ISSN (Print) 1226-1750
ISSN (Online) 2233-6656
Journal of Magnetics 31(1), 14-20 (2026) https://doi.org/10.4283/JMAG.2026.31.1.14

Mössbauer Spectroscopic Study of Cobalt-doped Superconductor $CaFe_{1-x}Co_xAsF$ (x=0.0, 0.1)

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(Received 10 December 2025, Received in final form 24 January 2026, Accepted 27 January 2026)

We have studied magnetic properties of cobalt-doped and undoped $CaFe_{1-x}Co_xAsF$ (x=0.0, 0.1) by using XRD, EDX, SQUID, and ^{57}Fe Mössbauer spectroscopy through wide temperature range. Polycrystalline samples were synthesized by 2-step solid-state reaction method using high purity Ca, Fe, Co, As and CaF_2 powders. The structural and the magnetic aspects of the observations have been examined by comparing results of the cobalt doped and the parent compounds together. The strategy of Mössbauer spectroscopic analysis has been set in terms of the structural and the spin density wave characteristics, according to which the temperature dependence of hyperfine parameters have been explained. Subsequently, various constituent phases in the materials have been separated out by comparing the results obtained from the SQUID with those from the Mössbauer spectroscopy. It has been found out that undoped CaFeAsF has two different kinds of phase transition happening around ~120 K, about 10 K apart from each other. On the other hand, *M-T* curve of cobalt-doped $CaFe_{0.9}Co_{0.1}AsF$ showed that the spin density wave phase and the superconducting phase coexisted below ~120 K, and finally displayed the onset of the superconductivity at 22 K. Moreover, superconductive $CaFe_{0.9}Co_{0.1}AsF$ at 22 K still showed paramagnetic doublet, indicating there was no abrupt changes in hyperfine parameters observed across either the spin density wave transition or the superconductive transition.

Keywords : mössbauer spectroscopy, spin density wave, superconductivity, CaFeAsF

1. Introduction

Since the discovery of the FeAs-based superconductor $LaFeAsO_{1-x}F_x$, its related materials have been intensively studied to clarify the detailed physics behind them especially about the interplay between superconductivity and magnetism [1, 2]. Especially, the unexpected high critical temperature (T_C) from FeAs-based compounds have attracted much attention of researchers who wanted to obtain higher T_C value by overcoming the conventional electron-phonon coupling mechanism. Undoped CaFeAsF, isostructural with the LaFeAsO, has alternating layers of edge-sharing CaF and FeAs tetrahedra sandwiched along *c*-axis [3]. It is well known that the CaFeAsF undergoes crystallographic transition from tetragonal (P4/nmm) to orthorhombic (Cmma) symmetry across ~120 K [4]. A spin density wave (SDW) transition has also been reported to occur at a temperature T_{SDW} very close to the crystallographic one. Stretching the FeAs square in *a-b* plane into rectangle splits four equal Fe-Fe bond lengths into two groups with two lengthened and two shortened, resulting in a stripe-like magnetic order of small moments on the 2-dimensional FeAs layers, finally initiating the SDW state [5, 6]. This material is paramagnetic above T_{SDW}. Of course, no superconductivity has been observed. It is of interest, however, that 10 % cobalt doping into Fe site in CaFeAsF induces a dramatic change. Both the crystallographic and the SDW transitions were suppressed and a superconductive transition is observed below the T_C of 22 K [7, 8]. The superconductivity is known to emerge on the FeAs layers by adding electrons via doping. Many theoretical and experimental researchers have proved that the FeAs layer plays a critical role of conducting charge whereas CaF layer acts as a charge reservoir [9]. For this reason, the detailed information on physical and/or chemical states of Fe in FeAs-based superconductors are of great importance. Mössbauer spectroscopy is one of the most powerful atomic-scale tools for investigating the electronic state of Fe ions. And there are so many examples of study concerning the Mössbauer spectroscopic

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properties of FeAs-based materials. To the best of our knowledge, however, there are no previous Mössbauer spectroscopic studies of cobalt doped $CaFe_{1-x}Co_xAsF$. In this study, we focus our discussion on the synthesis, structural, magnetic, and Mössbauer spectroscopic properties of undoped and cobalt-doped CaFeAsF compounds. Special attention will be paid to the changes in hyperfine parameters across the superconductive transition and the SDW transition present in the compounds.

2. Experiments

Polycrystalline $CaFe_{1-x}Co_xAsF$ (x=0.0, 0.1) were synthesized through a two-step solid-state reaction introduced in ref. [10]. First, stoichiometric amounts of Ca, Fe and As powders were mixed, annealed at 400 ºC for 10 hours, followed by 700 ºC for 20 hours to prepare CaAs and Fe_2As compounds. These precursors were mixed with stoichiometric amount of CaF_2 powder, pelletized, and annealed in a fused quartz tube at 1000 ºC for 20 hrs. For cobalt doping, we replaced some of Fe to Co in the starting materials resulting in $CaFe_{0.9}Co_{0.1}AsF$. The synthesized sample was ground into a powder in an agate mortar and re-heated under the identical conditions to improve homogeneity. All the starting material preparation procedures were carried out in an N_2-filled glove box (O_2 or $H_2O < 1$ ppm). In order to confirm the crystallographic structure of the prepared samples, x-ray diffraction (XRD) patterns were obtained with Cu $K\alpha$ radiation. In order for confirming the elemental composition of the samples, we performed the energy dispersive X-ray spectroscopy (EDX) analysis using the field emission scanning electron microscopy. The magnetic moment of the samples in the superconducting or non-superconducting regime was measured as a function of temperature using an MPMS (Quantum Design, KBSI, Daejeon). ^{57}Fe Mössbauer spectra throughout a wide temperature range from 16 K to 300 K were obtained using a conventional transmission type spectrometer. A ^{57}Co source (25mCi, RITVERC, Russia) in a Rh matrix was used at room temperature. In order to enhance the temperature-homogeneity, proper amount of sample was mixed with BN powder, sandwiched with thin BN discs and mounted to sample holder. The velocity scale of the spectrometer has been calibrated using α-Fe foil and the reference linewidth was at most 0.31 mm/s.

3. Results and Discussion

Figure 1 illustrates XRD patterns for $CaFe_{1-x}Co_xAsF$ (x=0.0, 0.1) at room temperature. All the peak positions

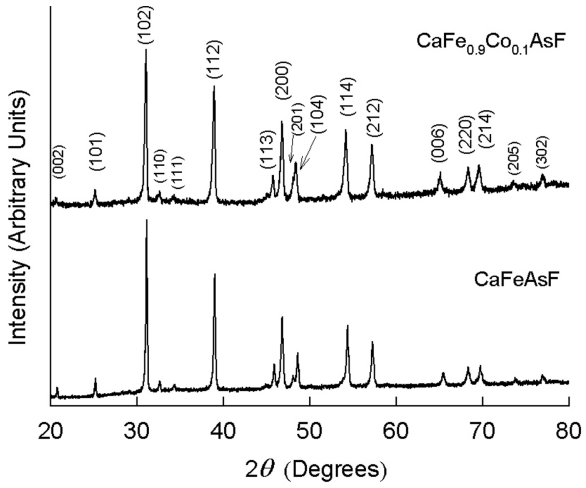

Fig. 1. Powder XRD patterns of $CaFe_{1-x}Co_xAsF$ (x=0.0, 0.1) with (h,k,l) indexed to major peaks.

are in good agreement with those already reported tetragonal phase in the P4/nmm space group (ZrCuSiAs type) without any noticeable trace of impurity [9, 11]. Eleven most intense peak positions marked in Fig. 1 were used to determine the lattice parameters a and c using the well-known interplanar distance relation for tetragonal structure:

$$\frac{1}{d_{hkl}^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \tag{1}$$

along with the Bragg relation $2d_{hkl}\sin\theta = \lambda$ (λ = 1.54056 Å). Least square fitting the above relations with given h,

Table 1. Results of XRD peak position analysis for $CaFe_{1-x}Co_xAsF$ (x=0.0, 0.1). θ_{EX} and θ_{TH} refer to the experimental and the theoretically expected peak positions, respectively.

h	k	l	$CaFe_{0.9}Co_{0.1}AsF$		CaFeAsF	
			$2\theta_{EX}$	$2\theta_{TH}$	$2\theta_{EX}$	$2\theta_{TH}$
1	0	1	25.2	25.2	25.1	25.2
1	0	2	31.1	31.1	31.1	31.0
1	1	2	39.0	39.0	39.0	38.9
1	1	3	45.9	45.8	45.8	45.7
2	0	0	46.8	46.8	46.8	46.8
1	0	4	48.6	48.6	48.4	48.4
1	1	4	54.4	54.4	54.2	54.2
2	1	2	57.3	57.3	57.2	57.2
0	0	6	65.4	65.4	65.1	65.0
2	2	0	68.3	68.3	68.3	68.3
2	1	4	69.8	69.7	69.6	69.5
a (Å)			3.8789		3.8826	
c (Å)			8.5510		8.5958	

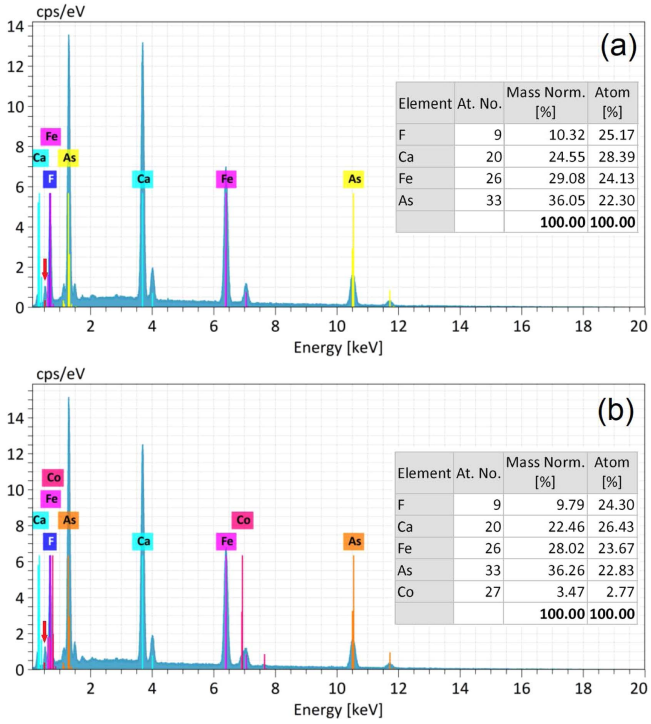

Fig. 2. (Color online) Results of EDX analysis for (a) undoped CaFeAsF and (b) $CaFe_{0.9}Co_{0.1}AsF$. Peaks with small red arrow at ~0.5 keV correspond to oxygen (> 2 atom %).

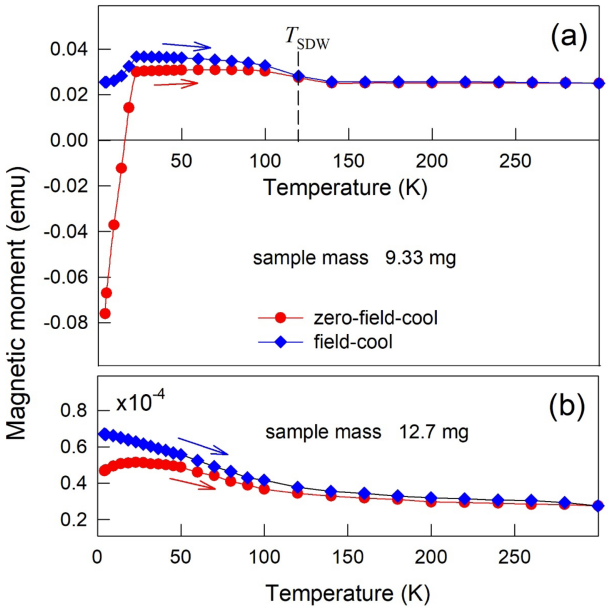

Fig. 3. (Color online) FC and ZFC magnetization measured as a function of temperature for (a) cobalt-doped $CaFe_{0.9}Co_{0.1}AsF$ and (b) undoped CaFeAsF. Arrows mean measurements were done with increasing temperature. Note the extremely small scale of magnetization for undoped CaFeAsF.

k, l and θ values, the optimum lattice parameters are found to be a = 3.8826 Å, c = 8.5958 Å for undoped sample and a = 3.8789 Å, c = 8.5510 Å for Co-doped sample, respectively (See Table 1). Significant shrink of c compared to a with doping of cobalt agrees well with previous results, which already reported that the Co-doping in Fe site induced considerable change in c whereas a is insensitive to the doping [3, 4]. Absence of noticeable satellite peaks near the strongest (102) peak and near 50° suggests that main phases of these samples are free from impurities such as FeAs, Fe_2As or $FeAs_2$. The EDX spectra in Fig. 2 indicated that the chemical compositions of the polycrystalline samples approximately satisfy the formulas but there is a tiny trace of oxidation (arrows in the figures) due to possible penetration of minute oxygen through quartz tube.

Figure 3(a) shows the temperature dependence of the zero-field-cooled (ZFC) and the field-cooled (FC) magnetizations of $CaFe_{0.9}Co_{0.1}AsF$ obtained under the external magnetic field of 100 Oe. Magnetization shows little change above 120 K. Below 120 K, however, an increase in magnetization appears indicating that magnetic moment is induced by the applied magnetic field. This temperature is very close to what is known as the SDW transition temperature (T_{SDW}) of the mother phase CaFeAsF. In fact, this kind of magnetic component in cobalt-doped $CaFe_{0.9}Co_{0.1}AsF$ has been reported by several studies. Matsuishi *et al.* ascribed it to a small amount of ferromagnetic impurity such as FeAs:Co [3]. Zhang *et al.* also observed the same trace of SDW state in the $CaFe_{0.9}Co_{0.1}AsF$. But he attributed it to a phase separation originating from cobalt inhomogeneity [11]. Since the superconducting volume fraction of our sample is estimated to be low, it can be supposed that this magnetic component below 120 K is coexisting as a cobalt-containing but non-superconducting impurity. This will be discussed below in relation with Mössbauer spectroscopic result. With further lowering the temperature, magnetization shows drastic drop and even goes negative so diamagnetism is finally observed at 22 K, indicating this point is the superconducting onset temperature T_C. This temperature is consistent with other studies of resistivity measurement [3, 11]. The temperature dependence of the zero-field-cooled (ZFC) and the field-cooled (FC) magnetizations of undoped CaFeAsF (Fig. 3(b)) shows the bifurcation behavior indicating a weak antiferromagnetism.

^{57}Fe Mössbauer spectra of the $CaFe_{0.9}Co_{0.1}AsF$ powder taken at several temperatures are illustrated in Fig. 4 in which all the spectra seemingly have paramagnetic singlet. However, as the symmetry of the surroundings of

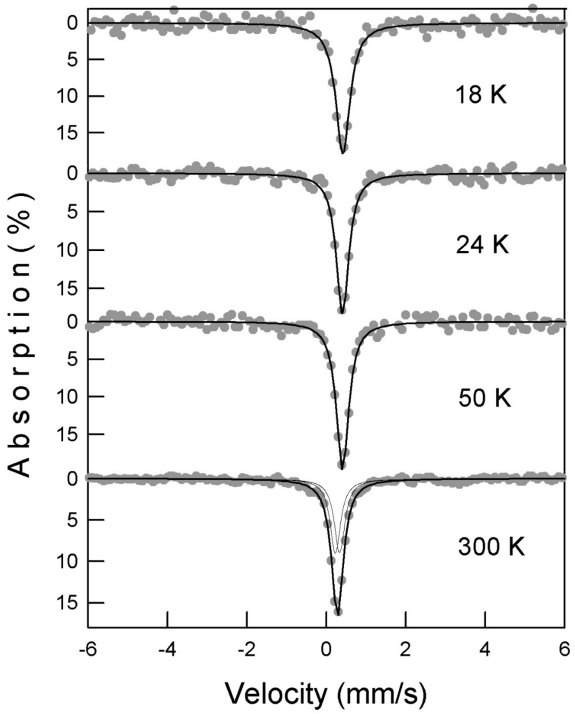

Fig. 4. ^{57}Fe Mössbauer spectra for cobalt-doped $CaFe_{0.9}Co_{0.1}AsF$ taken at some temperatures. Component spectra for doublet are displayed only for 300 K.

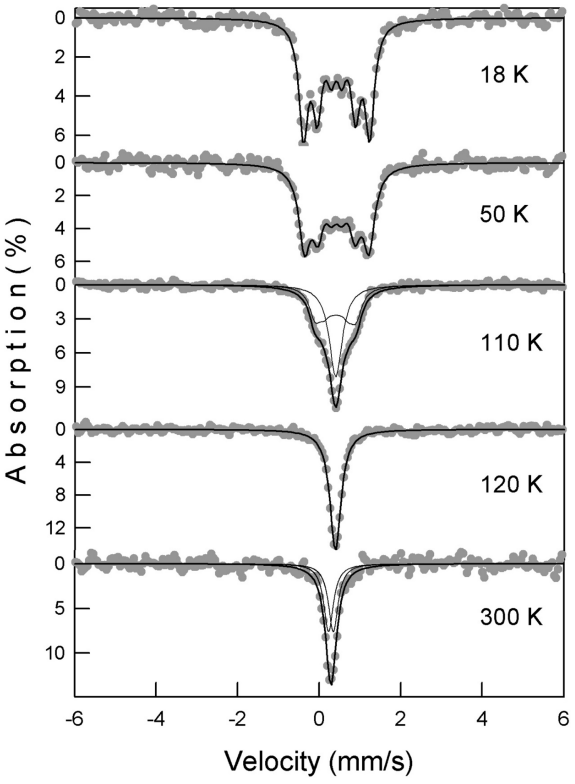

Fig. 5. ^{57}Fe Mössbauer spectra for parent composition CaFeAsF taken at some temperatures. Component spectra for doublet are displayed only for 300 K.

Table 2. Hyperfine parameters of $CaFe_{1-x}Co_xAsF$ (x=0.0, 0.1) for selected temperatures. H_{hf}, E_Q, δ, and Γ refer to the hyperfine magnetic field, the electric quadrupole splitting, the isomer shift, and the full linewidth at half maximum.

T (K)	Parameters	$CaFe_{0.9}Co_{0.1}AsF$	CaFeAsF
18	H_{hf}(T)	0	5.1
	E_Q(mm/s)	0.12	0.00
	δ (mm/s)	0.40	0.42
	Γ(mm/s)	0.32	0.36
120	E_Q(mm/s)	0.12	0.11
	δ (mm/s)	0.38	0.40
	Γ(mm/s)	0.30	0.31
300	E_Q(mm/s)	0.11	0.12
	δ (mm/s)	0.28	0.29
	Γ(mm/s)	0.30	0.29

Fe ion is tetragonal, we interpret the spectra as closely spaced quadrupole doublet. There are no discernable traces of foreign phases as were detected from other study [12]. Some selected hyperfine parameters are summarized in Table 2. No abrupt change of the spectra stemming from the SDW transition is observed throughout the whole temperature range examined. Moreover, there are no anomalies in any of hyperfine parameters across the onset temperature of superconductivity (T_C = 22 K), which is in line with the features of other FeAs-based compounds [13, 14, 15]. Fig. 5 depicts typical ^{57}Fe Mössbauer spectra of the parent composition CaFeAsF which doesn't show the superconductivity. In the case of this undoped sample, the spectra below 110 K consist of single set of hyperfine sextet indicative of the SDW magnetic order in the sample. Those spectra were least square fitted to 6 Lorentzian lines under the restraints in peak positions that hold when the electric quadrupole interaction is much weaker than the magnetic hyperfine interaction [16]. Above 120 K, on the other hand, the spectra are singlets again. In view of the facts of both the broad linewidth and the non-cubic crystal symmetry of the sample at those temperatures, it would be more plausible to interpret it as a quadrupole doublet [5]. The resultant hyperfine parameters are listed in Table 2.

Figure 6(a) shows the temperature dependence of the magnetic hyperfine field $H_{hf}(T)$ for the undoped CaFeAsF. Magnetic hyperfine field at 18 K is 5.1 T and decreases with increasing temperature until the magnetic ordering disappears at 120 K. At 110 K which is slightly below the transition temperature, the magnetic sextet pattern with H_{hf} = 3.1 T and the paramagnetic component (possibly a doublet) coexist and overlap in the spectrum

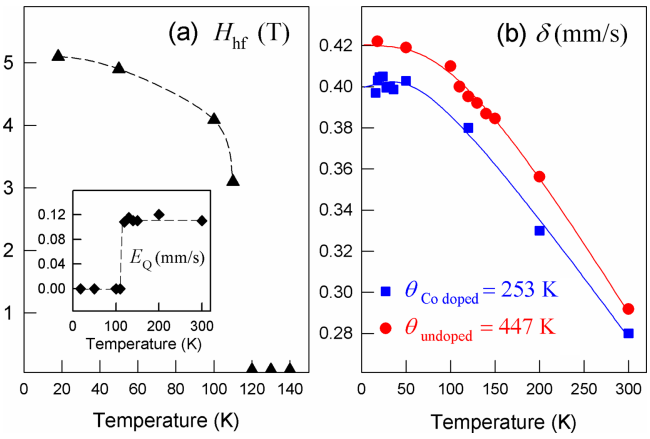

Fig. 6. (Color online) Temperature variations of Mössbauer parameters for $CaFe_{1-x}Co_xAsF$ (x=0.0, 0.1). (a) H_{hf}, E_Q, and (b) δ refer to the hyperfine magnetic field, the electric quadrupole splitting, the isomer shift, respectively. Dashed lines in (a) are just for eye-guide, solid lines in (b) is the optimum results of least square fitting.

(see Fig. 5). So far, many researchers have taken the magnetic SDW transition and the structural tetragonal-orthorhombic transition to happen simultaneously. But our result indicates that there should be two separate phase transitions; one above 110 K and the other below 110 K. Actually, Terashima *et al*. has reported two clearly separated resistivity anomalies at 118 K and 107 K, which were attributed to a structural and a magnetic transition, respectively [17]. Regarding the order of phase transition, many prior studies support above phase transitions are of second-order [5, 18] while others claim that it is of first-order [14, 19]. At the present stage, our data are not sufficient to assert the order of the phase transition. On the other hand, the Mössbauer spectra for the cobalt doped $CaFe_{0.9}Co_{0.1}AsF$ exhibit only the paramagnetic component throughout the whole temperature range examined (see Fig. 4). This seemingly contradicts to the magnetization measurement mentioned above in which a magnetic component was apparently induced by external magnetic field below 120 K. In fact, many publications referred to the existence of the phase separation or the coexistence of magnetic and superconducting phases in underdoped $CaFe_{1-x}Co_xAsF$ ($x < 0.06$) [4, 20] or in other FeAs-based compounds [5, 21, 22]. It seems that our cobalt doped sample is not sufficiently homogeneous that the under-doped phase ($x < 0.1$) coexists with $CaFe_{0.9}Co_{0.1}AsF$. We should further clarify why both phases give rise to the paramagnetic doublet and cannot be separated by Mössbauer spectroscopy in the future study. The inset of Fig. 6(a) shows the temperature dependence of the quadrupole splitting E_Q of the undoped CaFeAsF. As can also be seen from Table 2, however, the low temperature sextets spectra of CaFeAsF were fitted well with zero E_Q despite the orthorhombic crystal structure while we can observe doublet of non-zero E_Q above 120 K. This discordance can be explained as follows: when there exists magnetic order, E_Q could be averaged to zero as the principal axes of EFG tensor could take any direction with respect to hyperfine magnetic field. Even though the average quadrupole splitting E_Q is zero, the line broadening $\Delta\Gamma$ due to this random orientational distribution is not. Taking the quadrupole splitting of 0.11 mm/s above T_{SDW} = 120 K into account, we can calculate the line broadening below 120 K as:

$$\Delta\Gamma = [(E_Q - \langle E_Q \rangle)^2]^{\frac{1}{2}}$$

$$= 2\left[\frac{1}{4\pi}\int_0^{\pi}\int_0^{2\pi}\frac{e^4q^2Q^2}{64}(3\cos^2\theta - 1 + \eta\sin^2\theta\cos 2\phi)^2\sin\theta\, d\theta\, d\phi\right]^{\frac{1}{2}}$$

$$= \frac{1}{\sqrt{5}}\frac{e^2qQ}{2}\left(1+\frac{\eta^2}{3}\right)^{\frac{1}{2}}$$

$$= \frac{1}{\sqrt{5}}0.11$$

$$= 0.045 \text{ mm/s} \qquad (2)$$

Actually, the linewidth of the sextets below 110 K is about 0.36 mm/s (see Table 2) which is approximately the linewidth of a-Fe foil plus the 0.045 mm/s. Fig. 6(b) shows the isomer shift $\delta(T)$ as a function of temperature for $CaFe_{1-x}Co_xAsF$ (x=0.0, 0.1). The room-temperature isomer shift values correspond to the low-spin Fe(II) state [6, 13]. In fact, low-spin ferrous ion has effectively zero magnetic moment. However, this ionic picture doesn't seem to be appropriate because this material possesses the itinerant electron paramagnetism [15, 23]. So, the isomer shifts of $CaFe_{1-x}Co_xAsF$ (x=0.0, 0.1) can be attributed to those of the covalent compounds with itinerant electrons. As can be seen in the figure, the isomer shift of undoped sample is always higher than that for Co-doped sample for all temperatures. Cobalt doping apparently reduces the isomer shift. When the unit cell volume shrinks by the cobalt doping, the strong compression of $FeAs_4$ tetrahedra reduces the screening of localized 3d electron to s-electron at the nucleus. This will increase s-electron density at Fe nucleus, finally resulting in smaller isomer shift [6, 13, 19]. Center shift of a Mössbauer spectrum is the sum of the effects from s-electron density at the Fe nucleus (chemical shift) and the temperature dependent 2nd order Doppler effect. At high temperature region, our

isomer shift $\delta(T)$ decreases linearly with its slope of about -0.0006 mm/s·K, which is very close to the value theoretically anticipated when applying the classical Dulong-Petit law; $d\delta/dT = -3R/2Mc$ [24]. Here, R is the thermodynamic gas constant and M is the mass of Mössbauer atom. Assuming the harmonic atomic forces with the Debye model, isomer shift can be expressed as [25]:

$$\delta(T)=a+d\cdot T-\frac{9k_B\Theta_D}{16M_C}\times\left[1+8\left(\frac{T}{\Theta_D}\right)^4\int_0^{\Theta_D/T}\frac{x^3}{e^x-1}dx\right] \quad (3)$$

where Θ_D is the Debye temperature and a, d and Θ_D are obtained by the least square fitting of the experimental isomer shifts to above equation. Debye temperatures were found out to be 447 K and 253 K for x = 0 and 0.1, respectively, which indicates that the cobalt doping into the FeAs layer weakens the atomic binding and softens the materials. Similar values were obtained from previous studies of FeAs-based compounds [13, 19, 22]. Finally, a close look at Fig. 6(b) reveals that no significant change in the isomer shift takes place either across the superconducting transition (22 K) or across the SDW transition (120 K).

4. Conclusion

In this study, we have synthesized FeAs-based superconductor system $CaFe_{1-x}Co_xAsF$ (x=0, 0.1) and have examined their structural and magnetic aspects of cobalt doping by using XRD, EDX, SQUID magnetometer and Mössbauer spectroscopy. XRD analysis showed that the samples were tetragonal phase in the P4/nmm space group (ZrCuSiAs type) without any noticeable trace of impurity. EDX analysis indicated that the chemical compositions of the polycrystalline samples approximately satisfied the formulas. Temperature dependences of the zero-field-cooled (ZFC) and the field-cooled (FC) magnetizations of $CaFe_{0.9}Co_{0.1}AsF$ have shown a trace of the SDW state below 120 K as well as the superconductive transition at 22 K. Mössbauer spectra of the parent phase CaFeAsF showed the SDW transition at 120 K. But just below the temperature, magnetic sextet and paramagnetic doublet coexisted at 110 K, meaning that the structural and the magnetic phase transitions occur at slightly different temperatures. Mössbauer spectra of $CaFe_{0.9}Co_{0.1}AsF$ show paramagnetic doublet all through the temperature range. No abrupt changes in hyperfine parameters were observed across the SDW transition and superconductive transition.

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