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Abstract
We have investigated magnetoelastic coupling in $Sr_2(Fe_{1-x}Cr_x)ReO_6$
polycrystalline double perovskites. The end compound, Sr_2CrReO_6, shows
a high ferromagnetic transition temperature of 635 K and is thought to
exhibit a nearly half-metallic conduction band. We probed the unexpected
high orbital moment borne by the Re atom by means of volume and
anisotropic magnetostriction measurements in magnetic fields up to 12 T. Our
magnetostriction results can be explained by the existence of a large spin–orbit
coupling which, in combination with crystal-field effects, produces a single-
ion type magnetostrictive response. The Re orbital moment triggers a greatly
enhanced magnetocrystalline anisotropy compared to other ferromagnetic
double perovskites. From our magnetostriction data, the temperature
dependence of the coercive field as a function of Cr-doping is obtained. We
discovered that the coercive field increases as Fe is replaced with Cr, which is
linked to a strong enhancement of the magnetic anisotropy. This suggests a close
relationship between the Fe[Cr]–O–Re coupling and the magnetic anisotropy.
We also analysed the impact of the Re orbital moment on the spin-dependent
transport across Sr_2CrReO_6 grain boundaries. The present work opens up the
possible use of these compounds for magnetostrictive applications in a wide
temperature and magnetic field range.

(Some figures in this article are in colour only in the electronic version)

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1. Introduction

Double ordered perovskites (with chemical formula $A_2BB'O_6$) have attracted great interest in recent years because of their unique combination of highly spin polarized transport properties and high Curie temperature [1–4]. B and B' are transition metals in octahedral coordination with oxygen atoms forming an alternating perovskite structure. For the case of BB' = FeMo, FeRe, CrMo, CrW, CrRe and CrOs the compounds exhibit a double-exchange-like ferromagnetic interaction with fairly high Curie temperature, which is explained in terms of a B–O–B' indirect coupling. A recent review of the state-of-the-art in this field can be found in [5]. The picture of a kinetically driven ferromagnetism, proposed by Sarma *et al* [6] for FeMo compounds, is in good agreement with local spin density approximation (LSDA) and generalized gradient approximation (GGA) calculations of the electronic structure [1, 7–11], and explains the saturation magnetization [12, 13], the mixed valence $B^{2+/3+}/B'^{6+/5+}$ state and the large spin polarization at the Fermi level.

5d elements at the B' site (W, Re, Os) bring out new ingredients. Although the underlying physics of W- [10], Re- [7] and Os-based [14] double perovskites is essentially the same [11, 15], there are two main differences with respect to the Mo-based ones. These are the appearance of on-site electronic correlations and a spin–orbit (SO) coupling larger than in the lighter 4d counterparts. The enhanced SO coupling induces an unquenched orbital moment antiparallel to the Re spin moment [5, 16, 17]. Thus, the local charge distribution around the B' atom is not spherically symmetric, and the free energy might show absolute minima for certain orientations of the magnetic moment, basically depending on the surrounding crystal electric field. Correspondingly, the lattice might undergo deformations to accommodate a new direction of the magnetic moment, thus producing magnetostriction. This sort of magnetoelastic interaction is known as single-ion type [18].

The existence of giant volume magnetostriction (ω) and anisotropic magnetostriction (λ_t) can be considered as the fingerprint of an unquenched Re orbital moment. This is indeed the case in the A_2FeReO_6 series, where λ_t as large as $-1100\,\mu$st ($1\,\mu$st $= 10^6 \times \Delta L/L_0$) have been reported [19, 20], and it was related to the substantial orbital contribution to the Re magnetic moment measured by x-ray magnetic circular dicroism (XMCD) [21]. Subsequent experiments indicated that the unquenched orbital moment at the B'-site is ubiquitous in 5d-element based double perovskites [14, 22, 23]. In the Sr_2CrReO_6 compound, an orbital to spin Re magnetic moment ratio as large as 37% was found [22], in agreement with density-functional theory calculations of the electronic density of states [9]. Indeed, it has been shown very recently that the Re orbital magnetic moment in Sr_2CrReO_6 must be taken into account in order to fully explain the observed macroscopic saturation magnetization [24]. In this paper we report on the magnetostrictive behaviour of the series $Sr_2(Fe_{1-x}Cr_x)ReO_6$. This has allowed us to study the impact of Fe substitution by Cr on the magnetoelastic coupling. We also analyse the temperature dependence of the magnetic anisotropy stemming from the magnetocrystalline anisotropy brought about by the Re orbital moment [17, 16] and finally show its influence on the powder magnetoresistance of these compounds.

2. Experimental details

Samples were synthesized by the solid state reaction as reported elsewhere [25]. The same specimens were used in this work and in the neutron diffraction study reported in [25]. In brief, the $x = 0$ and 1 compounds are single-phase with refined antisite concentration of 0% and 10% respectively, whereas the intermediate compounds show secondary phases of the same structural type. The main phase is close to the nominal stoichiometry $Sr_2(Fe_{1-x}Cr_x)ReO_6$,

whereas the secondary phase (below 30% of the diffracted intensity) can always be indexed as Sr_2FeReO_6. The Curie temperature of the main phase increases linearly with the $Fe[Cr](t_{2g})$–$O(2p)$–$Re(t_{2g})$ transfer matrix elements up to 625 K for $x = 1$ [5, 25]. The magnetization at 5 T roughly follows the expected linear decrease with x from 3 μ_B/f.u. for $x = 0$ to 1 μ_B/f.u. for $x = 1$ within the ionic model [1, 13, 25] without accounting for the spin–orbit coupling. However, in Cr-rich compounds the saturation field can reach values up to 20 T [5, 24] and therefore a magnetic field of 5 T is not large enough to fully saturate magnetization.

In order to measure the magnetostriction and magnetoresistance, the samples were ground in a mortar and cold pressed. The result was a compact pellet which can be shaped into a parallelepiped. A resistive strain gauge was carefully glued on the sample surface and set onto one of the branches of an AC Wheatstone bridge. The sample deformation is then proportional to the bridge imbalance because the other branch is a strain gauge from the same batch but glued to a silica quartz, without magnetostrictive response. We measure the relative magnetic field-induced deformation of the polycrystals in the direction parallel ($\lambda_{\parallel}$) and perpendicular ($\lambda_{\perp}$) to the applied magnetic field. In this way we can account for the overall volume and shape change of the polycrystalline specimen, namely the volume magnetostriction ($\omega = \lambda_{\parallel} + 2\lambda_{\perp}$) and anisotropic magnetostriction ($\lambda_t = \lambda_{\parallel} - \lambda_{\perp}$) respectively. Magnetostriction experiments were performed applying a 12 T constant magnetic field produced by a superconducting coil. Magnetoresistance under high pulsed magnetic fields was measured within the four-probe geometry in AC mode by means of the lock-in technique. Data were acquired at the 60 T coil of the LNCMP (Toulouse, France). The field profile has a duration of 400 ms with rise time of 46 ms up to 42 T. The input impedance of the lock-in amplifier limits the temperature range for reliable measurements to $T > 150$ K. Magnetization measurements were performed in commercial *Quantum Design* SQUID equipment.

3. Results and discussion

Figure 1 displays the magnetic field dependence of $\lambda_{\parallel}$ and $\lambda_{\perp}$ for the end compounds. The magnetostriction loop shows hysteresis and reflects the magnetization process. As is the case of the intermediate compounds, $\lambda_{\parallel}$ is negative, whereas $\lambda_{\perp}$ is always positive. We point out that the magnetostriction at 12 T is still far from saturation, in contrast to the magnetostrictive behaviour previously reported in Ba_2FeReO_6 [5]. All these results can be interpreted as follows. The magnetization reversal process entails rotation of the Fe[Cr] localized spin moment, coupled antiparallel to the Re spin moment, which besides is coupled antiparallel to the Re orbital moment. Thus, the Re orbital moment tends to follow the direction of the external applied field, only constrained by the magnetocrystalline anisotropy and magnetic exchange energy. Therefore the lattice will deform in order to accommodate, for every magnetic configuration, the new charge distribution of the d electrons, which is not spherically symmetric if the orbital moment is nonzero. The ground state is achieved by minimizing the elastic energy in the presence of the electrostatic interaction with the crystal electric field created by the ReO_6 octahedra negative ligands. The magnetoelastic Hamiltonian describing such a complex process must be treated from a phenomenological point of view. From the spontaneous magnetostriction of the A_2FeReO_6 compounds around magnetic transitions [5, 20, 26, 27, 16], a general magnetoelastic coupling pattern can be inferred: the lattice experiences a shrinkage in the direction in which the magnetic moment is aligned (see [5] and references therein). As a consequence, we expect a negative $\lambda_{\parallel}$ and positive $\lambda_{\perp}$ [5, 19].

By combining $\lambda_{\parallel}$ and $\lambda_{\perp}$ one obtains an overall positive ω and negative λ_t. Figure 2 depicts the behaviour of ω and λ_t for Sr_2CrReO_6 while sweeping the field from 12 to -12 T in the range
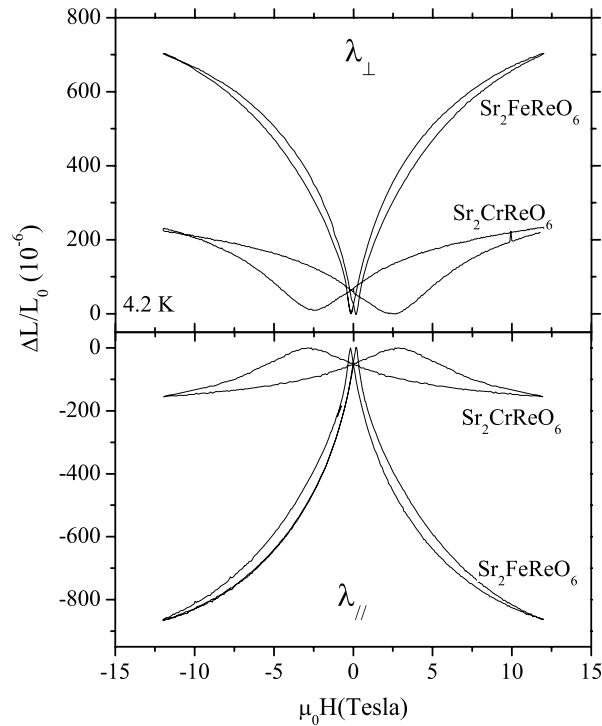
Figure 1. Perpendicular (top panel) and parallel (bottom panel) magnetostriction of Sr_2CrReO_6 and Sr_2FeReO_6 at 4.2 K.

4.2 K $< T <$ 300 K. The strong increase of the coercive field on cooling is clearly reflected in the leftwards shift of the zero magnetostriction state. The single-ion magnetoelastic term couples anisotropic lattice strain modes with the total magnetic moment via the orbital moment. Therefore, the giant λ_t observed reflects nicely the existence of a large orbital moment in the Re atom in both Sr_2FeReO_6 and Sr_2CrReO_6. Although the volume magnetostriction is usually associated with magnetoelectronic effects or exchange contributed magnetoelastic mechanisms, the observed volume magnetostriction can also be viewed as a pure single-ion magnetoelastic effect. This is supported by two facts. First, an exchange contributed magnetoelastic coupling can be ruled out, because the FeMo-based double perovskites show negligible magnetostriction as compared to the isostructural FeRe ones (see figure 35 in [5]) which have the same kind of double-exchange ferromagnetism. Second, since the crystal-field terms in the Hamiltonian also depend on the volume distortions (through the change in the interatomic distances when the deformations fulfils the crystal symmetry) [28], volume magnetostriction is naturally expected in these compounds bearing unquenched orbital moments, as was found in HoNi$_2$ [29].

The other compounds of $Sr_2(Fe_{1-x}Cr_x)ReO_6$ display the same features in the magnetostriction. Figure 3 shows the temperature dependence of λ_t measured at 12 T for the whole series. The maximum value of $\lambda_t = -1600$ μst is observed at 100 K for Sr_2FeReO_6. At a first glance, one can see that the lattice strain experienced by the Fe-rich compounds is much higher than that of the Cr-rich compounds. This is obvious at room temperature, where the coercive fields are much smaller than 12 T. The presence of a Sr_2FeReO_6 secondary phase in the intermediate compounds is expected to artificially increase the macroscopic value of the magnetostriction, but the systematic λ_t increase with the Cr
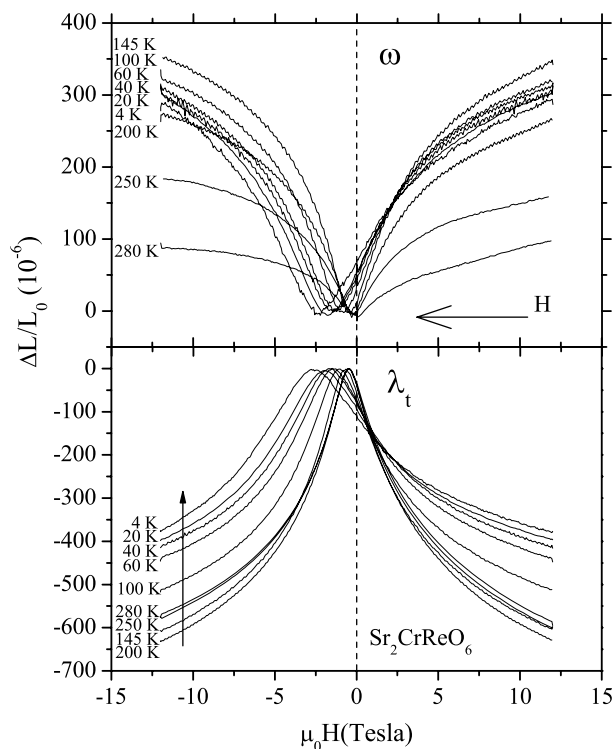
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Figure 2. Volume (top panel) and anisotropic (bottom panel) magnetostriction of Sr_2CrReO_6. The solid arrow marks the correspondence between the isotherms and the temperature label. The wide empty arrow gives the direction in which the magnetic field is swept.
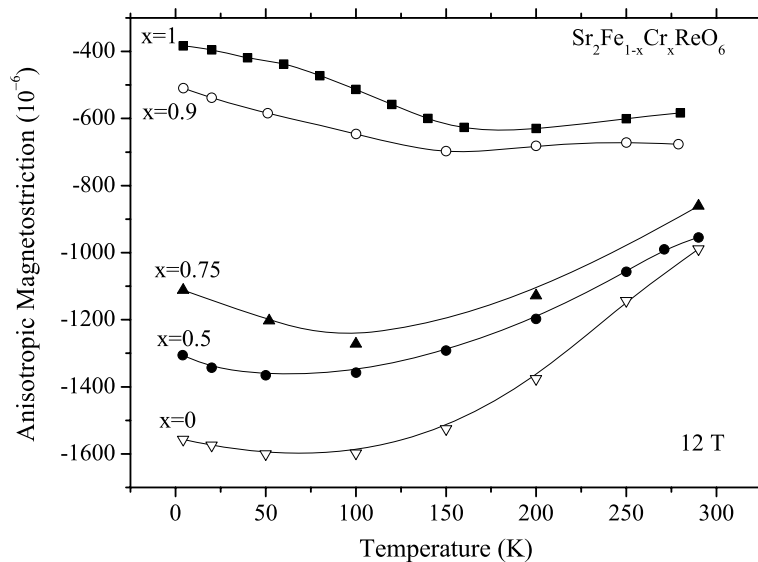

Figure 3. Temperature dependence of the anisotropic magnetostriction at 12 T for the $Sr_2(Fe_{1-x}Cr_x)ReO_6$ series.

concentration must be dominated by the main phase. Therefore, we can conclude that the decrease of the anisotropic lattice deformation as Fe is replaced with Cr is an intrinsic property of this series. This fact can be ascribed to the different extent of the tetragonal distortion when going from Sr_2FeReO_6 to Sr_2CrReO_6. Whereas Sr_2FeReO_6 already shows tetragonal structure at room temperature, Sr_2CrReO_6 is cubic at room temperature [25, 30]. The transition temperature from cubic to tetragonal decreases linearly with x, due to the better cation size matching of Cr in the cubic double perovskite cell [25]. Following this reasoning, one can also explain the marked increase of λ_t as the temperature is lowered. The increase of the tetragonal distortion on cooling, together with the depopulation of excited states with lower magnetization values, leads to the observed temperature dependence. Another explanation for the λ_t dependence on x could be found in the existence of substantially different values of the Re orbital moment and therefore of the spin–orbit coupling. However, this is very unlikely to occur, since the orbital to spin moment ratios reported for Sr_2FeReO_6 [21] and Sr_2CrReO_6 [22] are very similar. As a consequence, it is clear that the most important terms of the magnetoelastic energy must be those connecting deformations in expanded directions of the unit cell due to tetragonal distortions and magnetization components along these directions. Quantitative estimations of magnetoelastic constants could only be drawn from measurements in single crystals or fully epitaxial thin films. The correspondence between the magnetic easy axis and the contracted lattice parameters has been demonstrated in FeRe-based double perovskites [26, 27].

The abrupt upturn of λ_t in the $x = 1$ and 0.9 compounds and the slight change of curvature below 160 K in the $x < 0.9$ compounds (see figure 3) can be ascribed to the huge enhancement of the magnetocrystalline anisotropy at low temperatures. In figure 4 (top) a similar effect on the magnetization is shown. These measurements were performed on warming under 20 kOe after applying -50 kOe, so that the curve basically reflects the coercivity variation. One can see that below 160 K there is a steep decrease of the magnetization in the Cr-rich compounds, thus meaning that below this temperature the coercivity jumps to the order of tens of kOe, whereas above this temperature it is much smaller than 20 kOe. In the Fe-rich compounds ($x \leqslant 0.5$) magnetization versus temperature does not show any anomaly, which indicates that the coercivity only slightly increases as a result of domain wall dynamics. From our magnetostriction measurements we can extract the temperature dependence of the coercivity. Data are presented in figure 4 (bottom). We have chosen the data derived from $\lambda_\parallel$ because it is the strain measured along the magnetic field direction, which is the magnetization direction sensed in standard experiments. In addition, recent static field magnetization measurements up to 30 T show that partial hysteresis loops measured up to fields surpassing the domain wall movement range exhibit the same coercive field as the whole hysteresis loop, although the coherent domain rotation process was not saturated [31]. Therefore, we think that the coercivity values taken from $\lambda_\parallel$ hysteresis loops up to 12 T are a good measure of the actual coercive fields. Figure 4 (bottom) shows how the coercive fields determined from magnetization measurements correspond nicely to those derived from the magnetostriction. Furthermore, a recent report on the Sr_2CrReO_6 magnetization under pulsed fields confirms the same temperature dependence [24].

As shown in figure 4, at room temperature, the coercivity of the Cr-rich compounds is much larger than that of the Fe-rich ones. On cooling down, there is a magnetization drop in Cr-rich compounds that coincides with the onset of the rise in the low temperature coercivity. This is a strong indication that the anomalies in, for example, magnetization or magnetostriction, basically consisting of depressed responses at a constant magnetic field, are due to an abrupt increase of the magnetocrystalline anisotropy. The almost vanishing zero field χ_{ac} at 160 K in Sr_2CrReO_6 [5], as occurs in Ca-rich A_2FeReO_6 [16, 20], reinforces this argument. Our neutron
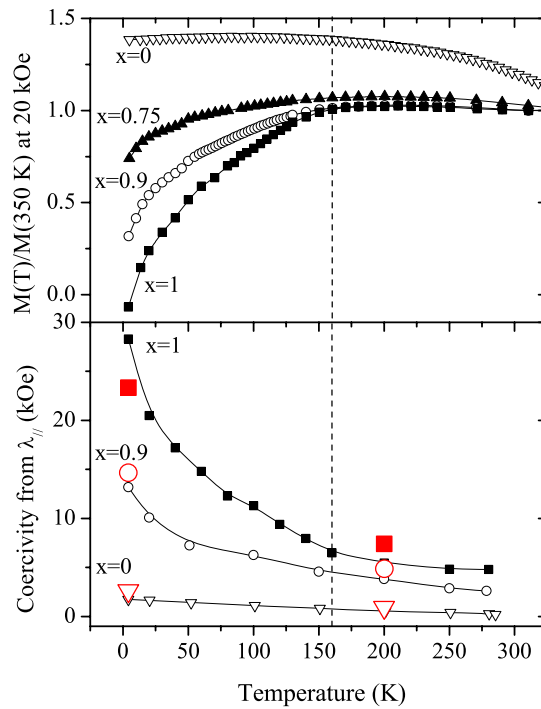
Figure 4. Top panel: magnetization warming curve under 20 kOe normalized at 350 K of $Sr_2(Fe_{1-x}Cr_x)ReO_6$. The measurement starts at 5 K after applying -50 kOe. Bottom panel: observed coercivity in $\lambda_\parallel$ as a function of the temperature. The dashed line is placed at 160 K, the temperature at which χ_{ac} drops on cooling [5]. Symbols refer to $x = 1$ (■), $x = 0.9$ (○), $x = 0.75$ (▲) and $x = 0$ (▽). The same legend applies for the large symbols, which correspond to coercivity values from magnetization loops. We do not plot the measurements for $0 < x < 0.9$ because they all nearly collapse in the same curve as for $x = 0$ and produce crowding of the data.

diffraction characterization of $Sr_2(Fe_{1-x}Cr_x)ReO_6$ [25] does not evidence a structural origin of this enhancement. It is not clear whether the change of the magnetocrystalline energy as a function of temperature is due to a structural transition beyond our experimental resolution or to the temperature dependence of the anisotropy constants due to some new electronic or magnetic ground state.

On the other hand, the Fe-rich compounds do not show any magnetic transition beyond the paramagnetic–ferromagnetic one, and all the compounds with $x \leqslant 0.75$ display coercive fields no larger than 3 kOe with smooth variations in the whole temperature range. Therefore, the Cr inclusion triggers the increase of the magnetocrystalline anisotropy at room temperature, which becomes especially important at low temperatures where the Cr-rich compounds evolve towards a much harder magnetic state. Since the Cr-rich compounds have ReO_6 octahedra closer to the perfect cubic symmetry [25], it is the optimization of the Fe[Cr]–O–Re coupling found in [5, 25] which is the only factor that could enhance the anisotropy on Cr-doping. The reason might be that when the B–O–Re hybridization increases, so does the coupling of the localized B spin moment with the crystal electric field at the Re site through the anisotropic $Re(t_{2g})$ charge distribution. Indeed, a highly enhanced electronic coupling between Re and B t_{2g}-states could even induce some orbital moment at the Cr-site, as is seemingly the case of the Fe atom in Ca_2FeReO_6, in which an orbital to spin moment ratio of 0.18(6) has been recently reported [32].
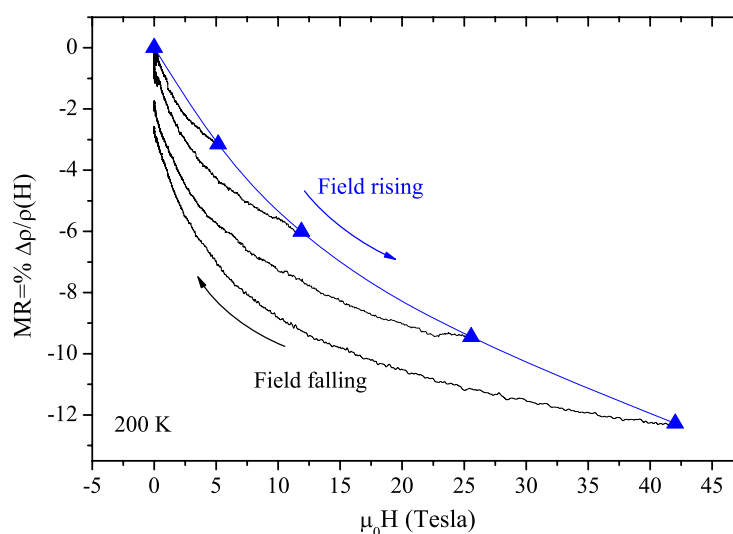
Figure 5. Pulsed field magnetoresistance measurements in Sr_2CrReO_6 at 200 K. From each shot of increasing field, we only record the initial resistance, the resistance at the maximum field (solid triangles) and the magnetoresistance during the field fall.

Sr_2CrReO_6 is an appealing compound for spin electronics because of its presumed large spin polarization [9] and high Curie temperature [25, 33]. Consequently, we also decided to study the magnetoresistance in Sr_2CrReO_6 polycrystalline specimens. Intergrain magnetoresistance (IMR) in metallic double perovskites arises from spin-dependent tunnelling across insulating grain boundaries [4, 34, 35]. Therefore, it is not surprising that the magnetic state of the first grain-surface layers plays a major role on the IMR, rather than the bulk magnetic state. The IMR effect on our Sr_2CrReO_6 sample at 200 K is shown in figure 5. Since the field pulse rise is too fast for reliable data acquisition, we only record the magnetoresistance during the field pulse fall. Several shots up to gradually increasing magnetic fields allow us to draw the IMR curve (triangles) during the field rise too. Between each field pulse we wait approximately 30 min in order to let the magnet cool down. The zero field resistance prior to the field shot is always 1740 Ω. However, the resistance after the field goes back to zero, namely the remanent resistance, decreases as the maximum achieved field increases. This indicates that there is a strong irreversibility of the magnetization in the vicinity of the grain boundaries, $m_{gb}(H)$, even after applying a magnetic field pulse of 25 T. This is obvious from the very different demagnetization paths as a function of the maximum applied field, which is evidence that the magnetization dynamics is slow enough to be observed in this kind of pulsed field measurements. Furthermore, the resistance relaxation from the remanent state to the initial zero field state suggests a spin glass behaviour of the magnetic moments at the grain boundary. The glassy magnetic nature of the outer grain shell can be understood in terms of a random anisotropy field arising from the large Re orbital moment. The randomness comes in because the defects (dislocations, lattice mismatch, oxygen vacancies, etc) define magnetic easy axes pointing in arbitrary directions, but correlated over a certain spatial scale due to the long range ferromagnetic interaction. Thus, the main difference between the IMR in Mo-based and Re-based double perovskites is again the presence of the Re orbital moment, which makes the model of weak random anisotropy [35] no longer valid, but produces a strong random anisotropy responsible for magnetic hardness of the IMR effect.

4. Conclusions

We have shown that Sr_2FeReO_6 and Sr_2CrReO_6 show giant anisotropic magnetostriction values just as other Re-based double perovskites do. This is a consequence of the unquenched Re orbital moment, and therefore must be a general property in any ferromagnetic double perovskite with 5d elements at the B$'$-site. From the systematic measurement in the $Sr_2(Fe_{1-x}Cr_x)ReO_6$ series, it is clear that the tetragonal distortion developed with decreasing x enhances the magnetoelastic coupling, thus strengthening the magnetostrictive response. On the other hand, as x increases from 0 to 1, the Fe substitution for Cr gives rise to a huge increase of the magnetocrystalline anisotropy. This can be understood in terms of an induced orbital moment at Fe/Cr-t_{2g} states owing to a larger hybridization with the Re states carrying a nonzero orbital moment.

The huge magnetocrystalline anisotropy impacts especially on the magnetic hardness of the grain boundaries, which results in modest IMR values even at 40 T. Then, our results seem to discard the use of Sr_2CrReO_6 as a spin polarizer for applications at low fields. In order to overcome these difficulties in real devices, surface engineering or grain size tailoring could provide stable remanent magnetization without the need for an applied magnetic field. Besides, the magnetostriction effect could be tuned for use as magnetostrictive sensors/actuators at certain temperatures and fields by means of varying the Cr content in $Sr_2(Fe_{1-x}Cr_x)ReO_6$. Multiferroic heterostructures or other devices in which some epitaxial strain gave rise to electric/magnetic responses could presumably harness the large magnetostriction values up to 1600 μst.

Acknowledgments

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