; Sasmal et al., 2008; Zhao et al., 2010). The superconducting phase in the RE-doped 122 (or A-doped 122 IBS) arises by the suppression of the SDW order via chemical pressure, electron (or hole) doping, or the combination of both (Saha et al., 2012). However, isovalent substitution of  $\text{Ca}^{2+}$  with  $\text{Eu}^{2+}$  neither adds any charge carriers



in the system nor suppresses the SDW order. As a result, no superconductivity has been observed in ECFA samples here. Nonetheless, ECFA provides a unique platform for studying magnetic orders arising from  $\text{Eu}^{2+}$  and  $\text{Fe}^{2+}$  sublattices. The doping dependence of  $T_{\text{SDW}}$  and  $T_N$ , as shown in Figure 3D, suggests that there exists a weak coupling between the SDW order of FeAs and  $\text{Eu}^{2+}$  localized moments. This result is consistent with NMR (Guguchia et al., 2011), neutron diffraction (Xiao et al., 2009), and ARPES (Zhou et al., 2010) studies on  $\text{Eu}_{122}$ .

## Summary

Magnetic properties of  $\text{Eu}_x\text{Ca}_{1-x}\text{Fe}_2\text{As}_2$  ( $0 \leq x \leq 1$ , ECFA) samples were presented. The resistivity data show clear anomalies due to the SDW and  $\text{Eu}^{2+}$  AFM transitions. The magnetic susceptibility ( $\chi$ ) of ECFA samples exhibits  $1/T$  behavior, which can be explained using the Curie-Weiss law. The  $\chi$  (T) data provide evidence of the  $\text{Eu}^{2+}$  AFM order but do not indicate the SDW transition due to the Fe-moments. A weak transition near  $T_{\text{SDW}}$  appears only after the subtraction of the Curie-Weiss contribution of the  $\text{Eu}^{2+}$  moments. With increasing Eu doping,  $T_{\text{SDW}}$  initially increases rapidly up to  $x \sim 0.4$ , and it then gradually saturates to a value of 190 K at  $x = 1$ . The best-fits of the Curie-Weiss law parameters, the Curie constant ( $C$ ), and the Weiss-temperature ( $\theta$ ), increase linearly with increasing Eu doping, while the effective magnetic moment,  $\mu_{\text{eff}}$  per  $\text{Eu}^{2+}$  ion remains nearly constant. Our results provide detailed information about how the magnetic properties evolve in the Eu-doped  $\text{Ca}_{122}$  system, which will eventually help in understanding superconductivity in ECFA.

## Data availability statement

The raw data supporting the conclusion of this article will be made available by the authors, without undue reservation.

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## Author contributions

BL synthesized high-quality samples of Eu-doped  $\text{CaFe}_2\text{As}_2$ . KS and LD carried out measurements. KS analyzed experimental data and wrote the manuscript. CC supervised the project.

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## Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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