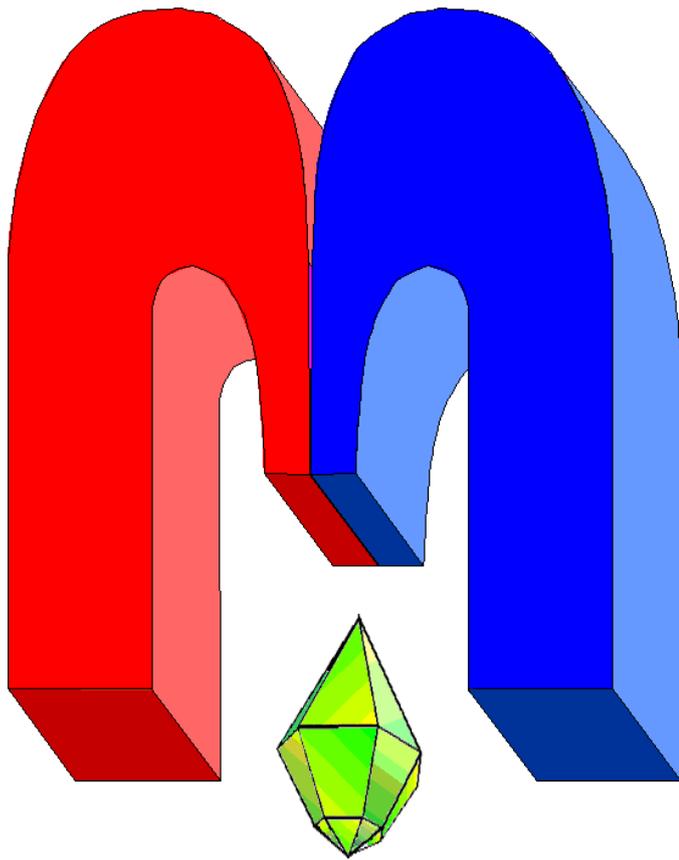


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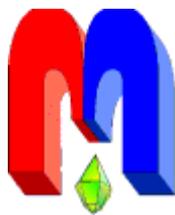
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Magnetic resonance analysis of spin dynamics in $\text{Sr}_2\text{MnTiO}_{5.87}$ double perovskite

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The spin dynamics of the $\text{Sr}_2\text{MnTiO}_{5.87}$ double perovskite have been studied via the magnetic resonance method. Two Lorenz lines below 100 K were used to describe magnetic resonance spectra. Using the Kawasaki-Mori-Huber and Vogel-Fulcher equations, the temperature dependence of the ESR line widths near the magnetic phase transition temperature have been described. The analysis of the line width in high-temperature limit allowed us to estimate the parameters of single-ion anisotropy and antisymmetric exchange interactions.

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Keywords: double perovskite, magnetic resonance, crystal field, Dzyaloshinskii–Moriya interaction, line width.

1. Introduction

SrTiO_3 -based (Sr_2MTiO_6) double perovskites are environmentally friendly high-temperature thermoelectric materials. Their synthesis condition (either solid-phase or precursor combustion method) determines the oxygen ion content. A non-stoichiometric oxygen content below 6 leads to a change in the valence of the $3d$ ions and drives complex magnetic interactions in the compound. Previously, we studied the magnetic properties of $\text{Sr}_2\text{MnTiO}_{5.87}$ double perovskite, where the oxygen concentration was determined using thermogravimetric analysis [1]. If the oxygen content is equal to 6, then the valence of all manganese and titanium ions should be $4+$, as soon as the oxygen ion content decreases, the valence of part of the $3d$ elements decreases to $3+$.

Two temperature $T_1 = 37 \pm 1$ K and $T_2 = 43 \pm 1$ K were occurred at transition from a paramagnetic to an ordered state. In addition, from the temperature dependence of the inverse magnetic susceptibility in the $T > 150$ K range, the Curie-Weiss temperature was determined $\Theta_{\text{CW}} = -400 \pm 18$ K, whose negative sign indicates antiferromagnetic-type isotropic exchange interactions between the spins of manganese ions in the $\text{Sr}_2\text{MnTiO}_{5.87}$ compound. The frustration parameter equal to $f = |\Theta_{\text{CW}}|/T_{\text{N}} = 10.8$ suggests a complex scenario of exchange interactions between Mn^{4+} ($S = 3/2$), Mn^{3+} ($S = 2$), and Ti^{3+} ($S = 1/2$) spins. We expect strong magnetic fluctuations in the sample, which affect the static and dynamic properties [2]. The magnetic resonance method is an effective tool for studying spin-fluctuation dynamics. One of the possible ways to quantify the effect of spin fluctuations on the magnetic resonance description in various compounds of spin-correlated systems is based on the Landau-Lifshitz equation and was suggested in [3]. Thus, an analysis of the temperature dependencies of the electron spin resonance (ESR) lines in Ti-, Fe-, and Co-doped CuGeO_3 single crystals showed that magnetic impurities lead to anomalous low-temperature line broadening jointly with a temperature-dependent g -factor shift $\Delta g(T)$. It was shown that at low temperatures the ratio of the line width to the g -factor shift $w(T)/\Delta g(T)$ scales linearly with temperature T for quasi-one-dimensional

antiferromagnetic chains [2].

On the other hand, due to strong exchange interactions, the magnetic resonance spectrum of strongly correlated compounds features one exchange-narrowed line, whose width is determined by anisotropic symmetric and antisymmetric interaction and additional single-ion anisotropy for ions with $S > 1/2$ [4]. The joint analysis of the magnetization and ESR data allows us to estimate anisotropic symmetric and antisymmetric interactions in strongly correlated systems [5].

The double perovskite Sr₂MnTiO₆ is composed of compounds SrTiO₃ and SrMnO₃, one of which is ferroelectric, and the second demonstrates antiferromagnetic ordering. As shown in [6] skyrmions can be formed in the interface between these compounds. Numerical calculations show that skyrmions can be formed at certain ratios between the magnitude of the Dzyaloshinsky-Moriya interaction, magnetoelectric interactions, and polarization.

Our research aims at using the magnetic resonance method to study spin correlations in new Sr₂M(1)M(2)O₆ double perovskites, where Mn is the cation M(1) and position M(2) is occupied by Ti ions, and to obtain the parameters of anisotropic exchange interactions from the angular dependence of the ESR line width. This is necessary to assess the possibility of the formation of skyrmions in magnetic compounds.

2. Experimental details and results

Polycrystalline powders of Sr₂MnTiO₆ perovskite were synthesized via the solution combustion precursor method using Sr(NO₃)₂, MnO₂, i-Ti(OC₃H₇)₄, and disubstituted ammonium citrate as a complexing agent. X-ray phase analysis (XFA) using the crystallographic database “Powder Standards Database – PDF2” (ICDD, USA, Release 2009), showed that the sample is single-phase and does not contain impurities. The diffractogram of Sr₂MnTiO₆ can be described on the basis of the Pm3m (No 221) spatial group with the cubic cell parameter $a = 3.85542(2)$ Å. According to thermogravimetric analysis, the oxygen content in the sample corresponds to Sr₂MnTiO_{5.87}. ESR studies of Sr₂MnTiO_{5.87} double perovskite in powder form were carried out at an ER 200 SRC Bruker spectrometer (EMX/plus) in the temperature range from 50-340 K at a frequency of 9.4 GHz.

The spectra of the Sr₂MnTiO_{5.87} powder are shown in Figure 1 and Figure 2. These ESR spectra can be satisfactorily described by the Lorentzian profile determined by the resonance-field value B_{res} , the line width ΔB , and the asymmetry parameter α [7], where the resonance at inverted field direction is included because of the large line width:

$$\frac{dP}{dB} \propto \frac{d}{dB} \left[\frac{\Delta B + \alpha(B - B_{\text{res}})}{(B - B_{\text{res}})^2 + \Delta B^2} + \frac{\Delta B - \alpha(B - B_{\text{res}})}{(B + B_{\text{res}})^2 + \Delta B^2} \right]. \quad (1)$$

One Lorentz line was used for description ESR spectra between 100 K and 340 K. To describe the shape of the ESR spectra below 100 K, it was necessary to use two ESR lines. An example of the decomposition of the spectrum into two lines is presented in the Figure 3e at 50 K. The approximation of magnetic resonance lines using equation (1) gives the temperature dependencies of the ESR linewidth, the asymmetry parameter α which is near zero, the resonance field B_{res} , and the integral signal intensity. The results are presented in Figure 3. The inverse temperature dependence of the integral intensity of the ESR line is presented in Figure 3d. A solid line is an approximation according to the Curie-Weiss law $1/\text{Intensity} \approx \text{const}(T + \Theta_{\text{CW}})$.

3. Discussion

Let us first consider the features of the behavior of the ESR spectrum in the high-temperature region. A single exchange-narrowed line was observed in the ESR spectrum of the $\text{Sr}_2\text{MnTiO}_{5.87}$ ceramics between 100 K and 340 K. In the case of strong exchange narrowing, the line width of the magnetic resonance is defined by the ratio of the second moment of the spectrum to the exchange frequency [4]:

$$\Delta B \approx \frac{\hbar M_2}{g \mu_B \omega_{\text{ex}}}. \quad (2)$$

The second moment M_2 can be calculated as:

$$M_2 = \frac{\langle [H_{\text{eff}}, S^+] [S^-, H_{\text{eff}}] \rangle}{\hbar^2 \langle S^- S^+ \rangle}. \quad (3)$$

The spin Hamiltonian of $\text{Sr}_2\text{MnTiO}_{5.87}$ can be defined as:

$$H_{\text{eff}} = J \mathbf{S}_i \mathbf{S}_j + \sum_{\alpha=x,y,z} G_{\alpha} ([\mathbf{S}_i \times \mathbf{S}_j])_{\alpha} + D S_{z'}^2 + E (S_{x'}^2 - S_{y'}^2) + \sum_{\alpha,\beta=x,y,z} g_{\alpha\beta} \mu_B H_{\alpha} S_{\beta}, \quad (4)$$

where J is the isotropic exchange interaction parameter, $G_{\alpha=x,y,z}$ are the antisymmetric exchange interaction parameters in the coordinate system in which the z -axis is parallel to the external magnetic field, D and E are the crystalline field parameters in the local coordinate system (x', y', z'). The interaction with the magnetic field is given by the last term of the Hamiltonian.

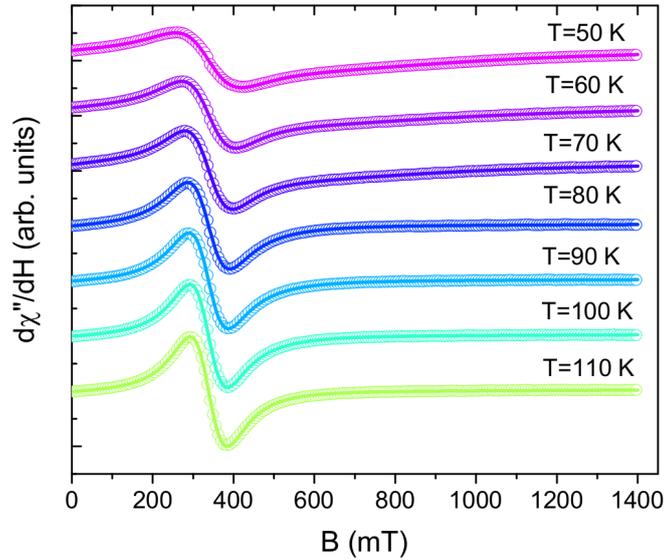


Figure 1. ESR spectra in the $\text{Sr}_2\text{MnTiO}_{5.87}$ ceramic in temperature range 50-110 K.

The second moment is determined by the sum of the contributions of anisotropic exchange interaction and single-ion anisotropy to the exchange-narrowed ESR line width from Mn^{4+} and Mn^{3+} ions with $S > 1/2$. The magnetic properties and features of the ESR spectra in manganites like the $\text{La}_{0.95}\text{Sr}_{0.05}\text{MnO}_3$ perovskite and $\text{Sr}_2\text{MnTiO}_{5.87}$ double perovskite are determined by manganese ions surrounded by six oxygen ions which form an octahedron. For $\text{La}_{0.95}\text{Sr}_{0.05}\text{MnO}_3$ the resonance-field and ESR line width data at 200 K have been described for three crystallographic planes only taking into account contributions of the crystalline field and antisymmetric anisotropic exchange interactions [8]. It has been shown that both Dzyaloshinskii-Moriya and

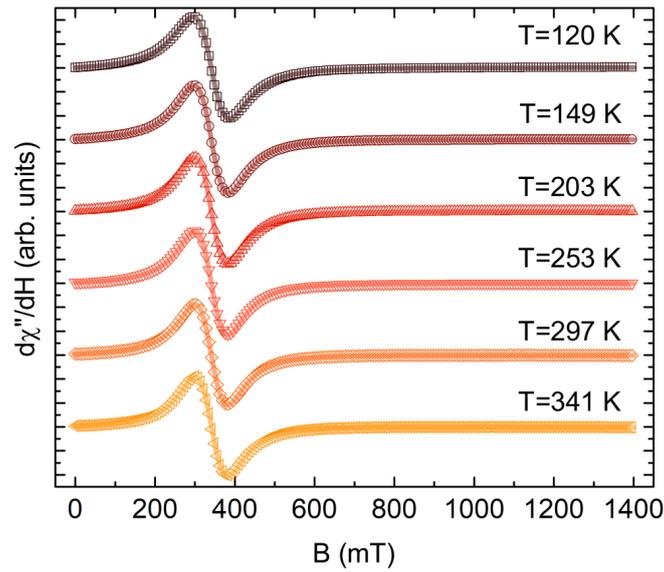


Figure 2. ESR spectra in the $Sr_2MnTiO_{5.87}$ ceramic in temperature range 120-341 K.

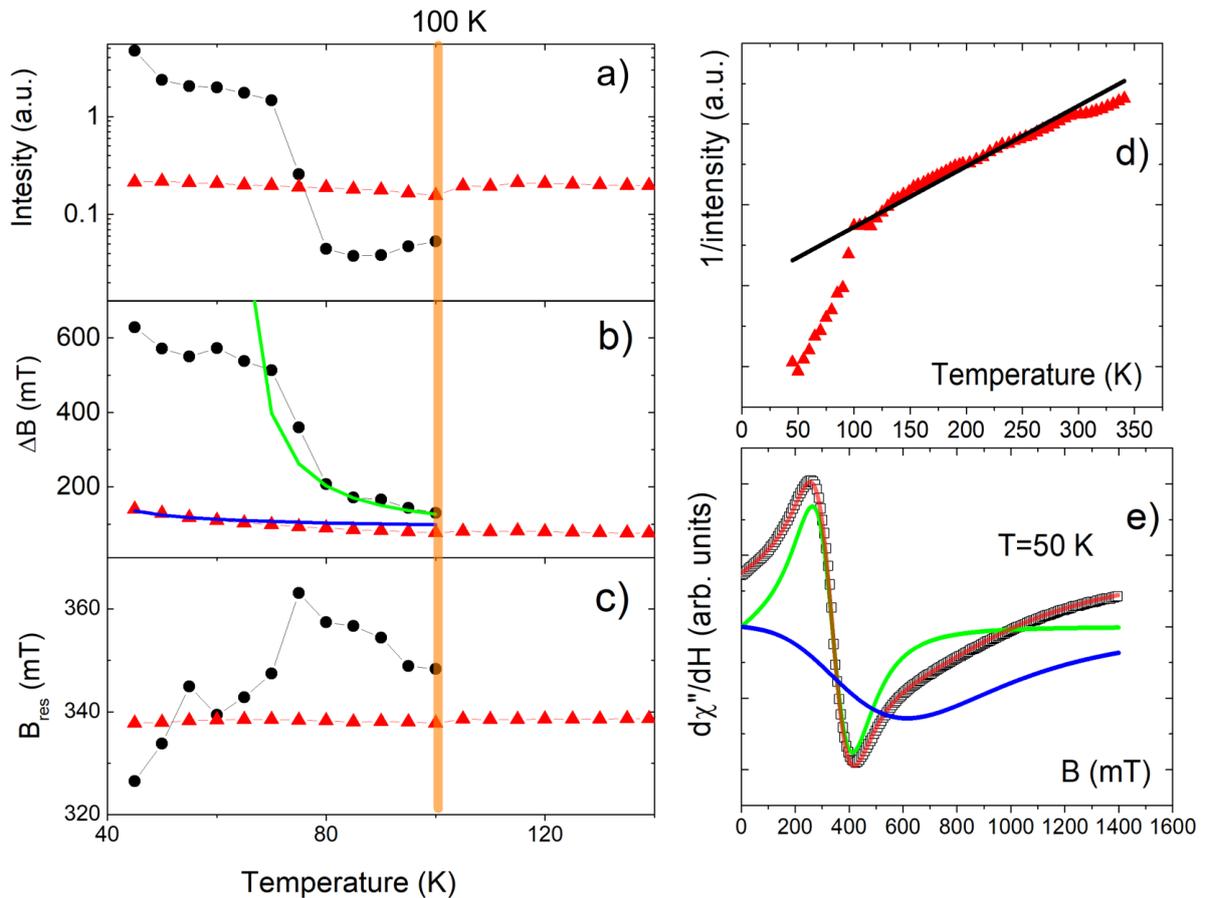


Figure 3. Temperature dependencies of (a) intensity; (b) line width; (c) resonance fields; (d) the inverse temperature dependence of the integral intensity of the first ESR line and (e) the decomposition of the spectrum into two ESR lines at 50 K in $Sr_2MnTiO_{5.87}$. The red triangles are related to the first ESR line, and the black circles are connected to the second ESR line. The black squares show the experimental ESR line. Solid lines are fit according equations (10) and (11) (see text).

crystalline-field interactions have the same order of magnitude and that the crystal-field parameters with $D = 0.73$ K and $E = -0.63$ K, indicate a strong mixing of orbital states $|3z^2 - r^2\rangle$ and $|x^2 - y^2\rangle$. Note that this early estimation of the crystal-field contribution was done in the Jahn-Teller phase of $\text{La}_{0.95}\text{Sr}_{0.05}\text{MnO}_3$, where 95% Mn^{3+} has 1 electron in the e_g orbital driving the Jahn-Teller effect. In the cooperative Jahn-Teller phase the octahedra exhibit the distortion which gives rise to the derived D and E crystal-field parameters. According to Huber *et al.* the crystal-field contribution in the related compound $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ is directly connected to the Mn^{3+} content [9]. Like in case of CaMnO_3 ($x = 1$), in $\text{Sr}_2\text{MnTiO}_6$ there is dominantly Mn^{4+} where only t_{2g} orbitals are occupied and hence no Jahn-Teller distortion is present. Therefore in pure $\text{Sr}_2\text{MnTiO}_6$, the line width should be mostly due to the Dzyaloshinskii-Moriya interaction with only Mn^{4+} (half-filled t_{2g} orbitals, $S = 3/2$). Nevertheless, an oxygen deficiency of 0.13 yields a Mn^{3+} content near 20 percent in ceramic $\text{Sr}_2\text{MnTiO}_{5.87}$ double perovskite, according to thermogravimetric analysis and analysis of magnetic susceptibility data. This minority of Mn^{3+} sites may show some local static (frozen) or dynamic Jahn-Teller distortions which give some contribution to the relaxation. But the effect should be weaker than in $\text{La}_{0.95}\text{Sr}_{0.05}\text{MnO}_3$. Therefore the above estimate gives only an upper limit for the crystal field contribution.

In the following we consider the contributions to the second moment due to the anisotropic antisymmetric exchange interaction and the crystal field sequentially. The contribution of the antisymmetric exchange interaction to the second moment is determined by the expression:

$$M_2 = \frac{2S(S+1)}{3} [G_x^2 + G_y^2 + G_z^2],$$

$$G_x = G_a \cos \beta \cos \alpha + G_b \cos \beta \sin \alpha - G_c \sin \beta,$$

$$G_y = G_a \cos \alpha - G_b \sin \alpha,$$

$$G_z = G_a \sin \beta \cos \alpha + G_b \sin \beta \sin \alpha + G_c \cos \beta.$$
(5)

where the angles α and β determine the transformation between the crystallographic coordinate system (a, b, c) and the coordinate system in which the z -axis is parallel to the external field. These α and β angles can be defined as:

$$\cos \alpha = \frac{A}{\sqrt{A^2 + B^2}}, \quad \cos \beta = \frac{C}{\sqrt{A^2 + B^2 + C^2}},$$
(6)

where

$$A = g_{aa} \sin \theta \cos \phi + g_{ab} \sin \theta \sin \phi + g_{ac} \cos \theta,$$

$$B = g_{ba} \sin \theta \cos \phi + g_{bb} \sin \theta \sin \phi + g_{bc} \cos \theta,$$

$$C = g_{ca} \sin \theta \cos \phi + g_{cb} \sin \theta \sin \phi + g_{cc} \cos \theta,$$
(7)

where the angles θ and ϕ determine the direction of the magnetic field relative to the a, b, c crystallographic axes.

The crystalline-field contribution to the second moment can be calculated as [5]:

$$M_2 = \frac{4S(S+1) - 3}{20 \hbar^2} [(\lambda_{xx} - \lambda_{yy})^2 + 4\lambda_{xy}^2 + (\lambda_{xx} + \lambda_{yy} - 2\lambda_{zz})^2 + 10(\lambda_{xz}^2 + \lambda_{yz}^2)],$$
(8)

where $\lambda_{\alpha,\beta}$ are determined by the parameters of the crystal field [5].

The superexchange interaction integral J , which enters equation (2) through the relation $\hbar\omega_{\text{ex}} = k_B J$, was estimated in the mean-field approximation using the Curie-Weiss temperature [8]:

$$\Theta_{\text{CW}} = \frac{S(S+1)}{3k_B} \sum J_i,$$
(9)

where J_i are isotropic exchange interactions, which in case of orthorhombic Jahn-Teller distortion differ in the interplane coupling J_{ac} with 4 neighboring Mn ions and the antiferromagnetic interplane bound J_b with 2 neighboring Mn ions. For La_{0.95}Sr_{0.05}MnO₃, assuming that $|J_{ac}| \approx |J_b| \approx J$ and taking into account that $S = 2$ for Mn³⁺, this approach yields $J=14$ K.

The Curie-Weiss temperature is obtained from the approximation of the inverse temperature dependence of the integral intensity of the ESR line, which is shown in the Figure 3d. Substituting $\Theta_{CW} = -400$ K from [1] into equation (9) and taking into account 6 nearest neighbors in a cubic lattice containing 80% Mn⁴⁺ ions with spin $S = 3/2$, 20% Mn³⁺ ions with spin $S = 2$, and 20% Ti³⁺ ions with spin $S = 1/2$, we estimated the average value of the antiferromagnetic isotropic exchange interaction in the Sr₂MnTiO_{5.87} ceramic as $\langle J \rangle \approx 46$ K. This is by a factor of 3.3 larger than in La_{0.95}Sr_{0.05}MnO₃ and can be understood by the smaller Mn-O distance in Sr₂MnTiO_{5.87}: for the nearest-neighbors manganese ion MnO₆ octahedra, the Mn-O distance in Sr₂MnTiO_{5.87} is 1.928(1) Å, while in La_{0.95}Sr_{0.05}MnO₃ the average distance is 1.978 Å (specifically, 2.004 Å for Mn-O_{2l}, 1.949 Å for Mn-O_{1m}, and 1.982 Å for Mn-O_{2s}) [10].

It should be noted that the weak ferromagnetic contribution to the magnetization observed in the ordered phase corresponds to a magnetic moment per unit cell not exceeding a hundredth of a μ_B [11]. Thus it is difficult to imagine a uniform distribution of Mn³⁺, Mn⁴⁺, and Ti³⁺ ions. If manganese Mn³⁺ and Mn⁴⁺ ions are nearest neighbors, a ferromagnetic double exchange is expected between them, with a higher ferromagnetic moment of about $0.7\mu_B$ per unit cell. Consequently, ions of the same valence are probably distributed over micro regions in the sample and are dominantly bound by antiferromagnetic exchange interactions.

Since the ESR linewidth in Sr₂MnTiO_{5.87} increases as it approaches the phase transition temperature, for the following estimate we will use the ESR line width at room temperature which is almost constant and equal to 610 Oe. To estimate the crystalline-field contribution to the ESR line width in Sr₂MnTiO_{5.87}, we use the results obtained in La_{0.95}Sr_{0.05}MnO₃ taking into account the larger isotropic exchange and the reduced number of Mn³⁺ ions. The maximum crystalline-field contribution to the ESR line width in La_{0.95}Sr_{0.05}MnO₃ is equal to 1600 Oe. As the average value of the isotropic exchange interaction in La_{0.95}Sr_{0.05}MnO₃ is smaller than in Sr₂MnTiO_{5.87} by a factor of 3.3, the estimation of the crystalline-field contribution to the ESR line width for 20% Mn³⁺ in Sr₂MnTiO_{5.87}, using equations (2)-(9), yields approximately 100 Oe. This estimation allows us to find the Dzyaloshinskii-Moriya contribution to the ESR line width as 550 Oe. Despite the cubic structure of Sr₂MnTiO_{5.87}, antisymmetric exchange interaction is nonzero due to the presence of Mn³⁺, Mn⁴⁺ and Ti³⁺ ions with different valence and distortion of the nearest-neighbor structure. Thus, using equations (2)-(9) allows to estimate the maximum of the possible value of the antisymmetric exchange interaction as 0.49 K. This value differs from the value of the Dzyaloshinskii-Moriya interaction in La_{0.95}Sr_{0.05}MnO₃ (~ 1 K) and is connected to the relative inclination of the MnO₆ octahedra.

Let's now consider the effect of fluctuations on the temperature dependencies of the ESR linewidth and position when approaching the magnetic phase transition temperature. We assume that the ΔB shift is due not to the components of the static magnetization, but to the fluctuating part of the magnetization. Figure 3 shows that, as temperature decreases, the ESR line width increases for first and second ESR lines.

The theory of critical broadening of the ESR line was developed in the works of Huber, Mori, and Kawasaki [12], [13] and for the first time successfully used to explain the behavior of $\Delta B(T)$ in antiferromagnets in the vicinity of the Néel temperature. In this model, the observed increase

in line width is described by the following formula:

$$\Delta B_N = \Delta B_0 + A_1 \left[\frac{T_N}{T - T_N} \right]^\beta, \quad (10)$$

where the first term $\Delta B_0 = 610$ Oe describes the high-temperature limit of the width of the exchange-narrowed line, which is temperature independent, while the second term is responsible for the critical behavior when approaching the temperature from above, T_N is the ordering temperature and β is the critical exponent. Using the equation (10) we described the ESR line width with $T_N = 37(1)$ K and $\beta = 1/3$, $A_1 = 40(2)$ Oe, as shown in the Figure 3b as blue curve. The temperature $T_N = 37$ K is in fair agreements with the magnetic ordering temperature [1]. Thus, the value of $\beta = 1/3$ extracted for $\text{Sr}_2\text{MnTiO}_{5.87}$ is equal for 3D antiferromagnets in the Heisenberg model [14].

The second line broadening upon approaching T_{VF} may be alternatively described in terms of a Vogel-Fulcher type scenario [15]. The temperature dependence of the ESR line width is very well approximated by the expression:

$$\Delta B_{KT} = A_2 \exp \left[\frac{b}{T - T_{VF}} \right], \quad (11)$$

with the Vogel-Fulcher temperature $T_{VF} = 54(2)$ K, the parameter $b = 28(6)$ K, the prefactor $A_2 = 690(8)$ Oe, as shown in the Figure 3b as green curve. The effects related to formation of a spin glass are easily observed in ESR experiments sensitive to spin dynamics. Measurements of ESR line width show increasing correlations starting at temperatures above the order temperature at temperature T_{VF} , which used to describe the spin-freezing process. We associate the separation of one ESR line into two below the temperature of 100 K with the phase separation observed in ceramics $\text{Sr}_2\text{MnTiO}_{5.87}$ and, accordingly, different spin dynamics in regions with different valency manganese ions. For this reason, two phase transition temperatures are observed in the imaginary part of the dynamic susceptibility. These characteristic temperatures fairly correspond to the peak and the shoulder observed by Meher *et al.* in the imaginary part of the AC magnetic susceptibility at about 39 K for $\text{Sr}_2\text{MnTiO}_6$ and minimum in inverse magnetic susceptibility (Fig.4) near 50 K, respectively [11]. The temperatures at which peaks are observed in the $\text{Sr}_2\text{MnTiO}_6$ samples may differ due to different oxygen stoichiometry, which depends on the synthesis conditions.

4. Summary

We performed measurements of the temperature-dependent $\text{Sr}_2\text{TiMnO}_{5.87}$ ESR spectra in the 50-340 K temperature range. Based on the ESR line width analysis at room temperature, we calculated the value of the antisymmetric exchange interaction. In particular, the Dzyaloshinskii-Moriya parameter was estimated as 0.49 K. In the molecular-field approximation, the average value of the isotropic exchange interaction is equal to $J \approx 46$ K. Fitting of the ESR line width yielded the Néel temperature of 37 K, which is consistent with the Néel transition temperature obtained from the temperature dependence of the magnetic susceptibility.

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