Magnetic Dynamics and Spin Freezing in the Ferromagnetic Phase of Ba-Doped Perovskite Cobaltites

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The magnetic properties of polycrystalline La0.5Ba0.5CoO2.5+δ (δ = 0.44 and 0.485) were investigated using muon spin relaxation and bulk magnetization experiments. Below a Curie temperature of Tc = 160 K and 180 K, respectively, a magnetic phase with a macroscopic ferromagnetic moment forms. µSR proves that the full volume inhomogeneously orders at Tc but that there is microscopically a phase separation into phase volumes possessing a fully static and a slow magnetic dynamic behavior. A peak in the dynamic relaxation rate 35 K respectively 55 K below the Curie temperature indicates the freezing of spin components. The spin fluctuations are thermally activated with a typical Jahn-Teller like phonon energy of £a = 340 K. Together with co-operative Jahn-Teller distortions appearing below the spin freezing temperature, this points to a strong magneto elastic coupling as the cause for the magnetic fluctuations and the spin freezing.

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I. INTRODUCTION

Materials with competing magnetic exchange interactions may adopt a randomly frozen arrangement of spins at low temperatures known as spin glass [1, 2]. Even starting from a long range magnetically ordered state, competing ferromagnetic (FM) and antiferromagnetic (AFM) interactions can lead to a loss of collinearity and the formation of a spin glass [3]. Also the phase separated state of a partially randomly frozen spin glass and the collinear magnet can be achieved. The nature of the spin glass formation wether it is achieved through an equilibrium phase transition or a more gradual freezing process has been strongly debated. For the study of spin dynamics, the muon spin rotation (µ+ SR) technique has been recognized to be a powerful probe among other complementary experimental methods. The possibility of zero-field measurements makes the technique well suitable for the spin glass systems for which a very small applied external field can change the internal configuration of the system.

Cobaltites of the chemical composition RBaCo2O5+δ have recently attracted great interest due to their intriguing electrical and magnetic properties [4-17]. On top of orbital/charge ordering, metal-insulator transition and giant magnetoresistance effects an additional degree of freedom namely the different possible spin states of Co make these materials a fascinating area for fundamental science research. The possibility to adopt different spin states in the cobaltites is due to the competition of the crystal field splitting (10 Dq) of the 3d states favoring lower spin state configurations and the Hund’s exchange interaction (JH) that lowers the energy of each pair of electrons with parallel spin. For example in LaCoO3 the ground state is a nonmagnetic Co3+(S = 0, t2g eg) due to the fact that 10 Dq is slightly larger than JH. A spin state transition to a higher spin state takes place with increasing temperature. The mechanism of the transition is still under debate and different scenarios were proposed. Some authors concluded a change from low spin to intermediate spin (S = 1, t2g eg) while others suggest the formation of the high spin state configuration (S = 2, t2g eg) 18-21. The introduction of additional charges by e.g. the substitution of La3+ by M2+ (M = Ba, Sr, Ca) in LaCoO3 results in remarkable changes in their magnetic and electrical properties.

In this study, we have investigated powder samples of La0.5Ba0.5CoO2.94 and La0.5Ba0.5CoO2.985. Compared to the charge-balanced R BaCo2O5.5 compound, these samples are hole-doped and possess a random distribution of La and Ba cations on the R site. The increased oxygen content introduces a corresponding amount of Co4+ into the undoped system consisting of Co3+ only. For the fully oxidized compound, La0.5Ba0.5CoO3 a 1:1 mixture of Co3+ and Co4+ is obtained. The physical properties are strongly influenced by this doping due to competing magnetic interactions. A predominantly FM order due to the double exchange interactions between Co3+-Co4+ compete with the AFM exchange mediated by the superexchange mechanism. This competition may lead to the coexistence of FM and glassy behavior observed e.g. in La0.5Sr0.5CoO4 [22]. The A-site ordered compound LaBaCo2O6 possesses a ferromagnetic transition at TC = 175 K while in its isochemical counterpart La0.5Ba0.5CoO3 the TC = 190 K. Below around

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140 K both La_{0.5}Ba_{0.5}CoO_3 and LaBaCo_2O_6 transform to a magnetic glassy state [23]. At the same temperature Fauth et al. [24] observed a structural change from cubic to tetragonal which has been interpreted as a cooperative Jahn-Teller effect and a partial d_{x^2−y^2} type of orbital ordering. This interpretation is supported by the observed elongation of the CoO_6 octahedra along the direction of the apical oxygen. At the same temperature a metal to insulator transition is observed. Since mobile charges are a prerequisite for the FM double exchange mechanism, a change in the balance is observed. Therefore AFM-FM can be anticipated at this temperature.

In this paper we present a susceptibility and μ+SR study of La_{0.5}Ba_{0.5}CoO_{2.5+δ} for two samples with different oxygen content, δ = 0.44 and 0.485. Macroscopic dc and ac-susceptibility measurements reveal a FM transition at T_C = 160 K and 180 K respectively. A clear frequency dependence of the ac susceptibility is observed below T_C, i.e., a magnetic behavior typically found in systems exhibiting a spin freezing. μSR reveals quasi-static disordered magnetic ordering below T_C with slow magnetic dynamics persisting in 70% of the sample volume down to lower temperatures. Far below T_C (35 K and 55 K) a peak in the dynamic relaxation indicates the freezing of the fluctuating spin components. The measured magnetic fluctuation rates can be well described by a thermally activated process with an activation energy typical for Jahn-Teller like phonons. This and the previously observed Jahn-Teller distortions in the crystal lattice at the same temperature indicate that the freezing process is governed by a magneto elastic coupling.

II. EXPERIMENTAL

La_{0.5}Ba_{0.5}CoO_{2.5+δ} (δ = 0.44 and 0.485) samples were synthesized by a standard solid state reaction. The rare earth oxide, cobalt oxide and barium carbonate of a minimum purity of 99.99% were mixed and annealed at temperatures 900 - 1200 °C for 100 h in air, with several intermediate grindings. The obtained material was separated into two batches. One of the batches was additionally annealed in oxygen flow for 20 hours. The oxygen content was determined with an accuracy of ±0.01 of the oxygen index in the chemical formula by iodometric titration. Since the synthesis requires elevated temperatures and high oxygen pressures to obtain a layered perovskite structure [23], a slight oxygen-deficiency remains without a high-pressure oxygen annealing. We found for the as-prepared sample an oxygen content of δ = 0.44 whereas for the oxidized sample δ = 0.485. The details of the sample preparation and determination of the oxygen content can be found in Ref. [25]. From the analysis of our X-ray data using the Rietveld profile refinements a cubic structure with a space group Pm̅3m was found at room temperature for both sample batches. The cubic symmetry implies a complete disordering of the lanthanum and barium ions on the same site of the perovskite. The bulk magnetic properties have been studied using a Physical Properties Measurements System (PPMS) in a temperature range T = 10 - 250 K. The μSR technique utilizes positively charged μ^+ which are implanted into the sample and thermalize at interstitial lattice sites, where they act as a volume sensitive magnetic microprobes. The amplitude of the different δμSR signals is proportional to the volume fraction of the corresponding magnetic phase.

III. RESULTS AND DISCUSSION

Fig. 1(a) shows dc-magnetization measurements performed with an applied external field of 0.1 T after zero
field cooling (ZFC) and field cooling (FC) in the temperature range 10-250 K for La$_{0.5}$Ba$_{0.5}$CoO$_{2.985}$. For tempera-

tures higher than 180 K the sample is paramagnetic. In fact at $T_C = 180$ K a ferromagnetic transition is observed with a sudden increase of the magnetization. Lowering the temperature the magnetization exhibits an irreversible magnetic behavior between ZFC and FC magnetization curves below 140 K. The saturation moment is close to 2 $\mu_B$/F.U. indicating a mixture of spin states which are most probably IS/LS of both Co$^{3+}$ and Co$^{4+}$ ions [23, 24]. The ac-susceptibility measurements as a function of temperature are shown in Fig. 1(b) and 1(c) for the in-phase $\chi'$ and out of phase $\chi''$ component of the susceptibility, respectively. The magnetic susceptibility shows a sharp increase close to the FM transition. At lower temperature, slightly below $T_C$ the susceptibility measurements show a peak followed by slow decrease with decreasing temperature. The observed peak as well as the low temperature tail of $\chi'$ shift to higher temperatures with increasing measuring frequency. The shift to the higher temperatures with increasing the frequency is better observed for $\chi''$ in Fig. 1(c). The magnetic behavior of La$_{0.5}$Ba$_{0.5}$CoO$_{2.985}$, i.e. the dc-magnetization below 140 K, the frequency dependent cusp in the ac-susceptibility and the small hysteresis, are consistent with a spin glass and cluster glass like freezing of the magnetic moments [27, 28]. The actual temperature where ZFC and FC magnetization deviate from each other depends on the externally applied field also in accordance with spin glass like systems. Interestingly our susceptibility data are in good agreement with previous measurements on the A-site ordered compound LaBaCo$_2$O$_6$ [29] which indicates that this structural feature is not of strong importance for the magnetic behavior studied here.

In order to get a better understanding of the magnetic order and to probe the spin dynamics in La$_{0.5}$Ba$_{0.5}$CoO$_{2.985}$, ZF-$\mu$SR was used. The technique can give a direct measure of the internal fields in bulk samples. The sensitivity of the technique to fluctuations of the internal field gives important information about the dynamics of the magnetic moments in the sample. The ZF-$\mu$SR spectra were collected over the temperature range 5 - 250 K. The ZF-$\mu$SR spectra are weakly damped with an increasing relaxation when $T_C$ is approached from above, see inset of Fig. 2(a).

To analyze our experimental data, a stretched exponential form was used at all temperatures above $T_C$:

$$P(t) = e^{-(\lambda t)^\beta}.$$  \hspace{1cm} (3)

Here, $\lambda$ represents the relaxation rate and $\beta$ the exponent. The spin relaxation in all this temperature range is attributed static nuclear moments and rapidly fluctuating cobalt moments in the paramagnetic phase. This stretched exponential function, Eq. 3, has been found to be appropriate in cases where a complex non-exponential fluctuation pattern is expected, e.g. due to broad distribution of fluctuation times [30]. The temperature dependence of $\lambda(T)$ is shown in Fig. 2(a). As the temperature approaches the magnetic phase transition from above the magnetic fluctuations slow down and lead to the sharp increase of $\lambda(T)$ at $T_C = 160$ K for La$_{0.5}$Ba$_{0.5}$CoO$_{2.94}$ and 180 K for La$_{0.5}$Ba$_{0.5}$CoO$_{2.985}$, respectively. The temperature dependence of the stretched exponential exponent $\beta$ above $T_C$ has been depicted in Fig. 2(b) for both samples with $\beta = 2.94$ and $\delta = 2.985$, respectively. In magnetically homogenous systems $\beta$ is equal to unity whereas in inhomogenous systems with spin frustration $\beta$ becomes smaller than unity [31]. The stretched exponential exponent $\beta$ reaches a value of about 1$\frac{1}{2}$ at $T_C$ for both samples which is typical for inhomogeneous systems like spin or cluster glasses.

Below $T_C$ the ZF-$\mu$SR spectra change completely. In Fig. 3(a) a spectrum at 170 K (below $T_C = 180$ K) is compared to a spectrum at 190 K for La$_{0.5}$Ba$_{0.5}$CoO$_{2.985}$. A strong depolarization of 2/3 of $P(t)$ is observed at early times, indicating a wide distribution of internal magnetic fields at the muon site, consistent with a magnetically

![Graph](image-url)
in a partially static system. Note that the early time relaxation is dominated by the presence of the static component of the field distribution and that the depolarization of 2/3 of the µSR signal implies that 100% of the sample is magnetic below $T_C$. If the field distribution at the muon site would be purely static within the time window of µSR the fast relaxation of 2/3 of the signal at early times would be followed at late times by a non-relaxing tail with $P(t) = 1/3$. This tail reflects, in a powder average, the static field components which are longitudinal to the initial muon spin which do not depolarize $P(t)$. This behavior is not observed in the present samples of La$_{0.5}$Ba$_{0.5}$CoO$_{2.985}$ and La$_{0.5}$Ba$_{0.5}$CoO$_{2.94}$. The late time spectra of La$_{0.5}$Ba$_{0.5}$CoO$_{2.985}$ for different temperatures below $T_C$ are shown in Fig. 3(b). Clearly, a relaxation of the 1/3-tail of the spectrum is observed for temperatures well below $T_C$. This relaxation is purely due to fluctuating electronic moments, i.e. it is of dynamic origin, see e.g. [32, 33]. The strongest dynamic relaxation is observed at $T = 125$ K, i.e. 55 K below $T_C$. Since $P(t)$ approaches 0.1 at late times it is evident that only 70% of the muons experience this dynamic relaxation. In other words, a phase separation into a purely static magnetic phase (30%) and into a fraction of the sample experiencing slow magnetic fluctuations (70%) is observed below $T_C$. Below 125 K the dynamical relaxation decreases and becomes insignificant at very low temperatures where the sample becomes fully static at a temperature of 5 K.

Based on the qualitative results discussed above a fit function given by the sum of a static and a dynamic depolarization function was used for the spectra below $T_C$:

$$P(t) = 0.3P_{\text{static}} + 0.7P_{\text{dynamic}}$$

The two functions used are:

$$P_{\text{static}} = \frac{2}{3} \cos(2\pi ft)e^{-\frac{1}{3}(\Delta t)^2} + \frac{1}{3}$$

$$P_{\text{dynamic}} = \left[\frac{2}{3} \cos(2\pi ft)e^{-\frac{1}{3}(\Delta t)^2} + \frac{1}{3}\right]e^{-\lambda t}$$

Here, $\Delta$ measures the width of the static field distribution, while the muon precession frequency $f$ is the mean value of this distribution. $\lambda$ is the dynamic relaxation rate. The µSR signal $P_{\text{dynamic}}$ which is connected to the magnetic phase in which static order and magnetic fluctuations are simultaneously present is described by a product of a static and a dynamic relaxation function. This estimation is valid in the present case, where $f \gg \lambda$ [34]. Static Lorentz and Gaussian Kubo-Toyabe functions have been tested for the static relaxation function also, but only unsatisfactory results have been obtained.
is given by the fluctuation rate $\nu_c = 1/\tau$, the muon spin Larmor precession frequency $\omega_\mu = 2\pi f$, and $\sigma$ describing the static field distribution. This formula is valid for a isotropic field distribution in the fast fluctuation regime ($\nu_c >> \sigma$). Following the argumentation of Baabe et al. [36] the Redfield theory modifies to

$$\lambda = 1.5\sigma_{VV}^2 \frac{\nu_c}{\nu_c^2 + \omega_\mu^2}$$

when electronic spins are the reason for the field fluctuations at the muon site, which themselves precess fast in a static magnetic field given by a magnetic environment. $\sigma_{VV}$ is the static Van Vleck linewidth. The solid lines in Fig. 4(c) are fits of the dynamic relaxation rate using the modified Redfield formula Eq. 8. To evaluate the function $\lambda(T)$ the temperature dependences of $\omega_\mu(T)$ and $\sigma_{VV}(T)$ have to be known. From a fit of the measured $\mu$SR frequencies $f(T)$ to a simple mean field approach, $\omega_\mu(T)$ is obtained (see solid lines in Fig. 4(a) and (b)):

$$\omega_\mu(T) = 2\pi f_0\left(1 - T/T_c\right)^{0.5}$$

The relation between $\sigma_{VV}$ and $\omega_\mu$ has been determined by evaluating the measured dynamic relaxation rate $\lambda$ at its maximum value, where, according to Eq. 8, $\nu_c = \omega_\mu$ and the following relation holds:

$$\lambda_{\text{max}} = \frac{3}{4}\frac{\sigma_{VV}^2}{\omega_\mu}$$

In other words, $\omega_\mu(T)$ and the proportionality constant between $\sigma_{VV}$ and $\omega_\mu$ have been determined experimentally. Strictly speaking the proportionality constant $4/3\lambda_{\text{max}}$ has been measured at the maximum of $\lambda(T)$ only. The analysis of $\lambda(T)$ using the Redfield formalism has been performed assuming that this proportionality holds for all temperatures, i.e.:

$$\lambda_{\text{max}} = \frac{3}{4}\frac{\sigma_{VV}^2}{\omega_\mu(T)}$$

Having the other parameters determined independently, the only free parameter in the modified Redfield formula Eq. 8 is the magnetic fluctuation rate $\nu_c$. The data shown in Fig. 4(c) are well described by an activated behavior of the fluctuation rate

$$\nu_c = \nu_0 \exp\left(-E_a/T\right)$$

with an attempt frequency $\nu_0$ and an activation energy $E_a$, the obtained fit results are summarized in Tab. 1. The fit to the data is excellent over the full temperature range even though the described formalism is strictly speaking only valid at high temperatures and slightly below the maximum of $\lambda(T)$. At low temperatures the fluctuations are so slow, that the Redfield formula can not be applied anymore. For both compounds, an activation energy of $E_a \approx 340$ K is observed, which is a typical activation energy for a Jahn-Teller like phonon. The present quantitative data are in good agreement with the neutron data.

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**FIG. 4.** (a) and (b) show the temperature dependence of the static relaxation rate $\lambda(T)$ for La$_{0.5}$Ba$_{0.5}$CoO$_{2.94}$ and La$_{0.5}$Ba$_{0.5}$CoO$_{2.985}$ together with the $\mu$SR frequency $f$ evolution. The solid lines in (a) and (b) are fits according to Eq. 8. (c) Temperature dependence of the dynamical relaxation rate for the temperatures below $T_c$. The lines represent the fit results according to the Redfield theory.
published by Fauth et al. [24] where a cooperative Jahn-Teller deformation develops lowering the temperature below 140 K where the metal-insulator transition takes place. Therefore we propose that the freezing of spin components at $T_f$ has its origin in the Jahn-Teller distortion and/or orbital ordering. In other words, we suggest that the observed magnetic fluctuations stem from local charge and corresponding spin state fluctuations between the Co$^{3+}$ and Co$^{4+}$ ions which are driven by Jahn-Teller fluctuations. Due to the La/Ba-site random disorder, the CoO$_6$ octahedra will be distorted locally which additionally favors the localization of the conduction electrons and an insulator state at lower temperatures. The charge carrier localization accompanying the Jahn-Teller distortions probably shifts the balance of the competing double and super exchange towards the AFM superexchange and in turn the randomly distributed ferromagnetic - antiferromagnetic interactions lead to a magnetic glassy state observed below $T_f$.

IV. CONCLUSION

In conclusion, susceptibility and $\mu$SR measurements on two La$_{0.5}$Ba$_{3.5}$CoO$_{2.5+\delta}$ samples with different oxygen content, $\delta = 0.44$ and 0.485 have been performed in order to study its static and dynamic magnetic properties. Both compounds are paramagnetic at high temperatures and a transition to an inhomogeneous ferromagnetic phase is observed at $T_C = 160$ K and 180 K, respectively. The samples are electronically phase separated into phases possessing static magnetic order (30%) and a major sample volume showing slow magnetic dynamics of electronic moments. A maxima in the dynamic magnetic relaxation rate at $T_f = 125$ K signals a freezing of spin components well below $T_C$. The simultaneous occurrence of the spin freezing and static cooperative Jahn-Teller distortions below $T_f$ and the observation that the magnetic fluctuations are thermally activated with a typically activation energy of Jahn-Teller like phonons suggest that the magnetoelastic coupling is important to the understanding of the low-temperature spin dynamics of La$_{0.5}$Ba$_{3.5}$CoO$_{2.5+\delta}$ and that it is responsible for the observed spin freezing. We propose that the magnetic fluctuations originate from local charge and hence spin state fluctuations of neighboring Co$^{3+}$ and Co$^{4+}$ ions driven by dynamic Jahn-Teller distortions. This interpretation is also consistent with the observed absence of a change of the macroscopic magnetization at $T_f$. Below $T_f$ a magnetically glassy state, i.e. a re-entrant spin glass phase is entered as evidenced by the difference of FC and ZFC magnetization curves and a typical frequency dependence of the ac-susceptibility.

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| $\nu_0$ (MHz) | La$_{0.5}$Ba$_{3.5}$CoO$_{2.94}$ | $E_a$ (K) | La$_{0.5}$Ba$_{3.5}$CoO$_{2.985}$ |
|--------------|-------------------------------|---------|-------------------------------|
| 1952(234)    | 336(14)               | 2241(526)| 343(28)                       |

TABLE I. Attempt frequency $\nu_0$ and activation energy $E_a$ obtained from the fit shown in Fig 4 c) using the modified Redfield theory explained in the text.

This formula is obtained from the fit shown in Fig 4 c) using the modified Redfield theory explained in the text.
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