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Electron Spin Resonance of Conduction Electrons and Spin Waves Dynamics Investigations in Bi-2223 Superconductor for Decoding Pairing Mechanism

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***Abstract*—**Electron spin resonance (ESR) spectroscopic investigations of (Bi, Pb)$_2$Sr$_2$Ca$_2$Cu$_3$O$_{10-x}$ (Bi-2223) bulk samples were carried out in both the normal and superconducting states. A broad asymmetric resonance signal with side signals is obtained in the normal state, and all of them disappear in the superconducting state. The temperature and angular orientation effects on these signals suggest that the broad asymmetric signal arises from electron spin resonance of conduction electrons (CESR) and the side signals from exchange interactions as Platzman-Wolff type spin waves. The disappearance of CESR and spin waves in a superconducting state demonstrates the role of exchange interactions in Cooper pair formation.

***Keywords*—**Bi-2223 superconductor, electron spin resonance of conduction electrons, electron spin resonance, Exchange interactions, spin waves.

I. INTRODUCTION

THE Bi$_2$Sr$_2$Ca$_2$Cu$_3$O$_{10-x}$ (Bi2223) superconducting compounds with critical transition temperature (Tc ~ 115 K) have the capability of attaining very high critical current density (Jc value about 10^5 A/cm^2). Their swift and consistent superconductor/normal state transition helps in limiting current peaks in grid lines and make them suitable materials for very promising electric power applications. These compounds have triple Cu-O layers. ESR investigations of Bi-based superconductor are mostly based on Cu^{2+}($3d^9$) paramagnetic ions [1]-[7] containing impurity phases and/or oxygen deficient or disordered CuO planes. Although these Bi-based superconductors consist of complex metal oxides, it is well known that they do have CuO conduction planes from hybridized 3d Cu^{2+} and 2p O^{2-} levels, therefore, are expected to show ESR signal due to conduction electrons (conduction electron spin resonance, CESR). However, very limited work is available on CESR. So far only Hayashi et al. [8], [9] and Shatiel et al. [10] analyzed ESR spectra in context to CESR signal in such systems.

In this work, we present ESR investigations of high-T_c phase superconductor - as known to be the most suitable candidate for high temperatures/high magnetic field applications [11]. The freshly fine crushed bare bulk rod Bi-2223 powdered sample is taken to avoid contamination with environment. The detailed synthesis procedure for these bulk rod samples including the phase, microstructural, compositional and resistivity vs temperature characteristics were described previously [12].

The main broad asymmetric ESR signal along with some side signals at room temperature (300K) has been observed for evenly spread single layer of paste of fine powder. The entire signal along with the side signals disappear at 78 K. ESR measurements at different temperatures and angular variations were made to explore the nature of the main and side ESR signals. The present results are also compared with the earlier reports on different conventional [13]-[18] and unconventional superconducting systems [8]-[10], [19]-[24].

X-ray diffraction (XRD), Scanning Electron Microscope (SEM)/Energy Dispersive Analysis for X-rays (EDAX) and alternating current (ac) susceptibility studies were also performed on the same powdered sample in order to check any degradation occurring in phase purity, composition and T_c due to grinding of the sintered rod sample.

II. EXPERIMENTAL DETAILS

Bare bulk rod Bi-2223 rod sample used in our previous study [12] is taken. Before recording ESR spectra, the obtained powder was characterized by XRD, EDAX and ac susceptibility analytical techniques. XRD patterns were recorded using Bruker D-8 powder X-ray diffractometer with CuK$_\alpha$ radiation for phase analysis. EDAX spectra were obtained using Oxford ISIS 300 system with EDAX attached with SEM (LEO 440 SEM). For ac susceptibility measurements, a close cycle refrigerator (Sumitomo model: SRD 204) was used and the temperature was monitored by a gold/iron chrome thermocouple.

ESR studies were carried out from room temperature (300 K) to liquid nitrogen temperature (78 K) by Varian Make X-band CW EPR spectrometer. The freshly ground (Bi, Pb)-2223 bulk rod sample contamination free fine powder of average particle size of 0.54 μm was taken for recording ESR spectra at ambient and low temperatures. Measurements were also made at 300 K for different angular orientations. To observe CESR signal with minimum distortions and saturation effects, an evenly spread single layer of fine powdered bulk rod sample mixed well in vacuum grease (Apiezon N-type) on flat surface of a quartz rod

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was taken as suggested by Aoi and Swihart [25]. Modulation amplitude was kept low to avoid line shape distortion. 10 mW microwave power was used to reduce saturation effects and spectrometer was retuned several times near superconducting transition temperature. The signal was modulated at 100 kHz frequency and DPPH (1, 1diphenyl-2 picrylhydrazyl) was used as standard reference sample as field marker and g-value determination.

III. Results

In Fig. 1 (a), XRD pattern of the Bi–2223 powder shows all the diffraction peaks pertaining to high-T_c (Bi, Pb)–2223 phase except two weak peaks (marked with star) belonging to Ca_2PbO_4. No peaks due to other secondary phases like low-T_c Bi–2212 phase and/or any impurity phases including the possibility of most expected copper containing phases e.g. $Sr_{14}Cu_{24}O_{41-x}$, $Bi_{2-x}Sr_xCu_2O_{5-y}$, Ca_2CuO_3 etc. are observed. In Fig. 1 (b), EDAX studies further confirm the absence of any impurity/copper containing phases and reveals the constituting elements Bi, Pb, Sr, Ca, Cu and O composition in at % as 10.55, 1. 99, 11.00, 11.31, 16.47 and 48.68, respectively. The sample has stoichiometric ratio of Bi:Pb:Sr:Ca:Cu:O = 1.99:0.37:2.07:2.16:3.10:9.18 which is in good approximation to the chemical formula of superconducting phase. Hence, both XRD and EDAX results exhibit nearly single phase of Bi–2223 powdered sample. Fig. 1 (c) depicts ac susceptibility versus temperature plot of Bi–2223 powder. An almost narrow superconducting transition (T_c^{on} 114.2 K and T_c^{off} 105.1 K) suggests that the powder sample has good superconducting properties of the (Bi, Pb)-2223 phase and grinding process has not degraded superconductivity of the sample. These studies also support the inference drawn from XRD and EDAX data.

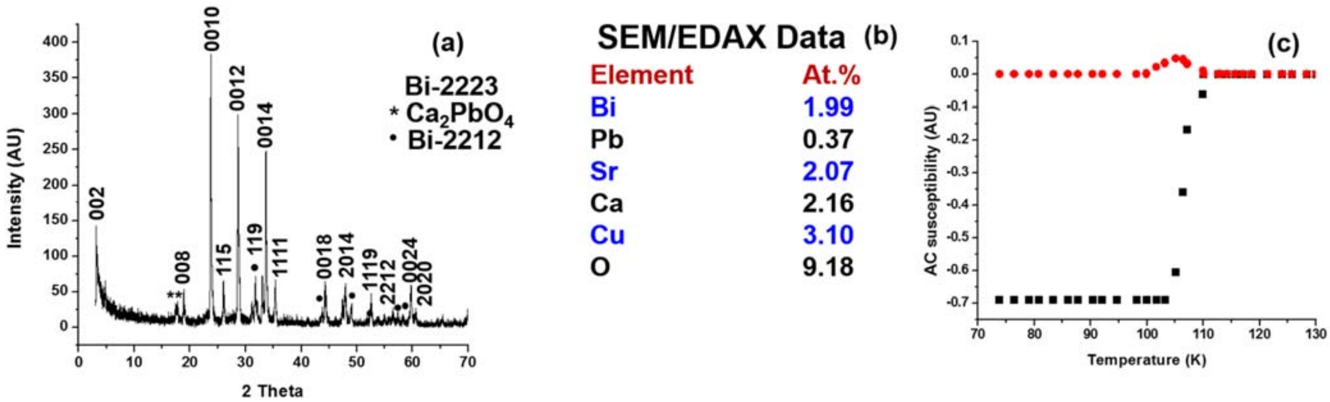

Fig. 1 (a) XRD pattern, (b) SEM/EDAX data and (c) temperature dependence of real (χ', black) and imaginary (χ'', red) parts of AC susceptibility of fine powdered Bi-2223 sample

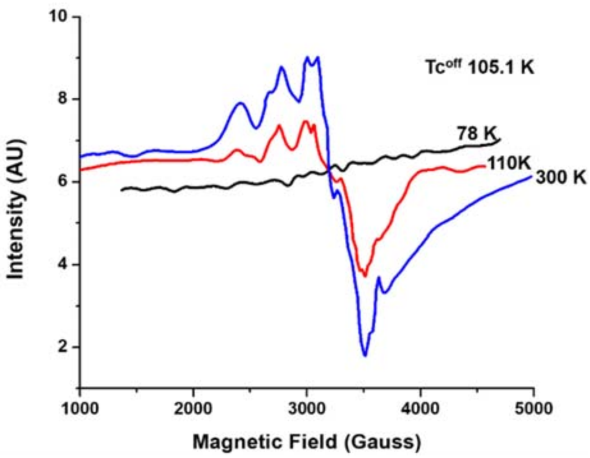

Fig. 2 CESR absorption spectra of single layer of Bi-2223 sample at 300 K, 110 K and 78 K

Fig. 2 shows ESR spectra of evenly spread powder single layer at 300 K, 110K and 78 K. A strong broad asymmetric main resonance signal with few side signals appears at 300 K (the normal state). The broad signal has g-value around 2.0248 peak–to-peak line width (ΔHpp) is 725 G at 300 K. The signal asymmetry is attributed to the characteristic of line shape. At 78 K temperature which is much below T_c^{off} (105.1 K), i.e. in the superconducting state, both the broad asymmetric and side signals disappear. The disappearance of entire signal was confirmed by repeating few runs. This vanishing of the main broad as well as side signals at T_c^{off} suggests their relation with the superconducting state.

Fig. 3 presents comparison of the behavior of asymmetric broad signal and side signals in some conventional superconductors: (a) Al [14], [15], (b) Nb_3Ge [15], and unconventional superconductors like: (c) MgB_2 [23], (d) $YBa_2Cu_3O_{7-\delta}$ [19] and (e) [8], [9] in their normal and superconducting states. It indicates that the behavior of present signals (Fig. 3) is similar to the signal shown by all these different superconductors. That is the CESR/spin waves signal are present in the normal state which in superconducting state either decrease drastically or not observable. However, the side signals are far away in the case of low-T_c ~ 1.2 K Al (Fig. 3 (a)) and 22.65 K Nb_3Ge sample (Fig. 3 (b)) whereas, these are close to the main ESR signal for the high-T_c 80 K $YBa_2Cu_3O_{7-\delta}$ (Fig 3 (d)) and for the present 105.1 K (Bi, Pb) – 2223 samples (Fig. 2). This observation reveals some correlation between T_c and the relative position of side signals with respect to the main signal.

Table I shows a comparison of the various ESR parameters as reported by the earlier and present researchers [8]-[10], [14], [15], [18]-[22], [24]-[27]. The behavior and parameters derived from present ESR signal and of the reported ones in different conventional as well as nonconventional superconductors are nearly identical.

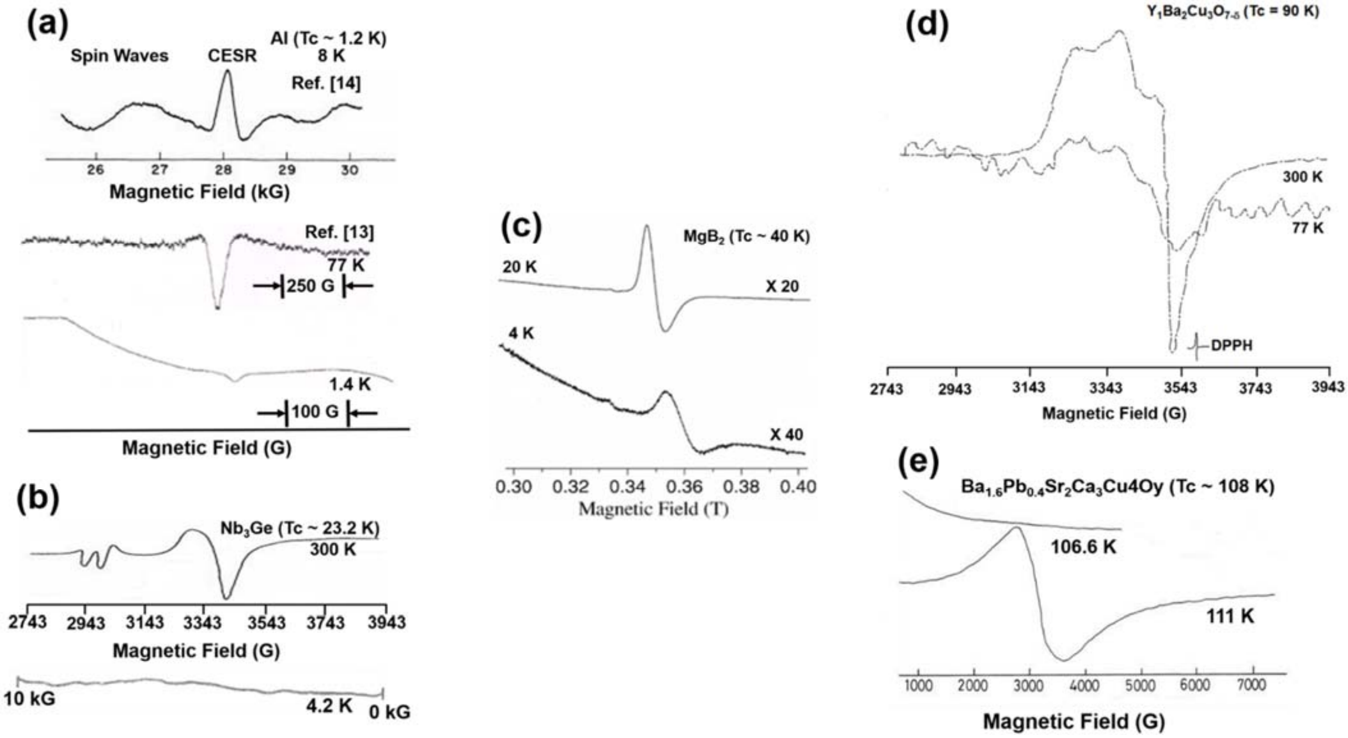

Fig. 3 Comparative CESR absorption signals reported in some conventional (a), (b) and unconventional (c), (d), (e) high-Tc superconductor

TABLE I
COMPARISON OF VARIOUS ESR PARAMETERS REPORTED BY EARLIER RESEARCHERS AND PRESENT WORK

S.No.	Sample	Normal State Signal CESR	Normal State Signal Spin Waves	Super-conducting State Signal CESR	Super-conducting Signal Spin Waves	CESR Signal g-value	CESR Signal ΔHpp (G)
1	Al	P	P	VW	A	1.997	130
2	Nb_3Ge	P	P	A	A	2.022	43.5
3	Mo_6Se_8	P	P	A	A	2.005	30
4	Nb	P	-	VW FNS	-	1.84	600
5	MgB_2	P	-	VW	-	NG	111
6	Rb_3C_{60}	P	-	A	-	NG	700
7	YBCO	P	-	A	-	2.00	200
8	YBCO	P	P	VW	A	NG	NG
9	Bi-2223	P	-	A	-	2.25	800
10	Bi-2223	P	-	A	-	2.24	520
11	**Bi-2223**	**P**	**P**	**A**	**A**	**2.025**	**725 PW**

P, VW, A, --, FNS, ΔH_{pp} and PW stand for Present, Very Weak, Absent, Not Given, Forced Normal State, Peak-to-peak linewidth and Present Work respectively.

Fig. 5 describes the angular orientation dependence [angle of orientation of external dc magnetic field (H_{ext}) with respect to sample surface] of the (i) main asymmetric ESR signal and (ii) side signals relative to the main signal at 300 K. The position and width of the main ESR line remains unaffected with orientation. However, the side signals slide around the asymmetric signal and also show merging behavior with orientation of the field. These results are in concurrence with the previous works [14]-[16], [25] in a variety of conventional superconductors, where these authors reported that if the position and width of the main signal remained unchanged with respect to orientation of H_{ext}, then this signal is due to conduction electrons. While the merging and sliding behavior of the side signals indicates that these signals are due to spin waves.

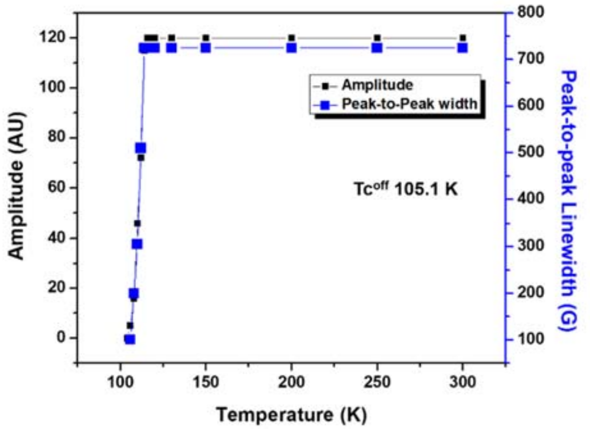

Fig. 4 CESR signal amplitude and peak-to-peak linewidth vs. temperature of single layer Bi-2223

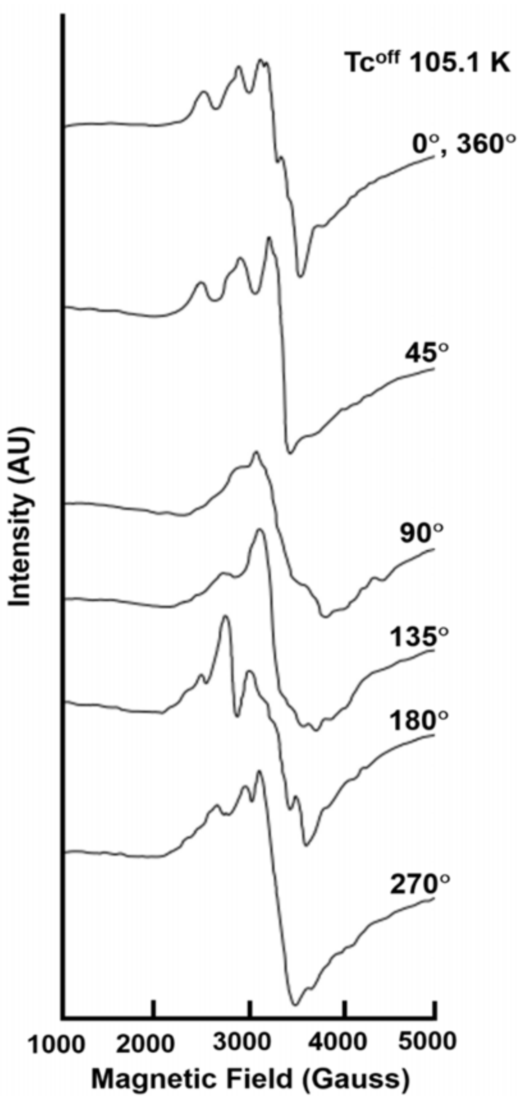

Fig. 5 CESR absorption signal at 300 K of Bi-2223 single layer for different angular orientations

IV. Discussion

In the light of above results an important aspect emerges that is, the main broad asymmetric ESR absorption signal in the present (Bi, Pb)-2223 sample seems to be of some other origin than as generally reported due to Cu^{2+} ions because of the following reasons. (i) From the XRD pattern and from SEM/EDAX data (Fig. 1), no traces of copper containing impurity phases which are the source of Cu^{2+} signal were found, (ii) moreover, the characteristic features of such signals (whether in Cu containing impure phases and/or due to Cu^{2+} ions in oxygen deficient/ imperfect CuO planes) as reported earlier [1]-[7] are more symmetric, narrower and weaker as compared to the observed signal (Fig. 2), (iii) these signals show Curie like behavior in the normal state, unlike the observed Pauli type (Fig. 4), (iv) in special cases, like "Anderson lattice" [26], they have Pauli behavior in the normal state and more importantly significant rise in the superconducting state, unlike the observed disappearance of the signal (Fig. 4). In addition, the present results also bring out another important aspect regarding the side signals.

In fact, it is mentioned in the results section that like the main signal, behavior and the parameters of the side signals in the normal state and in the superconducting state obtained in the present investigations (Fig. 3, Table I) are similar to the signals as reported earlier in different low-T_c conventional [13]-[17] (Figs. 3 (a), (b)) and in high-T_c unconventional [8], [18]-[21] superconductors (Figs. 3 (c)-(e)). For example, both the broad asymmetric signal and side signals appear in the normal state which either disappear or reduce markedly in the superconducting state. This strongly suggests that both of these signals are a consequence of presence of unpaired conduction electrons as reported by the above authors. This is further supported by the observation of Pauli like temperature independence (Fig. 4) and an insignificant orientation dependence (Fig. 5), which are characteristic features of unpaired conduction electrons as pointed out by Platzman-Wolff [26]. Also, the behavior/the parameters listed in Table I of this broad resonance signal are comparable with the earlier reports [8]-[10], [14], [15], [17]-[20], [22], [23] in different superconducting systems, therefore, this signal has also been attributed to CESR in the present (Bi, Pb)-2223 samples.

Now, in context to origin of side signals which shows merging behavior on orientation with respect to magnetic field unlike the main signal. Similar signals as reported earlier have been identified as the Platzman-Wolff (P-W) type spin waves [26] in conventional type-I superconductor (Al) by Dunifer et al. [14] and in a variety of both type I and type II superconductors by Ekbote et al. [16]. Platzman-Wolff [26] discussed the appearance of spin waves in terms of correlations and exchange interactions among conduction electrons in alkali metals. Based upon these similarities in the present signal and the reported ones, we have also assigned them as Platzman-Wolff Spin Waves (PWSW) originating from exchange interactions among conduction electrons. The decrease in ΔHpp on lowering temperature below T_c^{on} (Fig. 4) also support the presence of exchange interaction [10], [13], [29].

Further, noticeable closeness of spin waves to CESR signal in the present (Bi, Pb)-2223 sample (Fig. 3) indicates that intensity of the exchange interactions is high and are strong also. This contention is supported by the exhaustive study of Ekbote et al. [16], where the authors showed that closer the spin waves to CESR, stronger are the exchange interactions and higher is the T_c of the superconductor. The clear smaller separation between spin waves and CESR as reported in other high-T_c 80 K (Fig. 3 (c)) YBCO [20] in comparison to larger separation in the case of low-T_c: (Fig. 3 (a)) 1.2 K Al [14] and (Fig. 3 (b)) 22.65 K Nb_3Ge [15] support the presence of enhanced exchange interactions in the present samples having T_c^{off} of 105.1K which cannot be ignored in the ground state formation.

Returning to the main signal with regard to changes occurring in the signal parameters (Fig. 4): signal intensity [I = signal amplitude x $(\Delta Hpp)^2$] and peak-to-peak line width (ΔHpp) on decreasing temperature below 114.2 K (T_c^{on}). Nearly insignificant temperature change in signal intensity (Pauli behavior) in the temperature range: 300-114 K is due to no change in the number of unpaired conduction electrons because the sample is a normal metal at the temperature above Tc. However, on lowering temperature below 114.2 K (T_c^{on}), the sharp decrease in signal amplitude up to 107 K and disappearance of the signal at 105.1K reflects the well-known decrease in the number of unpaired conduction electrons as a result of their pairing in the near transition region from normal to superconducting state. These near T_c observations of decrease in the signal amplitude as well as in ΔHpp with simultaneous disappearance of CESR and side spin wave signals are indicative of appearance of some internal field (H_{int}) of – J type (antiferromagnetic) [12], [15], [16], [27], [29], [30] and resisting the external field.

Finally, all the above results and discussion provide the evidences of a dominate role of exchange interactions in the present high-T_c (Bi, Pb)-2223 samples. The disappearance of both the signals (CESR and the spin wave signal) in superconducting state has been explained on the basis of the role of antiferromagnetic exchange interactions (-J) among conduction electrons in pair formation as explained by Ekbote's extended study [28] in high -T_c cuprates.

V. Conclusion

The pivotal role of the paper is the unusual behavior of CESR signal in normal and below T_c, which are quite different from non-superconducting materials and suggest strongly a nature depending on exchange effects in the highly correlated system of conduction electrons of Bi-2223 system. Mainly they fall in three categories: (i) The appearance of strong broad asymmetric main ESR signal along with side signals in nearly single phase (Bi, Pb)-2223 samples in normal state, (ii) Nearly insignificant temperature dependent changes in the amplitude, g-value/peak-to-peak line width and orientation independent shifting of the main signal in the normal state suggest that this signal is due to highly correlated conduction electrons (CESR), and (iii) The orientation dependent merging behavior of the side signals around the main CESR signal suggests that these are due to P-W type spin waves caused by the exchange interactions resulting from high correlations among conduction electrons. The closeness of spin wave signal to CESR signal, line-width narrowing near T_c strengthens the authors' suggestion (also indicated in [29] and [30]) that the Bi-2223 system in its normal state has a highly correlated conducting particle system and has the dominance of exchange interactions among them as CESR is the sensitive tool to decipher the interactions and behavior of the metal conduction electrons too. Below superconducting transition, disappearance of CESR along with spin wave signal as observed in conventional superconductors also in the superconducting state of Bi-2223 suggests that the pair formation is the manifestation of antiferromagnetic exchange interactions among the conduction electrons.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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