PAPER

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Absence of low energy magnetic spin-fluctuations in isovalently and aliovalently doped $LaCo_2B_2$ superconducting compounds

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Abstract

Magnetization, resistivity and ^{11}B, ^{59}Co NMR measurements have been performed on the Pauli paramagnet $LaCo_2B_2$, and the superconductors $La_{0.9}Y_{0.1}Co_2B_2$ ($T_c \approx 4.2$ K) and $La(Co_{0.7}Fe_{0.3})_2B_2$ ($T_c \approx 5.8$ K). The site selective NMR experiment reveals the multiband nature of the Fermi surface in these systems. The temperature independent Knight shift and $1/T_1T$ clearly indicate the absence of correlated low energy magnetic spin-fluctuations in the normal state, which is in contrast to other Fe-based pnictides. The density of states (DOS) of Co 3*d* electrons has been enhanced in superconducting $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$ with respect to the non superconducting reference compound $LaCo_2B_2$. The occurrence of superconductivity is related to the DOS enhancement.

Keywords: superconductivity, nuclear magnetic resonance, low energy spin-fluctuations

(Some figures may appear in colour only in the online journal)

1. Introduction

The origin of superconductivity in unconventional superconductors has been one of the most interesting and debated subjects, in the last few decades, by theoreticians and experimentalists. Many experimental tools and theoretical ideas have been employed to investigate and analyze the normal state and the superconducting state to understand the pairing mechanism for such transitions. BCS theory which predicts a dominant role of phonons in transforming a metal to a superconductor, cannot satisfactorily explain the same in compounds where the magnetic spin-fluctuations are present in the normal state. In cuprates [1] and iron pnictides [2], superconductivity has been achieved upon chemical and physical pressure/carrier injection. Further the magnetic spin-fluctuations are not fully suppressed even in the superconducting state over a certain concentration range of doping. Though the iron pnictide superconductors have much lower superconducting transition temperature, T_c, than the cuprates, the complex interplay between several competing interactions in the former offer a fertile playground to gain further insights into the co-operative phenomena in high T_c superconductors.

In Fe pnictide superconducting families (namely the 1 1 1 1, 1 2 2, 1 1, 1 1 1 family etc) it became a general belief that low energy antiferromagnetic (AFM) spin-fluctuations due to the nesting of electron and hole Fermi surfaces are the driving force for superconductivity, though the importance of orbital fluctuations was also found [3–5]. Also, the $FeAs_4$ tetrahedral symmetry was found to be an important issue for the high-T_c values achieved [6–9]. In the 1 1 1 1 family the isotope effect on the superconducting transition temperature T_c was observed, indicating the importance of phonon contribution in superconductivity [10]. In the case of an Fe-P based superconductor, namely LaFePO, the presence of weak 2D

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ferromagnetic (FM) spin-fluctuations were found in the normal state [11]. However, the Co based non-superconducting analogue LaCoPO happens to be an itinerant ferromagnet where the 2D FM spin-fluctuations are present in the paramagnetic state [12, 13]. Recently a Co based superconducting family ($LaCo_2B_2$) with the same crystal structure as the Fe based pnictides, has been discovered by Hosono's group [14]. However, the Co based pnictide $LaCo_2P_2$ is an itinerant ferromagnet [15]. This raises the following questions: (1) are ferromagnetic spin-fluctuations also present in superconducting Y(Fe) doped $LaCo_2B_2$? similar to that in LaFePO and (2) due to the same crystal structure as that of the 1 2 2 pnictides, are antiferromagnetic spin-fluctuations present due to Fermi surface nesting?

NMR (nuclear magnetic resonance) is a powerful technique to probe the static and dynamic properties of electrons and has been considered as an indispensable tool in understanding the paring mechanism and related phenomena in high T_c superconductors. The NMR Knight shift probes the local static spin susceptibility (at $q = 0$) whereas the nuclear relaxation time can probe q-averaged dynamical spin-susceptibility. Therefore, the presence of low energy spin-fluctuations and also its nature in the Y(Fe) doped $LaCo_2B_2$ superconducting system can be probed by NMR. Further the NMR in a nucleus with $I > 1/2$ can also give information about the local crystal symmetry due to quadrupolar interaction which further helps to understand whether there is any role of tetrahedral symmetry on superconductivity. In this manuscript, we present the results of the detail ^{11}B ($I = 3/2$)) and ^{59}Co ($I = 7/2$) NMR studies in Pauli paramagnetic $LaCo_2B_2$ and superconducting $La_{0.9}Y_{0.1}Co_2B_2$ ($T_c \approx 4.2$ K) and $La(Co_{0.7}Fe_{0.3})_2B_2$ ($T_c \approx 5.8$ K) compounds. Furthermore, probing two different nuclei situated at different positions in the unit cell would help us to obtain local electromagnetic information from the different portions of the electronic structure.

2. Experimental details

Polycrystalline $LaCo_2B_2$, $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$ were synthesized in an arc furnace (M/s Centorr Vacuum Industries, USA) using a water cooled copper hearth under flowing argon atmosphere. Stoichiometric amounts of high purity La (>99.5%), Co (>99.95%), Fe (99.5%) and B (>99.5%) from Alfa Aesar were melted together to synthesize the respective compositions. After each melting, the samples were flipped and remelted for a total of six times to ensure compositional homogeneity. The weight loss during the melting process was less than 0.3 wt.% for all the compounds. Finally, the as-cast buttons were wrapped in tantalum foils, sealed under vacuum in a quartz capsule and annealed at 1000 °C for 10 d.

X-ray diffraction (XRD) measurements at room temperature were performed in a 9 kW rotating anode based diffractometer (TTRAX III, M/s Rigaku Corporation, Japan) using CuKα radiation. Although most of the diffraction peaks for all the three compounds, viz., $LaCo_2B_2$, $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$, could be indexed with a tetragonal $ThCr_2Si_2$ structure type (space group: I4/mmm), a few unindexed peaks

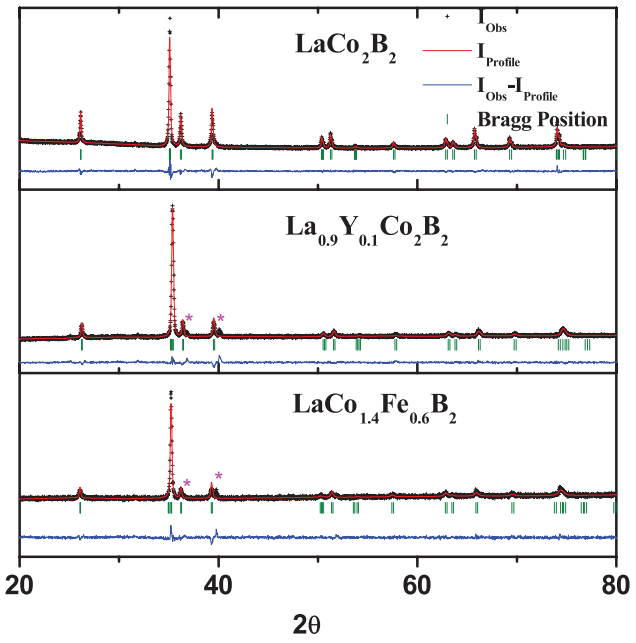

Figure 1. Rietveld refinement of XRD patterns for $LaCo_2B_2$, $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$.

of weak intensity (~7–8%) suggest the presence of minor secondary phases (figure 1). The lattice parameters were estimated using FULLPROF software [16]. The lattice parameters for $LaCo_2B_2$, $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$ are $a = 3.624$ Å, $b = 10.24$ Å; $a = 3.619$ Å, $b = 10.18$Å; $a = 3.641$ Å, $b = 10.15$ Å, respectively.

The magnetic moment was measured in a PPMS (9 T, Quantum Design) multipurpose device. The resistivity was obtained using a four probe method at zero field in a cryogen free magnet from M/S Cryogenics Limited and the temperature variation study was performed using a close cycle refrigerator, also from M/S Cryogenics Limited.

The ^{11}B- and ^{59}Co-NMR measurements were performed using a conventional phase-coherent pulse spectrometer (Thamway PROT 4103MR) with a 7.0 T superconducting magnet (Bruker) and standard pulsed NMR spectrometer (Tecmag) at a frequency of 45 MHz. The spectrum was recorded by changing the frequency (for ^{11}B NMR) and field (for ^{59}Co NMR) step-by-step and measuring the spin echo intensity by applying a $\pi/2 - \tau - \pi$ solid echo pulse sequence. The spin lattice relaxation time (T_1) was measured using the saturation recovery method by exciting the central transition using a 10μs $\pi/2$ pulse. To obtain the T_1 values $y = (1/10)\exp(-t/T_1) + (9/10)\exp(-6t/T_1)$ for B($I = 3/2$) and $y = (0.0119)\exp(-t/T_1) + (0.0682)\exp(-6t/T_1) + (0.2060)\exp(-15t/T_1) + (0.7139)\exp(-28t/T_1)$ for Co ($I = 7/2$) recovery functions have been used.

3. Results

3.1. Magnetization and resistivity measurements

Figure 2(a) shows the temperature dependence of magnetization (FC and ZFC) at 50 Oe indicating an usual superconducting

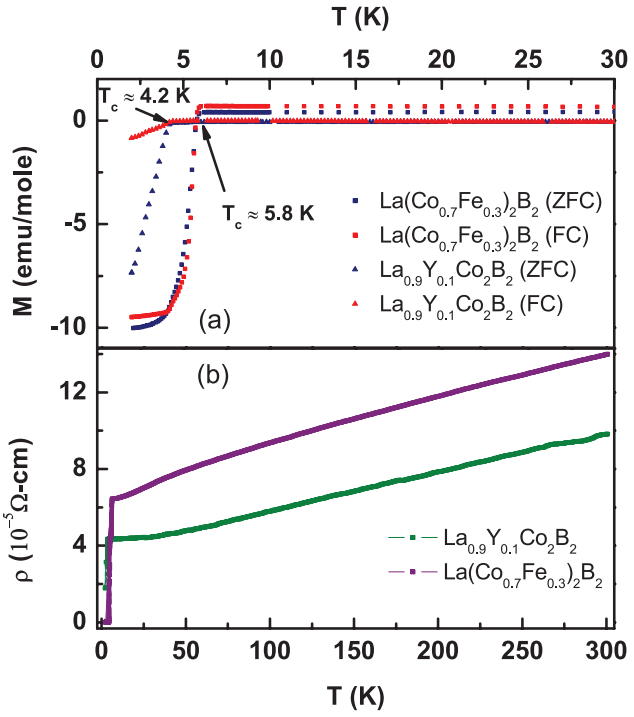

Figure 2. Temperature dependence of the magnetization at 50 Oe. (b) Temperature dependence of resistivity for $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$.

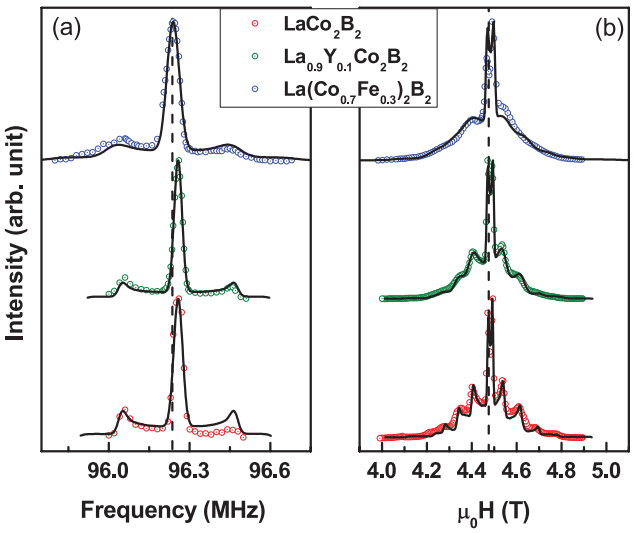

Figure 3. (a) ^{11}B NMR spectra at 300 K at the field of 7 T in $LaCo_2B_2$, $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$. (b) ^{59}Co NMR spectra at 4.3 K at 45 MHz in $LaCo_2B_2$, $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$. Dashed lines are the reference frequency/field. The solid lines show the best spectrum simulation for the powder pattern.

Table 1. Parameters estimated by simulated theoretical curve for the powder pattern of ^{11}B NMR at 300 K.

Compound	LW (kHz)	ν_Q (MHz)	K_{iso}(%)
$LaCo_2B_2$	33.15 ± 0.1	0.43 ± 0.05	0.025 ± 0.005
$La_{0.9}Y_{0.1}Co_2B_2$	33.15 ± 0.1	0.43 ± 0.05	0.025 ± 0.005
$La(Co_{0.7}Fe_{0.3})_2B_2$	44.55 ± 0.1	0.46 ± 0.1	0.008 ± 0.005

transition below 4.2 K and 5.8 K in $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$, respectively. Resistivity at zero field (figure 2(b)) also shows the superconducting transition for both the samples, and in the normal state it shows metallic nature. The magnitude of resistivity is similar to that of the reported results [14].

3.2. ^{11}B and ^{59}Co NMR

3.2.1. ^{11}B and ^{59}Co NMR spectra. Figures 3(a) and (b) show the ^{11}B and ^{59}Co NMR spectra in $LaCo_2B_2$, $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$. All the spectra show a typical nature expected for a nuclear spin 3/2 (for B) and 7/2 (for Co), where the spectra consists of one central line and two satellites (six satellites) for ^{11}B (^{59}Co) due to the existence of nonzero electric field gradient at the B and Co nuclear sites for the non-cubic (tetragonal) crystal structure. The symmetric ^{11}B central transition indicates that at the B site, local magnetic field is isotropic in nature for all the samples.

The best spectrum simulation for the powder pattern of the ^{11}B and ^{59}Co NMR lines (represented by the continuous line in figure 3) by considering the effect of magnetic and electrostatic interactions, revealed that the splitting of the ^{59}Co NMR central transition is due to the anisotropy in the local magnetic field. All the parameters estimated from simulations have been shown in tables 1 and 2. The negative shift seen in ^{59}Co NMR spectra is due to the core polarization and the anisotropy increased to some extent from $LaCo_2B_2$ to $La_{0.9}Y_{0.1}Co_2B_2$ to $La(Co_{0.7}Fe_{0.3})_2B_2$.

Further, the central transition also shows a slight broadening due to the weak distribution of local magnetic fields arising from substitution as seen from ^{11}B and ^{59}Co NMR. We did not find any temperature dependencies of the shift in all the three systems from ^{11}B and ^{59}Co NMR which indicates no magnetic correlations were developed in contrast to LaFePO [11] (with a similar value of T_c), where weak FM correlations were observed via the weak temperature dependence of shift in the normal state. Furthermore, our study indicates the absence of any pseudogap opening as was observed in Fe pnictide 1 2 2 superconductors [3, 4]. These observations ruled out the possibilities of ferromagnetic correlations and the pseudogap to be the driving forces for superconductivity in substituted $LaCo_2B_2$ superconducting compounds.

The estimated quadrupolar coupling constants (ν_Q) at B and Co sites are nearly similar for all the systems (tables 1 and 2). Thus the crystal symmetry is not changed much due to the substitution at the La or Co site.

The substitution is found to introduce broadening (tables 1 and 2) in the satellite lines with respect to that in the pure sample and is highest for $La(Co_{0.7}Fe_{0.3})_2B_2$. The broadening of the satellite indicates a distribution of electric field gradient due to substitution. Such a distribution was often seen in several substituted systems [17–19]. The observed effect of EFG distribution due to the substitution is higher at the Co site than that at the B site. In the present systems the EFGs at the B and the Co sites are higher than those at the As and Co sites in Fe pnictide based 1 2 2 superconductors [20]. This supports the fact that in the present systems the CoB_4 tetrahedra is more

Table 2. Parameters estimated by simulated theoretical curve for the powder pattern of ^{59}Co NMR at 4.3 K.

Compound	LW (kHz)	ν_Q(MHz)	K_{iso}(%)	K_{ax}(%)
$LaCo_2B_2$	57.75 ± 0.1	1.46 ± 0.1	-0.162 ± 0.005	-0.139 ± 0.005
$La_{0.9}Y_{0.1}Co_2B_2$	72.65 ± 0.1	1.47 ± 0.1	-0.162 ± 0.005	-0.154 ± 0.005
$La(Co_{0.7}Fe_{0.3})_2B_2$	99 ± 0.1	1.6 ± 0.15	-0.2 ± 0.005	-0.186 ± 0.005

distorted than that of $FeAs_4$ tetrahedra because of the deviation of bond angles from perfect tetrahedra. The negligible difference in the quadrupolar coupling constants for the superconducting samples compared to the pure one, indicates that the symmetry of the crystal structure might not be related to the superconductivity in this systems whereas $FeAs_4$ tetrahedral symmetry has often been connected to the superconductivity in Fe based superconductors. But it should be pointed out that with doping due to the broadening of satellites, the exact estimation of ν_Q was not possible.

3.2.2. Nuclear spin-lattice ($1/T_1$) relaxation rate. Nuclear spin-lattice ($1/T_1$) relaxation rate measurements are performed in all the three samples both at B and Co sites. In the case of Fe pnictide superconductors belonging to the 1 2 2 series, the presence of magnetic spin-fluctuations with its role on superconductivity were observed from nuclear relaxation rates along with the evidence of pseudogap in the normal state. In order to explore the presence of $q \neq 0$ (AFM) or $q = 0$ (FM) spin-fluctuations we measured $1/T_1$ which probes the q averaged dynamical spin-susceptibility.

In general, $(1/T_1T)_{SF}$ is given by

$$(1/T_1T)_{SF} \propto \sum_q |H_{hf}(q)|^2 \chi_\perp''(q, \omega_n)/\omega_n \quad (1)$$

where the sum is over the wave vectors q within the first Brillouin zone, $\chi_\perp''(q, \omega_n)$ is the imaginary part of the transverse dynamical electron spin susceptibility, γ_n and ω_n are the nuclear gyromagnetic ratio and Larmor frequency. $H_{hf}(q)$ is the q dependent hyperfine coupling constant.

The hyperfine form factors for the Co and B sites are the followings [21, 22]

$$|^{59}H_{hf}(q)|^2 = |A + 2B(Cos(q_x a) + Cos(q_y b))|^2 \quad (2)$$

$$|^{11}H_{hf}(q)|^2 = |4C(Cos(q_x a/2)Cos(q_y b/2))|^2 \quad (3)$$

where a is the distance between the Co sites and A, B and C are the hyperfine couplings. At the Co site since A (on-site hyperfine coupling) <0 and B (transferred coupling) >0, then for some intermediate wave vectors $|^{59}H_{hf}(q)|^2 = 0$ which indicates that for those wave vectors $^{59}(1/T_1T)$ will be insensitive whereas $^{11}(1/T_1T)$ will not be insensitive for those wave vectors. Furthermore $|^{11}H_{hf}(q)|^2 = 0$ for wave vectors q first Brillouin zone boundary will be insensitive for commensurate AFM modes. Thus by measuring T_1 both at the Co and B site one can have the idea about the spin-fluctuations at the different portions of the Brillouin zone.

The temperature independent behaviour of $1/T_1T$ and shift in the pure compound from ^{11}B and ^{59}Co NMR indicate it to be a Pauli paramagnet (figure 4 and inset of figure 4).

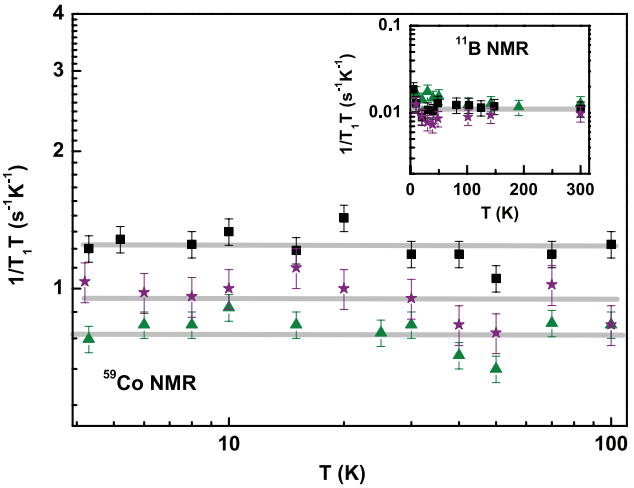

Figure 4. Temperature dependence of $^{59}(1/T_1T)$ for $LaCo_2B_2$ (triangle), $La_{0.9}Y_{0.1}Co_2B_2$ (star) and $La(Co_{0.7}Fe_{0.3})_2B_2$ (square). Inset shows $^{11}(1/T_1T)$ versus temperature.

Additionally for the superconducting compounds in the normal state we have found that $^{11}(1/T_1T)$ and $^{59}(1/T_1T)$ do not have any temperature dependence (figure 4). The temperature independence indicates that there are no ferromagnetic or antiferromagnetic spin-fluctuations in the normal state. In the case of K, Co and P doped $BaFe_2As_2$, strong antiferromagnetic spin-fluctuations were seen [3, 23, 24] from NMR studies in contrast to the Co based boride 1 2 2 superconductors. The AFM spin-fluctuations in the arsenides are due to the nesting of the Fermi surface but in Fe doped $LaCo_2B_2$, the Fermi surface nesting at a wave vector responsible for AFM spin-fluctuations is ill defined according to ARPES results which further supports our NMR findings for the absence of any magnetic spin-fluctuations.

In general in a system with no magnetic spin-fluctuations, the total $1/T_1T$ can be written as $1/T_1T = (1/T_1T)_s + (1/T_1T)_{orb}$, where the first term is due to the conduction electrons (which is proportional to the square of the density of states of s conduction electrons) and the second term is the orbital contribution (which is proportional to the square of the density of states of non-s electrons) to the nuclear spin-lattice relaxation rate. The magnitude of temperature independent $^{11}(1/T_1T)$ do not have any change with substitution which indicates that B orbitals have no role in inducing superconductivity in these systems. Interestingly, the magnitude of $^{59}(1/T_1T)$ increases in $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$ with respect to $LaCo_2B_2$, which indicates that Co 3d orbitals have a role in superconductivity unlike B 2p orbitals.

The ratio $(1/T_1T)/(\gamma_N)^2$ is two orders of magnitude higher at the Co site than that at the B site, which indicates that the Co 3d orbitals mainly form the Fermi level compared to the B

2*p* orbitals. The different values further indicate that the participation of B 2*p* electrons are different than the participation of Co 3*d* electrons at the Fermi level which is a signature of a multiband system. These findings are in agreement with those reported from ARPES measurements [25]. The interesting point to make is that even if B orbitals participate in the Fermi surface they do not have any role in inducing superconductivity, whereas Co 3*d* have.

4. Conclusion

Magnetization and resistivity data show that $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$ undergo a superconducting transition at $T_c \approx$ 4.2 K and 5.8 K, respectively. The site selective NMR experiment dictates a multiband nature of the Fermi surface in these systems. The temperature independent Knight shift and $1/T_1T$ signals clearly the absence of correlated low energy magnetic spin-fluctuations in the normal state, which is in contrast to other Fe-based pnictides. The density of states of Co 3*d* electrons have been increased in superconducting $La_{0.9}Y_{0.1}Co_2B_2$ and $La(Co_{0.7}Fe_{0.3})_2B_2$ with respect to the pure one and responsible for inducing superconductivity, whereas B 2*p* orbitals are unimportant. Our findings will instigate more experiments which can probe DOS specially on the Y doped superconductors to further confirm our findings. It would also be interesting to study the superconducting state to understand the pairing mechanism, which might be different from the Fe-pnictides because of the absence of magnetic spin-fluctuations. Furtheremore, one should not neglect the role of the phonon to the pairing in the $LaCo_2B_2$ superconducting family.

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