Ultrasound study of $FeCr_2S_4$ in high magnetic fields

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Abstract

We report on ultrasound studies of $FeCr_2S_4$ in static and pulsed magnetic fields exhibiting an orbital-order transition at 9 K. A longitudinal acoustic mode exhibits distinct features in the phase space of temperature and magnetic field due to magnetic and structural transformations. Pulsed-field measurements show significant differences in the sound velocity below and above the orbital-ordering transition as well as the spin-reorientation transition at 60 K. Our results indicate a reduction of the magnetocrystalline anisotropy on entering the orbitally ordered phase.

1. Introduction

The ferrimagnetic spinel $FeCr_2S_4$ belongs to a family of ternary magnetic chalcogenides which manifest a rich variety of fascinating magnetic and electronic phenomena [1, 2], i.e. colossal magnetoresistance [3, 4], high magneto-capacitive coupling and multiferroicity [5, 6], or half-metallicity [7]. The remarkable properties of magnetic chalcogenides are also of high interest for basic research as well as for spintronic applications. The unconventional magnetic and electronic properties of $FeCr_2S_4$ are known since more than four decades; however, the origin of several effects observed in this compound is still under debate. In particular, this concerns the orbital-ordering (OO) transition at $T_{OO} \approx 10$ K and the low-field magnetization irreversibility that appears below 60 K. A step-like change of the electric field gradient at the Fe sites observed at 10 K in Mössbauer experiments [8] on polycrystalline $FeCr_2S_4$ and a λ-like anomaly in the specific heat at the same temperature [9] were interpreted as evidence for a static cooperative Jahn-Teller (JT) effect related to the orbital degeneracy of the Fe^{2+} ions in a $3d^6$ configuration [3, 4, 10]. The anomaly in the specific heat at T_{OO} can be tuned by controlling the stoichiometry of the samples [2, 9, 11]. When the anomaly is fully suppressed, the orbital degrees of freedom undergo a freezing into a complex orbital state described as an orbital glass [12]. Earlier x-ray and neutron powder-diffraction studies [13, 14] have not detected any structural anomaly at T_{OO} expected for a JT transition, although inhomogeneous lattice distortions were revealed below the Curie temperature, $T_C \approx 170$ K, down to

4.2 K [15]. Single crystal x-ray diffraction studies on samples with an orbital-glass state did not find any structural transition and reported a cubic spinel structure for temperatures down to 4 K [16]. In contrast, a very recent high-resolution synchrotron x-ray diffraction study of $FeCr_2S_4$ crystals with an orbitally ordered ground state found a broadening of the diffraction lines that sets in below 60 K, reaching a maximum at T_{OO}, indicating possible structural anomalies at these temperatures [17]. Close to the orbital-order transition a clear peak in the temperature dependence of the mean-square displacements was found for all ions, with the strongest effect for iron and the weakest for chromium. It is also worth mentioning the observation of a field-induced anomaly in the magnetization in fields above 5 T in polycrystalline $FeCr_2S_4$ which is present only in the orbitally ordered phase [18, 19]. Note that the magneto-crystalline anisotropy in $FeCr_2S_4$ is rather strong (of the order of 10 T) due to the combination of spin–orbit coupling and crystal-field splitting of the Fe orbital states of about 2500 cm^{-1} [20, 21].

An irreversibility of zero-field-cooled (ZFC) with respect to field-cooled (FC) magnetization was found in $FeCr_2S_4$ below $T_m \sim 60$ K [22]. It was further studied by electron-spin-resonance [23], *ac* susceptibility [24], ultrasound spectroscopy [25] and transmission-electron microscopy [26]. A strong influence of hydrostatic pressure on the magnetic anomaly at T_m revealed considerable spin-lattice coupling in this compound [27]. A reduction of the crystal symmetry and magnetic domain reorientation processes have been proposed as explanation for the anomaly at 60 K in single crystals [24–27] while other studies on polycrystalline $FeCr_2S_4$ attribute the magnetic irreversibilities to coercivity and magnetic anisotropy [28, 29].

A major progress in understanding the low-temperature magnetic behaviour was provided by μSR experiments [30] which established a transformation of the collinear ferrimagnetic structure into an incommensurate (possibly helical) structure below 50 K, which remains the stable spin configuration in the ground state. Recent Mössbauer studies [31] found three non-equivalent Fe sites appearing below 50 K which corroborates the μSR results regarding the change of the spin structure from collinear to a helical one. To get further insight into the intriguing physics of $FeCr_2S_4$ we utilized ultrasound spectroscopy using a propagation techniques which are known to be very sensitive for detecting spin, orbital and structural correlations [32]. The measurements were performed on recently synthesized bulk samples which exhibit a pronounced anomaly in the specific heat at T_{OO} associated with the orbital-order transition [17] allowing to study Jahn–Teller and spin-strain interactions. Earlier ultrasound studies [25] were performed on samples with an orbitally disordered state and in fields below 2 T. The present study in high magnetic fields is aimed to reveal the role of the magnetocrystalline anisotropy in the magnetic anomalies mentioned above.

2. Experimental details

Dense samples of $FeCr_2S_4$ were prepared by spark plasma sintering (*SPS*) similar to [33]. Details on the sample preparation and characterization of their quality and stoichiometry were published earlier [17]. The elastic properties were studied by measurements of the velocity of the longitudinal waves with wave vector k and polarization u. The ultrasound measurements in static magnetic fields up to 14 T were performed for temperatures between 1.5 and 300 K. A phase-sensitive detection technique based on a pulse-echo method [34] was used. For generation and detection of the ultrasound waves *PVDF* film transducers working at a frequency of ∼62 MHz were used. Further ultrasound measurements were carried out in pulsed magnetic fields up to 62 T with a rise time of 35 ms and pulse duration of 150 ms in the temperature range from 1.5 to 200 K. The magnetic properties were studied using *SQUID* magnetometers (Quantum Design *MPMS*-5 and *MPMS*-7) in static fields up to 7 T.

3. Results and discussions

In figure 1 the temperature dependence of the relative change of the sound velocity, $\Delta v/v$, (upper panel) is shown together with that of the magnetization (lower panel) measured in FC and ZFC sequences. Below 0.5 T, ZFC and FC magnetizations of the *SPS* sample reveal two pronounced features at temperatures above 60 K, similarly to the single-crystalline sample studied in [22]. The onset of ferrimagnetic order is marked by a step-like increase of the magnetization below the Curie temperature $T_C = 165$ K. On decreasing temperature below T_C, both ZFC and FC magnetizations show a non-monotonic temperature dependence with increasing magnetization up to $T_m = 65$ K and decreasing below this temperature. At lower temperatures, FC and ZFC magnetizations of the *SPS* sample exhibit a third anomaly, an upturn and downturn at 9.8 K, respectively, related to the onset of long-range orbital order at T_{OO}. It is interesting to note that the ferrimagnetic transition is indicated only by a small dip in the sound velocity, while the spin-reorientation is visible via a broad and dominant maximum.

On decreasing temperatures from 200 to 60 K, the sound velocity, $\Delta v/v$, of the longitudinal mode exhibits an overall increase of about 1.3% indicating a significant stiffening of the lattice. The sound velocity shows a broad maximum at about 60 K and decreases towards lower temperatures. These results are in good agreement with earlier ultrasound studies of this sample performed in zero magnetic field utilizing an experimental setup with $LiNbO_3$ transducers at a frequency of 10 MHz [17]. The maximum in $\Delta v/v$ is broader and occurs at temperatures slightly lower than the maximum in the FC and ZFC magnetizations, which might be due to a superposition of anharmonic lattice contributions and changes in the spin system [35]. The softening of the acoustic lattice vibrations continues down to the orbital ordering temperature T_{OO}, where the sound velocity reveals a sharp minimum at a temperature of 8.7 K. This correlates well with the anomalies in the ZFC and FC magnetization at T_{OO} and with the maximum in the specific heat at this temperature reported in previous studies [17]. On decreasing the temperature below T_{OO}, the sound velocity increases again indicating a further stiffening of the lattice. Such a behaviour of the sound velocity is commonly observed in orbitally ordered Jahn–Teller systems [36, 37] and evidences strong coupling of strain fields to orbital degrees of freedom.

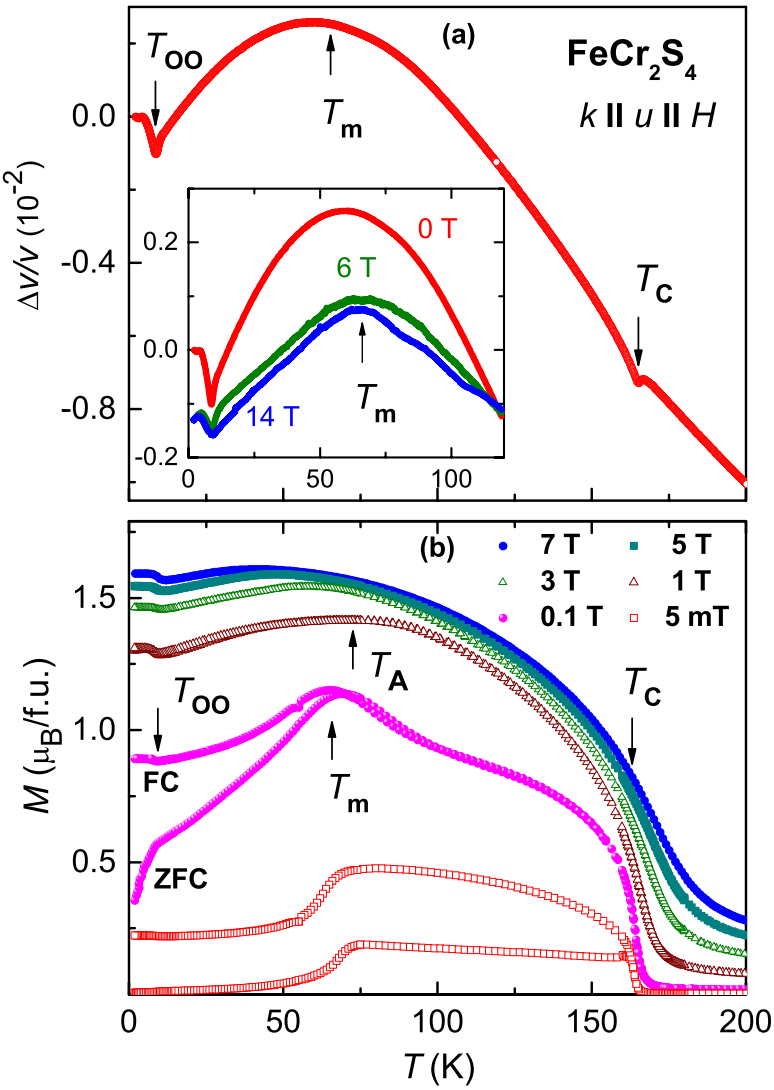

Figure 1. (a) Temperature dependence of the relative change of the sound velocity, $\Delta v/v$, of the longitudinal acoustic mode normalized to its value at 1.6 K measured at a frequency of 62 MHz in zero field. Inset: temperature dependencies of the $\Delta v/v$ for different applied magnetic fields. (b) Temperature dependence of the ZFC and FC magnetization measured in different magnetic fields. The arrow at T_A marks the temperature where the magnetization reaches its maximum in a field of 1 T. The characteristic temperatures related to phase transformations at the OO transition, T_{OO}, appearance of the magnetic irreversibility, T_m and at the Curie temperature, T_C, are indicated by vertical arrows.

The effect of a static magnetic field on the sound velocity is shown in the inset of figure 1(a). The zero-field data are normalized to the value of the sound velocity at 1.6 K while the data in applied magnetic fields are normalized taking into account the field dependence of $\Delta v/v$ for the lowest temperature of 1.6 K which will be discussed later. Importantly, the data for all fields match each other at around 120 K implying a negligible effect of reorientation of magnetic domains and magnetocrystalline anisotropy which is expected at high temperatures. Under applied magnetic fields the sound velocity still keeps the non-monotonic temperature dependence between T_C and T_{OO}, however, with reduced changes in the absolute value compared to the data in zero field. At the same time, the maximum of $\Delta v/v$ is shifted to higher temperatures (65 K) for fields of 6 and 14 T while the temperature of the minimum in $\Delta v/v$ at the OO transition is only slightly shifted to higher temperatures in agreement with recent specific-heat measurements in magnetic fields [38].

Figure 1(b) presents the temperature dependence of the magnetization of the sample in different applied fields. In the lowest field of 5 mT, the difference between FC and ZFC magnetizations appears just below T_C due to the contribution of domain effects. Already in a field of 0.1 T the irreversibility due to domain effects disappears and the difference between ZFC and FC is detected only below T_m. With further increasing magnetic field, the irreversibility between FC and ZFC magnetizations becomes less pronounced and for fields above 1 T the magnetization anomaly at T_m is not further distinguished. In contrast to the anomaly at T_m, the anomaly in the magnetization at the orbital-order transition at T_{OO} is still clearly visible up to the highest applied field (7 T). However, the downturn in the ZFC magnetization seen in low fields disappears in high fields, while both FC and ZFC magnetizations show only an upturn below T_{OO}.

In magnetic fields of 1 T and above, the magnetization M in figure 1(b) shows an additional feature, namely, a broad

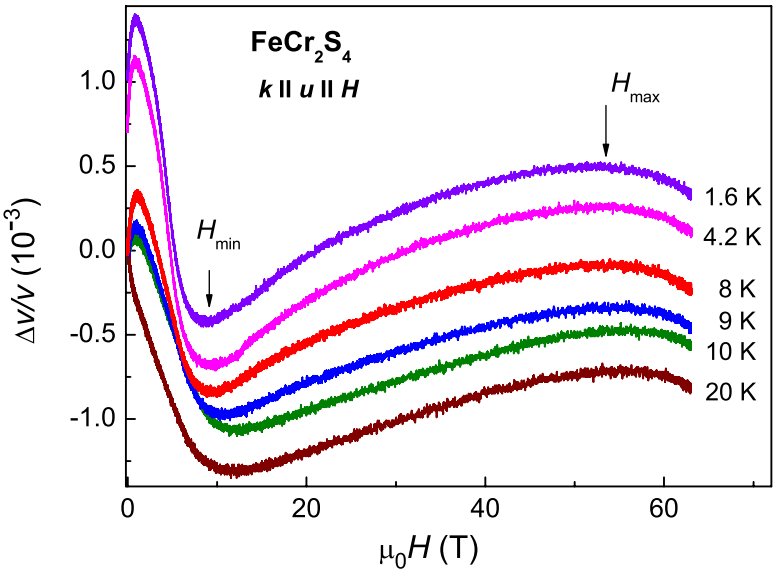

Figure 2. Magnetic-field dependence of the relative change of the sound velocity, $\Delta v/v$, for different temperatures around the orbital-order transition. All curves are normalized to the zero field value. Curves at 4.2 and 1.6 K are shifted for clarity. Arrows at $H_{\rm min}$ and $H_{\rm max}$ mark the fields of minimum and maximum in $\Delta v/v$, respectively, at 1.6 K.

but well discernible maximum, at a temperature $T_{\rm A}$, which is shifted to lower temperatures on increasing fields revealing effects of magnetocrystalline anisotropy. The decrease of the magnetization on decreasing temperatures below $T_{\rm A}$ indicates that the magnetocrystalline anisotropy overcomes the applied field leading to a reduction of the measured longitudinal component of the magnetization. The observed shift of the maximum of the magnetization at $T_{\rm A}$ with decreasing temperature implies a correlation between the temperature $T_{\rm A}$ and the anisotropy field.

As was already noticed, in fields above 0.1 T the sample is in a single-domain state at all temperatures below $T_{\rm C}$. Therefore, the increase of the magnetization above this field reflects the effect of competition of the applied magnetic field and anisotropy forces. In a polycrystalline ferrimagnetic sample, such as the present *SPS* sample, the information on the anisotropy field can be obtained by the method developed in [39]. This method utilizes the derivatives of the magnetization curve $M(H)$ which locate a singular point corresponding to an anisotropy field $H_{\rm A}$. However, for the *SPS* sample the derivatives of the magnetization curve do not show a clear peak-like anomaly at $H_{\rm A}$ but only a smooth change of slope. Therefore, we rely on the temperature dependent data measured in a given constant magnetic field H. Then, we determine $T_{\rm A}$ as the temperature at which the magnetization reaches the maximum and define the anisotropy field $H_{\rm A}(T_{\rm A})$ as the applied magnetic field H. This approach is valid for the single-domain state and in fields approximately above 1 T where the low-field magnetic irreversibilities are masked by anisotropy effects.

Figure 2 presents the magnetic-field dependence of the relative changes of the sound velocity, $\Delta v/v$, measured in pulsed fields for several temperatures below 20 K. The sound velocity reveals non-monotonic changes with magnetic field. At the lowest temperature of 1.6 K on increasing magnetic field, $\Delta v/v$ shows an initial growth with a sharp maximum at about 1 T. At temperatures above 10 K, this low-field maximum vanishes. At fields above 1 T, $\Delta v/v$ exhibits a strong decrease with a minimum at $H_{\rm min}$. The maximal changes of $\Delta v/v$ with field at 1.6 K are about 0.2% and by more than one order of magnitude larger than the magnetostriction reported in 38, indicating a weak contribution of this effect to the ultrasound velocity changes. With increasing temperatures and approaching the orbital-order transition the variations of $\Delta v/v$ become somewhat weaker. In the orbitally ordered state (below 10 K) a pronounced hysteresis in the sound velocity was observed for up and down field sweeps (not shown here) possibly indicating the irreversibility in the spin alignment in the polycrystalline sample in fields up to 4 T. For magnetic fields higher than $H_{\rm min}$, the sound velocity exhibits a further increase with a second broad maximum at $H_{\rm max}$ which varies within the range from 53 to 56 T on increasing temperature from 1.6 to 20 K with a notable anomaly at $T_{\rm OO}$ (see inset in figure 4). No hysteretic behaviour above $H_{\rm min}$ is detected suggesting that in this case the magnetic field overcomes the anisotropy forces and the magnetization reaches full saturation. At temperatures between 10 K and T_m, the sound velocity decreases monotonously with fields up to $H_{\rm min}$ without any hysteresis. The disappearance of the sharp maximum in $\Delta v/v$ at low fields at temperatures above 10 K indicates that this anomaly is not related to a movement of magnetic domains.

The magnetic-field dependence of the relative change of the sound velocity, $\Delta v/v$, at temperatures between 60 and 180 K is presented in figure 3. At 60 K, $\Delta v/v$ shows an initial decrease similar to that observed at lower temperatures (figure 3) with $H_{\rm min}$ shifted to lower fields compared to the data at 20 K. Between 80 and 100 K, $\Delta v/v$ changes sign compared to that at lower temperatures. We notice that the change of sign in $\Delta v/v$ occurs at temperatures about 30 K higher than the sign change of the magnetostriction which takes place at around T_m [38] implying notable differences of these two effects. For high fields, $\Delta v/v$ shows a qualitatively

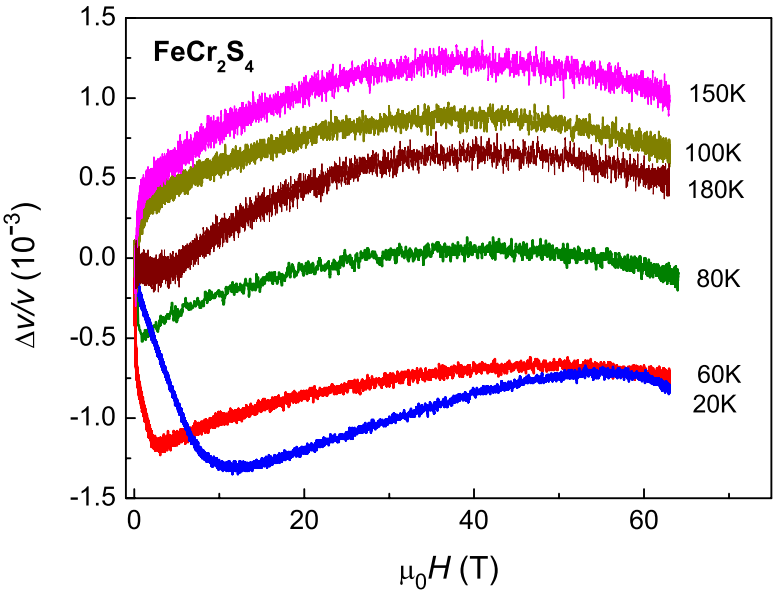

Figure 3. Magnetic-field dependence of the relative change of the sound velocity, $\Delta v/v$, for temperatures between 60 and 180 K.

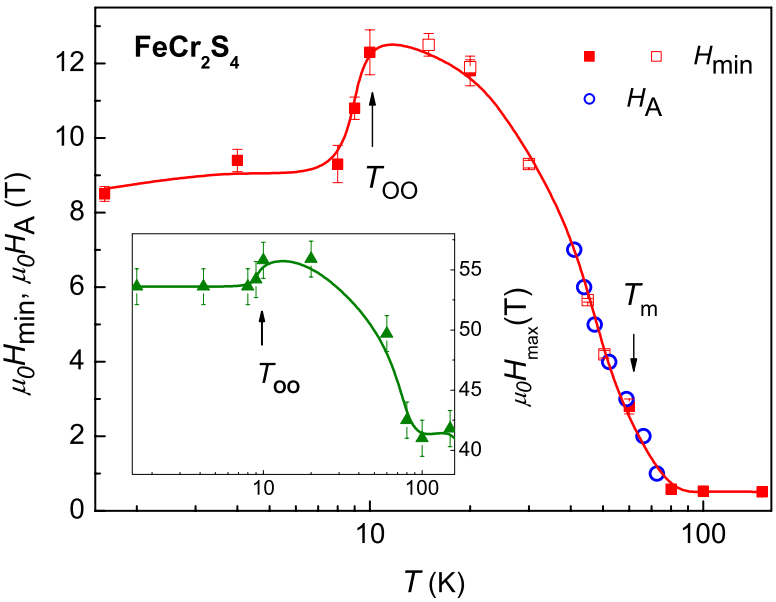

Figure 4. Temperature dependence of the characteristic field $H_{\rm min}$ (squares) and of the anisotropy field $H_{\rm A}$ (circles) extracted from the ultrasound and magnetization data, respectively. The open and closed squares correspond to static and pulsed fields, respectively. The data are shown on a semilogarithmic plot to emphasize the low-temperature features. The solid line is a guide to the eye. The inset shows the temperature dependence of the broad maximum in $\Delta v/v$ observed at high fields.

similar behaviour as below 20 K with a broad maximum shifted from 50 to 42 T on temperatures increasing from 60 to 150 K (see inset in figure 4). Above the Curie temperature, $\Delta v/v$ exhibits negligible changes at low fields, but for high fields the variations of $\Delta v/v$ are also similar to those in the magnetically ordered state between $T_{\rm C}$ and T_m.

Finally, in figure 4 the temperature dependence of the characteristic field $H_{\rm min}$ derived from the pulsed-field ultrasound measurements is presented together with that of the anisotropy field $H_{\rm A}$ estimated from the magnetization data. An obvious correlation between these two quantities in the temperature range above 40 K suggests that the changes in $H_{\rm min}$ follow the changes of the magnetocrystalline anisotropy. A quite similar behaviour in this temperature range was reported for the anisotropy constant extracted from the magnetic-torque measurements of single-crystalline samples [40]. At low temperatures $H_{\rm min}$ reveals a non-monotonic behaviour with a maximum at $T_{\rm OO}$ and decreasing values in the orbitally ordered state. The decrease of $H_{\rm min}$ below $T_{\rm OO}$ suggests a significant reduction of the magnetocrystalline anisotropy which may result from additional splitting of the orbital doublet ground state of the tetrahedral Fe^{2+} ions by the low-symmetry component of the crystal field related to the static Jahn–Teller effect. Such behaviour was neither found in earlier studies performed on $FeCr_2S_4$ single crystals with an orbital-glass ground state [22, 25, 27] nor in crystals grown by chlorine

transport [19, 20] in which the λ-like anomaly in the specific heat is suppressed. This implies that the stabilization of orbital order significantly modifies the magnetic anisotropy. It is worth to notice that the magnetic-field-induced anomaly in the magnetization found below 10 K in orbitally ordered polycrystalline $FeCr_2S_4$ [18] together with the anomalous behaviour of the spontaneous magnetostriction observed in the temperature range around T_{OO} in our sample [38] can be well understood as due to a modification of the magnetocrystalline anisotropy.

4. Summary

In conclusion, our ultrasound studies in static and pulsed magnetic fields of a $FeCr_2S_4$ *SPS* sample with orbital order reveals a significant difference in the behaviour of the sound velocity below and above the orbital-order transition at $T_{OO} \approx 10$ K and the spin-reorientation transition at $T_m \approx 60$ K. Together with the magnetization data, the obtained results provide evidence for the reduction of the magnetic anisotropy in the orbitally ordered state.

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