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Unusual magnetic properties of the polar orthorhombic $BiFe_{0.5}Sc_{0.5}O_3$ perovskite

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ABSTRACT

Magnetic properties of new multiferroic $BiFe_{0.5}Sc_{0.5}O_3$ perovskite which exists in two different orthorhombic polymorphs, the antipolar *Pnma* and the polar *Ima*2, were studied using a SQUID-magnetometer technique in magnetic fields up to 50 kOe in the temperature range of 5–300 K. Both polymorphs show a weak ferromagnetic component below the Néel temperature, $T_N \sim 220$ K. A wide cusp-like anomaly of the temperature dependent magnetic susceptibility was revealed below 200 K. The anomaly is dependent on the magnetic field applied and can be associated with the glassy magnetic behavior of the compound. The best fit of the *initial* magnetization curves taken at each temperature can be obtained as a sum of two contributions, namely the nonlinear in field and the linear one. The nonlinear contribution appeared to be temperature and magnetic field dependent, which is in a good agreement with the magnetization experimentally obtained as a function of temperature in different magnetic fields applied. Increase of external magnetic field leads to a decrease of the nonlinear contribution to the total magnetic moment.

1. Introduction

Multiferroics are materials which exhibit two or more ferroic properties simultaneously. Multiferroic magnetoelectrics combine both a permanent magnetization (ferromagnetism) and a permanent electric polarization (ferroelectricity). If the magnetization and polarization are coupled, they have a spontaneous magnetization that can be switched by an applied electric field, a spontaneous polarization that can be switched by an applied magnetic field. It opens new ways to control devices for spintronics and other applications. Multiferroics are considered as a most promising basis for developing novel electronic devices [1,2,3,4,5]. However, very few magnetoelectrics are known till now because ferromagnetism requires the transition metal *d* orbitals to be partially filled, which reduces the tendency for off-center ferroelectric distortion preventing the appearance of spontaneous polarization [6]. Consequently, an additional electronic or structural driving force must be present for ferromagnetism and ferroelectricity to occur simultaneously.

$BiFeO_3$ perovskite is a rare example of multiferroics that is both magnetic and strongly ferroelectric at room temperature. However, the polar nature of the crystal structure of $BiFeO_3$, in the magnetically ordered phase, induces a slow spin rotation due to the relativistic part of the exchange interactions, forming a long-period cycloid and averaging the local weak-ferromagnetic components to zero [7]. This problem is usually solved by means of chemical modifications. It should be noticed that the substitution in the Fe sublattice is rather problematic because of the limited solubility, even in the case of trivalent cations which are close to octahedrally coordinated Fe^{3+} in terms of ionic size. The high-pressure synthesis technique makes possible to extend considerably a solubility in a $BiFeO_3$-"$BiB^{3+}O_3$" system even when the size difference between Fe^{3+} and B^{3+} is big and to obtain new perovskite phases which cannot be stabilized at ambient pressure [8,9]. Using the high-pressure synthesis method, single-phase perovskite solid solutions derived from $BiFeO_3$, in which 50 and more at.% of iron was substituted either by the cations of transition metals (Mn^{3+}, Co^{3+}, Cr^{3+}) with the ionic size similar to that of Fe^{3+} or by Ga^{3+} whose size is by 8% smaller, have been produced [8]. In all the above-mentioned solid solutions a weak ferromagnetism exists (see, for example, [10,11,12]).

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The substitution of Fe^{3+} by Sc^{3+} appeared to be successful as well, a significant difference in sizes between scandium and iron (the ionic size of Sc^{3+} is by 24% bigger than that of Fe^{3+}) induces structure distortion, which permits to stabilize weak ferromagnetism in the system. Using a high-pressure synthesis a metastable perovskite $BiFe_{0.5}Sc_{0.5}O_3$ ceramics which are in the focus of the present study were prepared [13]. $BiFe_{0.5}Sc_{0.5}O_3$ can be obtained in two different orthorhombic polymorphs, namely the antipolar *Pnma* and the polar *Ima*2 [13]. It was found that the perovskite phase quenched under pressure is antipolar and can be tuned into the polar phase by means of irreversible heating/cooling thermal cycling. This polar *Ima*2 modification is a new type of a distorted perovskite structure which combines spontaneous polarization and magnetization below the Néel temperature $T_N \sim 220$ K.

Parent $BiFeO_3$ perovskite demonstrates ferroelectric phase transition at ~1100 K and a long-range antiferromagnetic (AFM) ordering below $T_N = 635$ K with no further indication of ferromagnetic (FM) or glass contributions down to 2 K [14,15,16]. A substitution of 50 at.% of iron by scandium results in a transformation from rhombohedral to orthorhombic symmetry and in a decrease of the Néel temperature of the compound and appearance of weak ferromagnetism. Below $T_N \sim 220$ K, both $BiFe_{0.5}Sc_{0.5}O_3$ polymorphs exhibit a long-range AFM ordering with a weak FM component driven by the Dzyaloshinskii-Moria antisymmetric exchange [13,17,18].

In the present work, we report the results of detailed studies of the temperature- and magnetic field dependent magnetic properties of the polar and antipolar polymorphs of the $BiFe_{0.5}Sc_{0.5}O_3$ perovskite. The data obtained are associated with a glassy magnetic behavior of the compound.

2. Experimental details

In order to achieve the most homogeneous and single-phase ceramics, the $BiFe_{0.5}Sc_{0.5}O_3$ precursor was prepared *via* a sol-gel combustion route using nitrates of the respective metals. High pressure was generated using an anvil press DO-138A with a press capacity up to 6300 kN equipped with a Bridgman-type apparatus. Ceramic samples were synthesized at 6 GPa and 1500 K for 1–3 min. The polar polymorph was obtained by annealing at 870 K during 3 h at ambient pressure.

X-ray diffraction study of the powdered samples was performed using a PANalytical X'Pert MPD PRO diffractometer (Ni-filtered Cu Kα radiation) at room temperature.

Neutron powder diffraction data were collected at the ISIS pulsed neutron and muon facility of the Rutherford Appleton Laboratory (UK) on the WISH diffractometer.

Static magnetic properties of the antipolar and polar $BiFe_{0.5}Sc_{0.5}O_3$ perovskites were studied using a SQUID magnetometer technique, namely using a commercial Quantum Design MPMS-3 and a homemade SQUID-magnetometer [19,20], between 5 K and 300 K in an applied fields up to 50 kOe under both zero-field-cooled (ZFC) and field-cooled (FC) conditions. Isothermal magnetization measurements were performed between −50 kOe and 50 kOe in the temperature range of 5–300 K after a ZFC process at the set of fixed temperatures.

Residual magnetic field is a well-known problem of the superconducting solenoids in SQUID-magnetometers. Residual fields of few dozens and sometimes even hundreds of Oersteds can remain in the solenoid after removal even of the medium magnetic fields (H ≥ 5 kOe). The use of a so-called *Oscillate Mode* does not always lead to success. We have developed and used the following practical algorithm to minimize the unwanted fields. After each measuring procedure a residual field in the solenoid was set to "zero" by using a standard *Oscillate Mode*. Then the field of 10 kOe was set with a speed of 0.7 kOe per second using a *No Overshoot Mode* and removed again with the same speed in an *Oscillate Mode*. After that by using only *No Overshoot Mode* with speed of 1.6 Oe per second the following set of fields was set in the solenoid: 10, 5, 2, and 1 Oe. After such a procedure, the field was controlled by measuring the magnetic moment of a standard paramagnetic sample. As a result, a residual field (in our case) did not exceed 0.1 Oe, and what is more, its sign always was the same. The described procedure was performed at 300 K with an exposure time of 10 s. It should be mentioned here that our homemade SQUID magnetometer has no uncontrolled residual field due to the low operating field up to 1000 Oe frozen and controlled by a Hall sensor [19,20].

The effect of uniaxial pressure of 0.25 kbar on *dc* magnetic susceptibility was measured in the temperature range of 10–220 K in the external magnetic field of 50 Oe using a special holder in the homemade SQUID-magnetometer. The corresponding installation and experimntal technique are similar to those described in Ref. [19].

3. Results and discussion

It was found from the XRD study that both the as-prepared and the annealed $BiFe_{0.5}Sc_{0.5}O_3$ ceramics are single-phase with the antipolar *Pnma* and the polar *Ima*2 perovskite structure, respectively [8,9,13].

3.1. Magnetic characterization

Both antipolar and polar $BiFe_{0.5}Sc_{0.5}O_3$ polymorphs show a weak ferromagnetic component below the Néel temperature estimated as $T_N \sim 220$ K: below this temperature, the magnetization gradually increases (Fig. 1). No saturation of magnetization is observed up to $H = 50$ kOe. Ferromagnetic contribution vanishes above T_N (Fig. 2).

For both polymorphs, the temperature dependent magnetization curves measured in ZFC and in FC modes in low magnetic fields are strongly different (Fig. 1), which may be associated both with a high anisotropy of the material studied, as well as with its glassy magnetic state [21,22].

Besides, the magnetic moment of the antipolar polymorph is more than twice as much as that of the polar one at any temperature below T_N. It has been found from analysis of the neutron diffraction data that although the magnetic scattering was very similar in $BiFe_{0.5}Sc_{0.5}O_3$ polymorphs, the magnetic reflections were broader in the polar *Ima*2 polymorph [13]. As one of the reasons of such an effect, the presence of an incommensurate modulation due to the polar nature of the *Ima*2 structure similar to that observed in $BiFeO_3$ was suggested. One can assume that the observed difference in the values of magnetic moments of the of $BiFe_{0.5}Sc_{0.5}O_3$ polymorphs is caused by incommensurate modulation which leads to lowering of the net ferromagnetic component.

Low field temperature dependent magnetization curves for the polar

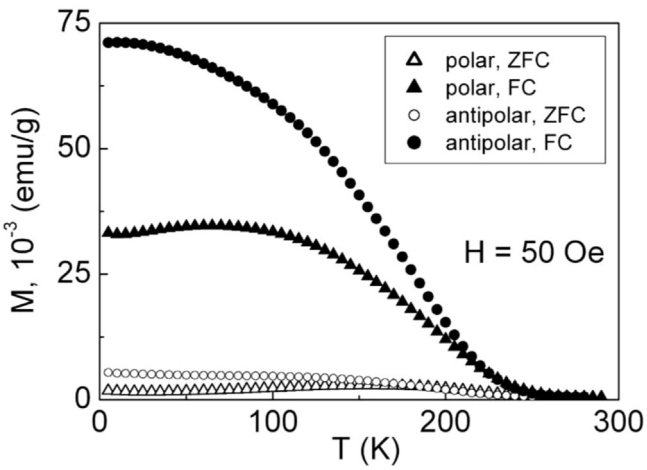

Fig. 1. Temperature dependent *dc* magnetic moment for $BiFe_{0.5}Sc_{0.5}O_3$ perovskite in polar and antipolar modifications measured in magnetic field of 0.05 kOe in ZFC and FC modes.

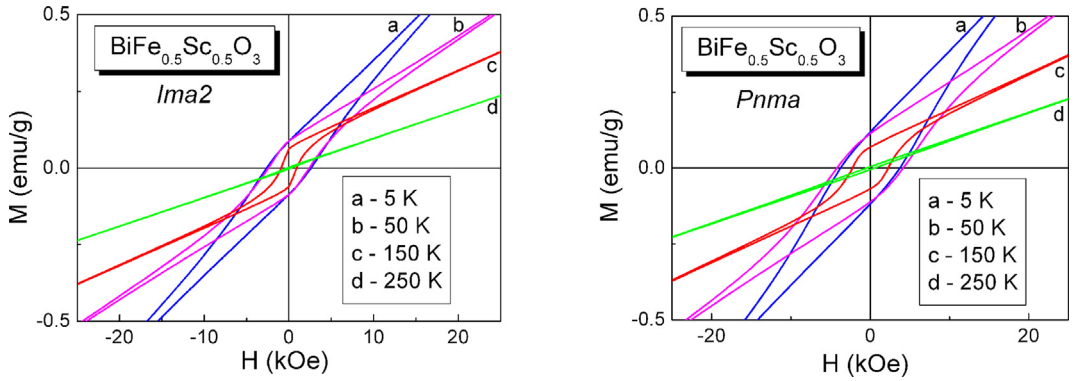

Fig. 2. Magnetic hysteresis loops for the (a) polar and (b) antipolar $BiFe_{0.5}Sc_{0.5}O_3$ polymorphs measured after a ZFC procedure at 5, 50, 150, and 250 K.

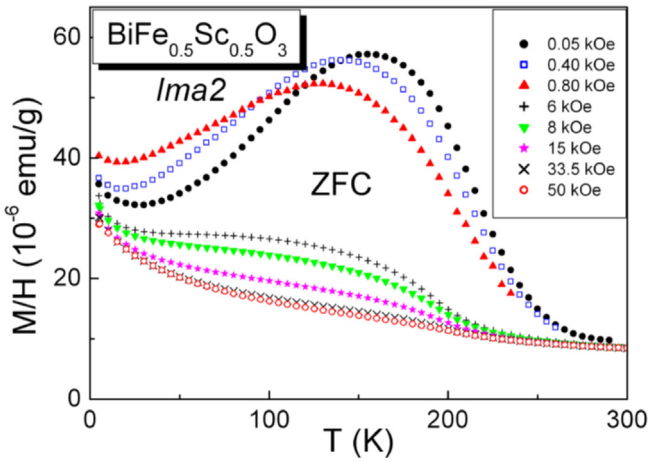

Fig. 3. Temperature dependent magnetic moment of the polar $BiFe_{0.5}Sc_{0.5}O_3$ polymorph measured in magnetic fields of 0.05–50 kOe in a ZFC mode and normalized to the measuring magnetic field.

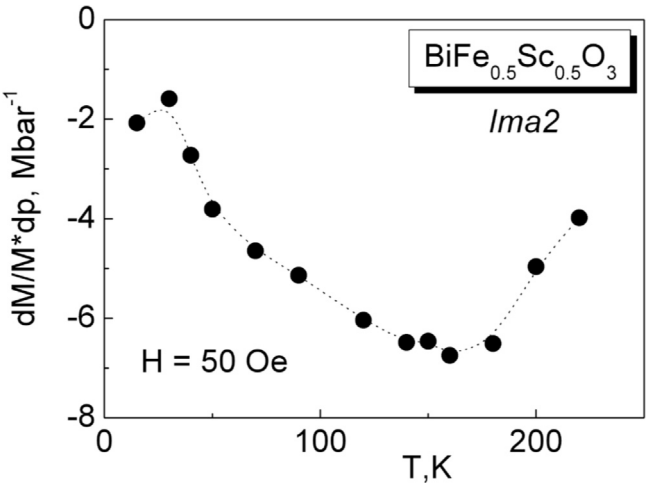

Fig. 4. Temperature dependent reduced change of the dc magnetic moment, $dM/M * dp$, under the uniaxial pressure $P = 0.25$ kbar for the polar $BiFe_{0.5}Sc_{0.5}O_3$ polymorph.

polymorph are non-monotonous (Fig. 3): the magnetization increases with cooling below the Néel temperature, reaches the highest value in the vicinity 120 K, then decreases and increases again below 20 K. Such a temperature behavior of the magnetization may be associated with a freezing of the magnetic moments and complex interplay between ferromagnetic and antiferromagnetic contributions. A similar behavior was found in the antipolar polymorph as well, although less pronounced, may be due to a higher magnetic moment value as compared with that of the polar polymorph.

The magnetic field applied significantly affects the magnetic state of the perovskite polymorphs. Fig. 3 shows the temperature dependent magnetic moment normalized to the measuring magnetic field, *M/H (T)*, for polar $BiFe_{0.5}Sc_{0.5}O_3$ polymorph measured in 0.05–50 kOe in a ZFC mode. Shape of the curves measured in relatively low magnetic fields (below 1 kOe) is strongly different from that of the curves measured in relatively high fields of 6–50 kOe. In low fields, a wide cusp-like anomaly of the temperature dependent magnetic susceptibility *M/H(T)* is observed below 200 K. The cusp is broadened in higher fields and shifts toward lower temperatures. At high fields, the temperature dependent ZFC curves demonstrate no cusp; the ZFC and FC data practically coincide in magnetic fields of 30–50 kOe. Such a behavior is reminiscent of spin-glass or cluster-glass systems [23].

To understand better the unusual magnetic behavior of the compound, an effect of low uniaxial pressure on the temperature dependent magnetic moment was studied. In the present study, 0.25 kbar was applied to reveal temperature ranges of the magnetic instability and possible magnetic phase transitions. It was found that only the polar polymorph of $BiFe_{0.5}Sc_{0.5}O_3$ is sensitive to the low uniaxial pressure applied (Fig. 4). The change of the magnetic moment is negative all over the studied temperature range. *dM/M* curve has a bright minimum in the vicinity of 150 K, which is in a good agreement with the anomaly of the *M/H(T)* dependence measured in the same magnetic field (Fig. 2). The anomaly found is associated with the transformation of the magnetic structure, possibly, with the formation of the glassy magnetic state of the compound. It should be noticed that a careful examination of the neutron diffraction patterns collected between 1.5 and 300 K (see Ref. [13] for details of the neutron diffraction experiment) has revealed no evidence of the phase coexistence.

For both polymorphs studied, the magnetic hysteresis loops taken below T_N change their shape with decrease of temperature (Fig. 2). In the vicinity of 150 K, the loops are the most rectangular indicating a ferromagnetic behavior; then they are blurred with temperature decreasing down to 50 K. At the same time, the coercive field at 50 K is almost twice bigger than that at 150 K. It may be associated with a formation of the magnetic glass state below about 150 K. Similar to the case of magnetic moments, the coercive field values are about twice higher for the antipolar (as prepared) polymorph as compared with the polar (the annealed) one.

3.2. Magnetic field dependent magnetization

Initial magnetization curves *M(H)* for the polar *Ima*2 polymorph of $BiFe_{0.5}Sc_{0.5}O_3$ were measured in magnetic fields of up to 50 kOe at 5, 25, 50, 100, 150, 200, 250, and 300 K after zero-field cooling down to the corresponding temperatures (Fig. 5).

It has been appeared that in a range of a relatively low magnetic field (up to 15 kOe) the field dependent magnetization taken at each

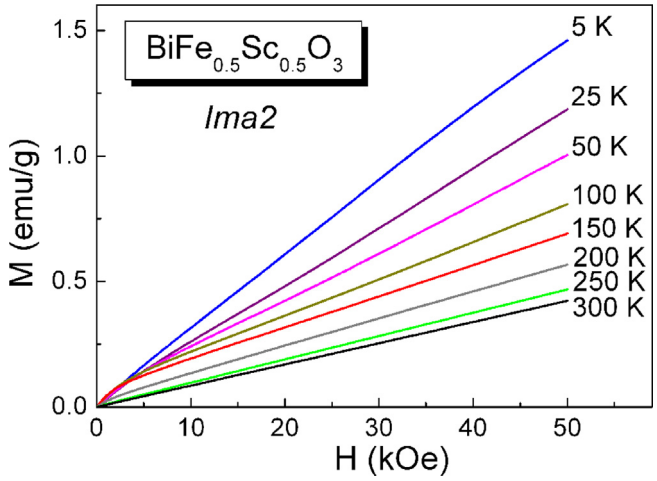

Fig. 5. *Initial* magnetization curves for the polar $BiFe_{0.5}Sc_{0.5}O_3$ polymorph measured after a ZFC procedure at different temperatures.

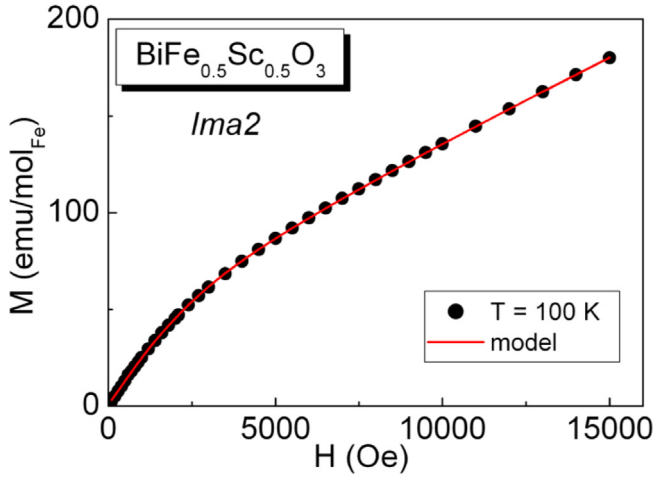

Fig. 6. Experimental *initial* magnetization curve *M(H)* for the polar $BiFe_{0.5}Sc_{0.5}O_3$ polymorph measured after zero field cooling at 100 K (solid circles) fitted using Eq. (1) (solid line).

temperature below T_N can be well fitted as a sum of two contributions, namely the nonlinear in the field and the linear one. The experimental data for the fitting procedure were recalculated in the iron mass fraction as the magnetic moment of the studied compound is generated by the iron ions only. The best fit of the experimental *initial* magnetization curves was obtained using the following equation:

$$M(H) = M_1 + M_2 = N\left(cth\left(\frac{M_{eff}\mu_B H}{k_B T}\right) - \left(\frac{M_{eff}\mu_B H}{k_B T}\right)^{-1}\right) + \chi H, \quad (1)$$

where M_1 and M_2 are the contributions nonlinear in the field and the linear one, correspondingly; μ_B is Bohr magneton, k_B is Boltzmann constant, H is magnetic field, T is temperature, χ is magnetic susceptibility, N is quantity of magnetic particles, and M_{eff} is effective magnetic moment of each magnetic particle.

The first term, $M_1 = N\left(cth\left(\frac{M_{eff}\mu_B H}{k_B T}\right) - \left(\frac{M_{eff}\mu_B H}{k_B T}\right)^{-1}\right)$ is a Langevin function, which describes magnetization of ideal paramagnetics [24]. Although initially the Langevin function was used for description of paramagnetic state, it was shown by P. Weiss [25] that it can be also applied to the magnetic ordered state. The second term, $M_2 = \chi H$ describes the linear AFM contribution to the total magnetic moment. An example of such approximation is shown in Fig. 6.

To understand better a complex quasi equilibrium magnetic state of $BiFe_{0.5}Sc_{0.5}O_3$, a distribution of Fe and Sc ions in the Fe/Sc sublattice was simulated [26]. The modelling showed that almost all iron ions (about 98%) form a connected area, and an occurrence of separated groups of iron ions, clusters, is hardly possible. Hence, the weak ferromagnetic contribution is not coming from ferromagnetic impurities (clusters of Fe ions).

Temperature dependent contributions to the total magnetic moment, $M_1(T)$ and $M_2(T)$, in the applied magnetic field of 6 kOe and 15 kOe are shown in Fig. 7. It is seen, that the nonlinear contribution is enough large (~40% of total magnetic moment) in the temperature range of 100–150 K, which is in a good agreement with the experimental temperature dependent magnetic moment (Fig. 3). An increase of external magnetic field results in a suppression of this contribution to the total magnetic moment.

4. Conclusions

Magnetic properties of the antipolar *Pnma* and the polar *Ima*2 perovskite polymorphs of new multiferroic $BiFe_{0.5}Sc_{0.5}O_3$ have been studied using a SQUID-magnetometer technique in magnetic fields up to 50 kOe in the temperature range of 5–300 K. Both polymorphs show a weak ferromagnetism below the Néel temperature $T_N \sim 220$ K. In low magnetic fields, magnetic moment of the polar polymorph is less than half the moment of the antipolar (the as prepared) one at any temperature below T_N. Magnetic moment of the polar polymorph, in contrast to that of the antipolar one, was found to be sensitive to the low uniaxial pressure applied.

The best fit of the *initial* magnetization curves taken at each temperature is obtained as a sum of two contributions, the nonlinear

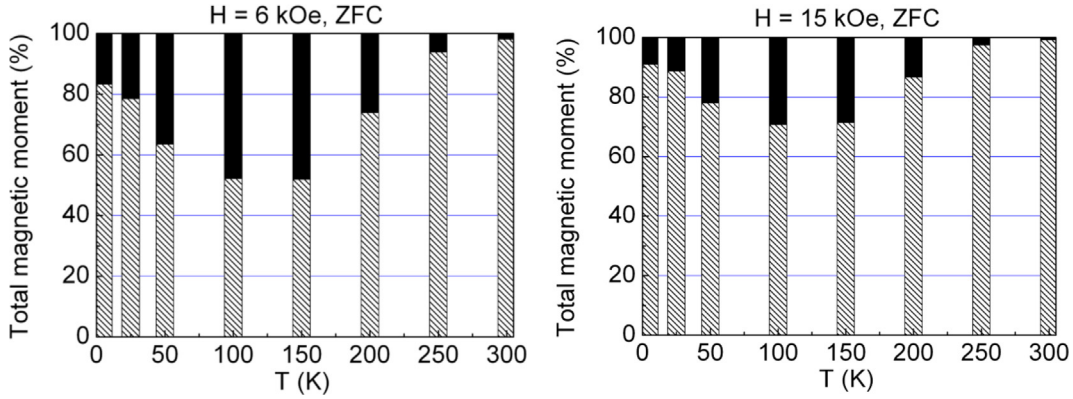

Fig. 7. The temperature dependent nonlinear (solid black columns) and linear (shaded columns) contributions to the total magnetic moment, $M_1(T)$ and $M_2(T)$, correspondingly, in the applied magnetic field of 6 kOe (a) and 15 kOe (b) obtained by fitting of the experimental *initial* magnetization curves *M(H)* for polar $BiFe_{0.5}Sc_{0.5}O_3$.

contribution and the linear one. The nonlinear contribution is found to be temperature and magnetic field dependent. An increase of external magnetic field leads to a decrease in the nonlinear contribution to the total magnetic moment.

A wide cusp-like anomaly of the temperature dependent magnetic susceptibility *M/H(T)* is revealed below 200 K. The temperature of the cusp maximum shifts to the low temperature range with an increase of the applied magnetic field while its amplitude decreases. The anomaly found is associated with the glassy magnetic behavior of the compound.

Acknowledgments

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