Anisotropy tuned magnetic order in pyrochlore iridates

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Attention of the condensed matter community was recently attracted by the iridates. Due to the interplay between a strong spin-orbit coupling, crystalline electric field (CEF) and moderate electronic interactions, the iridates are currently of interest. The magnetic behavior of polycrystalline samples of Er$_2$Ir$_2$O$_7$ and Tb$_2$Ir$_2$O$_7$ pyrochlores is studied by magnetization measurements and neutron diffraction. Both compounds undergo a magnetic transition at 140 and 130 K respectively, associated with an ordering of the Ir sublattice, signaled by thermomagnetic hysteresis. In Tb$_2$Ir$_2$O$_7$, we show that the Ir molecular field leads the Tb magnetic moment to order below 40 K in the all-in/all-out magnetic arrangement. No sign of magnetic long range order on the Er sublattice is evidenced in Er$_2$Ir$_2$O$_7$ down to 0.6 K where a spin freezing is detected. These contrasting behaviors result from the competition between the Ir molecular field and the different single-ion anisotropy of the rare-earths on which it is acting. Additionally, this strongly supports the all-in/all-out iridium magnetic order.

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bic space group, with the O occupying the 4f and 8b Wyckoff positions, the rare-earth and the Ir occupying the 16d and 16c positions respectively [23]. Polycrystalline samples of R$_2$Ir$_2$O$_7$, with R = Tb$^{3+}$ (4f$^8$, S=3, L=3, J=6, gJ=9 $\mu_B$) and Er$^{3+}$ (4f$^{11}$, S=3/2, L=6, J=15/2, gJ=9 $\mu_B$), were synthesized, by solid-state reaction starting from the binary oxides and by a new flux method using CsCl as flux, for neutron diffraction and magnetometry measurements respectively. The structure and quality of the samples were checked by X-ray diffraction. The lattice parameter and the $x$ coordinate of the 4f O were found at room temperature equal to $10.1606(2)$ Å and 0.334(2) for the Er compound and equal to $10.2378(5)$ Å and 0.35(2) for the Tb compound. A contamination by less than $\approx 2$-3% of Tb$_2$O$_3$ and Er$_2$O$_3$ parasitic phases was found in the Tb and Er samples used for the magnetometry measurements.

The temperature and field dependence of magnetization ($M$) were measured, for both compounds using Quantum Design VSM and MPMS® SQUID magnetometers down to 2 K, and down to 80 mK using a homemade SQUID magnetometer equipped with a dilution refrigerator [24]. Neutron diffraction experiments were performed on powders i) at the Institut Laue-Langevin on the D7 diffractometer at 2 K and 50 K for Er$_2$Ir$_2$O$_7$ and ii) at the ISIS facility for Tb$_2$Ir$_2$O$_7$ for which high-resolution data were collected on heating between 2 and 200 K, on the WISH diffractometer. Rietveld refinements for Tb$_2$Ir$_2$O$_7$ were carried out with the magnetic form factor of the Ir$^{4+}$ determined by Kobayashi et al. [25] using the FULLPROF program [26].

For both compounds, the ZFC-FC magnetization was measured in an applied field of 100 Oe (see Fig. 1). A separation between the ZFC and FC curves is observed at 140 and 130 K for Er$_2$Ir$_2$O$_7$ and Tb$_2$Ir$_2$O$_7$ respectively. This ZFC-FC behavior is consistent with previous results reported for this family showing that it coincides with the MIT [9, 27]. Below this bifurcation, although the shape of the FC magnetization depends on the sample preparation, a general behavior is observed, summarized as follows: for Er$_2$Ir$_2$O$_7$, below $T_M$, the FC magnetization remains above the ZFC one, both increasing down to 2 K without any sign of saturation. In Tb$_2$Ir$_2$O$_7$, the two curves increase with decreasing temperature, the FC one lying above the ZFC one. Then at lower temperature, there is a crossing of the ZFC and FC curves, the former increasing faster than the FC one. Finally, both FC and ZFC curves in Tb$_2$Ir$_2$O$_7$ present a bump around 6 K. The isothermal magnetizations as a function of magnetic field are shown for Er$_2$Ir$_2$O$_7$ and Tb$_2$Ir$_2$O$_7$ on Fig. 2. At the lowest temperature, a tendency towards saturation is observed in both compounds although not yet reached for the highest measured magnetic field of 80 kOe. For Tb$_2$Ir$_2$O$_7$, the magnetization additionally presents an inflection point, characteristic of a metamagnetic process at $\approx 18$ kOe, which is absent above 10 K.

**FIG. 1:** (Color online) $M/H$ vs $T$ measured in 100 Oe in a ZFC-FC procedure for Er$_2$Ir$_2$O$_7$ (a) and Tb$_2$Ir$_2$O$_7$ (b). The FC curves were measured while cooling. Inset: $M/H$ vs $T$ for Er$_2$Ir$_2$O$_7$ measured between 0.08 and 2 K after ZFC and FC in different magnetic fields, the magnetic field in the FC procedure being applied below 4 K.

**FIG. 2:** (Color online) $M$ vs $H$ for Er$_2$Ir$_2$O$_7$ (a) and Tb$_2$Ir$_2$O$_7$ (b) measured at different temperatures. Right side of (b): derivative of the magnetization curve for Tb$_2$Ir$_2$O$_7$ below 10 K showing a maximum indicative of a metamagnetic process.

Neutron powder diffraction was performed on Tb$_2$Ir$_2$O$_7$ and Er$_2$Ir$_2$O$_7$. The difference between the diffractograms of Tb$_2$Ir$_2$O$_7$ at 200 K and at 10 K (above and below $T_M$) reveals additional Bragg peaks...
indexable with the propagation vector $\mathbf{k} = (0, 0, 0)$ (see Fig. 3). From group theory and representation analysis [28], for the Ir $16c$ site and the rare-earth $16d$ site, the representation of the magnetic structure involves 4 irreducible representations (IR) among 10: $\Gamma = GM^+_7 + GM^+_3 + GM^+_5 + 2GM^+_4$ (notation of Miller-Love) [29], corresponding to the possible magnetic structures compatible with the $Fd\bar{3}m$ group symmetry. The Rietveld refinement of the neutron data shows that the AIAO magnetic configuration ($GM^+_7$ IR - shown in Fig. 4a) is the only one accounting correctly for the Tb magnetic ordering below 40 K and down to 2 K. The refined Tb$^{3+}$ magnetic moment at 10 K is $M(Tb) = 4.9 \pm 1 \mu_B$. The very weak magnetic moment at the Ir$^{4+}$ site could not be refined, being too small for the experimental sensitivity, i.e. lower than 0.2 $\mu_B$/Ir.

The Tb$^{3+}$ ordered magnetic moment, proportional to the square root of the intensity of the Tb$_2$Ir$_2$O$_7$ magnetic Bragg peaks, starts to increase significantly below $\approx 40$ K (see inset of Fig. 3). Its temperature dependence down to 2 K does not follow a Brillouin function, as also reported for the Nd$^{3+}$ magnetic moment in Nd$_2$Ir$_2$O$_7$ [16]. Its variation rather indicates that it is induced, through an effective Tb-Ir magnetic coupling, by the Ir molecular field, $\lambda M_{Ir}$. To check this, we calculated the Tb$^{3+}$ induced magnetic moment by assuming a Brillouin function for the Ir$^{4+}$ magnetic moment temperature dependence and considering the following CEF model Hamiltonian:

$$
\mathcal{H}_{CEF} = B_2^0 O_2^0 + B_4^0 O_4^0 + B_6^0 O_6^0 + B_8^0 O_8^0 + B_0^6 O_6^6
$$

where $O_n^m$ are Stevens’ operators and $B_n^m$ are adjustable Stevens’ parameters calculated from inelastic neutron scattering data for Nd$_2$Ir$_2$O$_7$ [30]. Assuming that the environment is exactly the same as in Nd$_2$Ir$_2$O$_7$, we took for Tb$_2$Ir$_2$O$_7$ the Stevens’ parameters

$$
B_n^m(Tb) = \frac{B_n^m(Nd)}{\Theta_J(Nd)} \Theta_J(Tb) < r^n >_{Nd} \Theta_J(Tb) < r^n >_{Tb}
$$

where $\Theta_J = \alpha, \beta, \gamma$ stands respectively for the Stevens reduced matrix elements associated with $O_n^m$, $O_n^0$ and $O_n^m$ and where $< r^n >$ are radial integrals. The Tb-Ir interaction is taken into account adding a term in the Hamiltonian $\mathcal{H}_{Tb-Ir} = \lambda M_{Ir}(T) g_{J} \mu_B \vec{J}$. The Tb$^{3+}$ magnetic moment is then computed as $M_{Tb} = g_{J} \mu_B \text{Tr}(\vec{J} \exp(-\beta H))$, where $H = \mathcal{H}_{CEF} + \mathcal{H}_{Tb-Ir}$. This model accounts well for the observed slow increase of $M_{Tb}$ below $T_{MI}$ which accelerates on lowering the temperature without any sign of saturation (see Inset Fig. 3). It allows to extract a value for the Ir$^{4+}$ molecular field $\lambda M_{Ir}$, found $\approx 33$ kOe at 10 K.

This is very different from the Er$_2$Ir$_2$O$_7$ case, where no additional Bragg peak was observed down to 2 K in neutron powder diffraction (data not shown) indicating the absence of long-range magnetic ordering of the Er$^{3+}$ sublattice. Additional ZFC-FC magnetization measurements in various magnetic fields were performed down to 80 mK, in which the FC procedure started around 4 K (see inset of Fig. 1). This allows to evidence thermomagnetic irreversibilities occurring below 4 K, hence not related to the Ir$^{4+}$ ordering. A ZFC-FC difference, most probably associated to some freezing of the Er$^{3+}$ magnetic moments, is observed around 600 mK.

The contrasting magnetic behavior of Er$_2$Ir$_2$O$_7$ and Tb$_2$Ir$_2$O$_7$ can be understood by considering the difference of magnetocrystalline anisotropy between the two rare-earth ions. In the pyrochlore family, the O$^{2-}$ environment of the rare-earth is a distorted cube, constituted of puckered six-membered ring and two apical oxygens, which provides to this site a very pronounced axial symmetry along the $<111>$ direction [23]. The sign of the $l=2$ main Stevens parameter, $B_2^0$, changes from negative for Tb$^{3+}$ to positive for Er$^{3+}$ ions, conferring to the former an axial anisotropy along the $<111>$ direction and to the latter a perpendicular easy-plane anisotropy. In the stannates and titanates, the magnetic behavior of the Tb and Er members is indeed in agreement with the easy-axis and easy-plane anisotropy respectively, hence with the sign of their $B_2^0$ term determined from neutron scattering [18, 22, 31–33].

Assuming that the transition at $T_{MI}$ is second order as suggested by the absence of hysteresis in macroscopic measurements [34], if the two Ir and rare-earth sublattices are coupled, they must order with the same IR. Our neutron diffraction results indicates that the Tb$^{3+}$ sublattice orders in the AIAO magnetic structure univ-
cally given by the $\text{GM}^+_2\text{Ir}$. Moreover, as shown above, the temperature dependence of the integrated intensities supports an induced magnetic ordering, the $\text{Tb}^{3+}$ moments being polarized by the molecular field of the $\text{Ir}^{4+}$ sublattice in the AIAO arrangement. Figure 4b shows the net molecular field along the local $<111>$ direction produced by the six first-neighbor $\text{Ir}^{4+}$ of each $\text{Tb}$ site, assuming isotropic $\text{Tb}$-$\text{Ir}$ exchange terms which are thus sufficient to induce the ordering of the $\text{Tb}$ magnetic moments. On the contrary, the AIAO magnetic order of the $\text{Ir}$ sublattice is incompatible with the easy-plane of anisotropy (magnetic configurations spanned by the $\text{GM}^+_3$ or $\text{GM}^+_5\text{Ir}$). Indeed, neither isotropic nor anti-symmetric exchange terms between $\text{Ir}$ and first neighbor $\text{Er}$ can induce magnetic ordering, the latter producing terms in the Hamiltonian that cancel out when summed over all $\text{Ir}$ neighbors on an hexagonal plaquette. For this reason, no induced magnetic moment is observed below $T_{\text{MI}}$ on the $\text{Er}$ sublattice.

In addition to the $\text{Ir}$-rare-earth coupling, magnetic interactions between the rare-earth moments are expected to occur at lower temperature. The signature of these interactions could result in the features observed in the temperature and field dependence of the $\text{Er}_2\text{Ir}_2\text{O}_7$ and $\text{Tb}_2\text{Ir}_2\text{O}_7$ magnetization below $T_{\text{MI}}$. In the Er$^{3+}$ case, this could be responsible for the onset of the freezing observed around 0.6 K, while in the $\text{Tb}^{3+}$ case it could explain the bump in the temperature dependence of the magnetization and the concomitant presence of a metamagnetic process below 6 K. The $\text{Tb}^{3+}$ sublattice could then first be polarized in the $\text{Ir}^{4+}$ molecular field yielding the AIAO arrangement, before it would feel, at low temperature, its own molecular field that has become dominant due to the large difference in the $\text{Ir}^{4+}$ and $\text{Tb}^{3+}$ ordered moments. This would lead to the same magnetic order (compatible with $\text{Tb}$ - $\text{Tb}$ antiferromagnetic interactions and easy-axis anisotropy), since no change is observed in the magnetic order by neutron scattering below 6 K.

Finally, we come back to the bifurcation observed between the ZFC and FC magnetizations starting at $T_{\text{MI}}$, which remains puzzling for a perfect AIAO antiferromagnetic arrangement. The same behavior is visible in another AIAO pyrochlore compound, $\text{Cd}_2\text{Os}_2\text{O}_7$ [35–37]. The most probable explanation of this FC-ZFC characteristic behavior invokes intrinsic and/or extrinsic defects, present in these pyrochlore systems: an off-stoichiometry can lead to the excess rare earth/Ir ions occupying the site of the counterpart ion, or to a $\text{Ir}^{4+}$/$\text{Ir}^{4+}$ substitution leading to a non magnetic site. The first type of defect has been shown to decrease the FC-ZFC difference [34], whereas the second type increases this difference [38]. Another source of intrinsic defects comes from the presence of magnetic domains at 180° [11, 39]. The magnetic moments located at the domain wall boundary have been shown to be free with respect to the nearest neighbor exchange coupling in another antiferromagnet with strong multi axial anisotropy [40, 41]. In the pyrochloro iridates, the rare-earth ions feel the molecular field associated to the defective $\text{Ir}^{4+}$ neighborhood, and then be also polarized along the applied magnetic field. The molecular field of defective hexagons has a component along and perpendicular to the $<111>$ directions, allowing the coupling of the $\text{Ir}^{4+}$ with the $\text{Er}^{3+}$ as well as the $\text{Tb}^{3+}$ ions (see Fig. 4cd).

In conclusion, our study of two members of the iridate pyrochlores gives a unified picture of the magnetic behavior of these materials, highlighting the strong coupling between the rare-earth and the Ir atoms. This coupling in $\text{Tb}_2\text{Ir}_2\text{O}_7$ allows us to establish the AIAO magnetic order of the $\text{Ir}^{4+}$ sublattice, which is thus expected to be a common feature of the family. The magnetic order of the rare-earth sublattice strongly depends on its magnetocrystalline anisotropy and on its compatibility with the $\text{Ir}^{4+}$ magnetic order. Beyond the interest in these systems for their potential topological non triv-
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