

Complete magnetization compensation in the intrinsic ferromagnetic semiconductor $\text{Gd}_x\text{Sm}_{1-x}\text{N}$ J. D. Miller¹, H. J. Trodahl², M. Al Khalfioui³, S. Vézian³, and B. J. Ruck^{2,4,*}¹*Paihau-Robinson Research Institute, Victoria University of Wellington, P.O. Box 33436, Petone 5046, New Zealand*²*School of Chemical and Physical Sciences, Victoria University of Wellington, P.O. Box 600, Wellington 6140, New Zealand*³*Université Côte d'Azur, CNRS-CRHEA, Valbonne 06560, France*⁴*MacDiarmid Institute for Advanced Materials and Nanotechnology, P.O. Box 600, Wellington 6140, New Zealand*

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We present a ferromagnetic material with full spin-polarization, but with zero net moment (a magnetic moment compensation point). The compensation of the magnetic moment stems from the combination of spin-dominated and orbital-dominated lanthanide ions in solid-solutions of the rare-earth mononitrides and is robust over a range of temperatures but limited to a critical composition.

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The goals of providing greater data storage, faster operation, and more energy-efficient computers (in particular in low-temperature applications) have driven materials researchers to look more and more widely for material properties that can be leveraged toward these objectives. This has led to a proliferation of materials research within the spin- and orbit-tronics communities in all kinds of magnetic materials, including antiferromagnets [1–3] and ferrimagnets [4,5]. One of the oft-cited benefits of these materials is the absence of a fringe field associated with the ordering, either due to the rigid anti-alignment conditions imposed in antiferromagnets or to the compensation of the magnetic moments that can be achieved in ferrimagnetics [6]. Here we report a remarkable magnetic compensation achieved by engineering an opposing orbital magnetic moment in a fully spin-aligned state.

Magnetism in solids is all but universally associated with the alignment of electrons' spin magnetic moments. Indeed, the exchange interaction central to the magnetic ordering of a material acts only on the spins of participating atoms. The orbital states of most ions also carry magnetic moments, but these are discouraged from participating in bulk magnetism by their participation in crystal binding (forming full electron shells with $L = 0$) and the resulting crystal field at the ion site ("quenching" the orbital angular momentum). This prioritization of the spin state leads to a natural magnetic-material classification as ferromagnetic (fully aligned spins), antiferromagnetic (anti-aligned spins of identical magnitude and zero net moment), or ferrimagnetic (anti-aligned spins of dissimilar magnitude and non-zero moment), and within this scenario the macroscopic magnetization follows naturally

from the spin alignment; it is simply the net electron spin magnetic moment imbalance.

The spin-only picture fails among compounds of the heaviest stable ions, the lanthanides and actinides. In these, the strong spin-orbit interaction couples orbital magnetic moments firmly to the spin moments and leads to fully spin-aligned states with a net moment that is the sum of both spin and orbital contributions; the net moment no longer resides only in the spins. That net magnetization can lie parallel or antiparallel to the spin moment or even have spin/orbit cancellation that shows zero net magnetic moment in the fully spin-aligned state. Exactly this has been reported in $\text{Sm}_{1-x}\text{Gd}_x\text{Al}_2$, a ferromagnetic metal that shows zero magnetization for $x = 0.05$ [7–10]. The magnetic compensation depends strongly on both the composition x and the temperature [11], and the complete magnetization compensation exists only at a single temperature for a given doping.

The present paper reports studies of another Sm/Gd compound, $\text{Gd}_x\text{Sm}_{1-x}\text{N}$, which forms a dopable ferromagnetic semiconductor with full spin polarization in the conduction band [12]. Rare-earth nitride solid solutions crystallize in the same rock salt structure as the parent materials [13]; in our case, co-deposition of Gd and Sm metals in an environment of molecular nitrogen leads to a stoichiometric material with the cation sites randomly occupied by Gd^{3+} and Sm^{3+} [13]. This yields a homogeneous material that orders ferromagnetically below a Curie temperature that lies between the extremes of 30 K (SmN) and 70 K (GdN) [13–15].

Typically for these materials—the rare-earth nitrides (RN with R a lanthanide)—the insulating nature of the stoichiometric nitride ensures that the magnetic moment lies overwhelmingly in the strongly localized lanthanide $4f$ states [16]. The potential to form a compensated ferromagnetic material follows from the contrasting magnetic moment in those $4f$ states on the cations; Gd^{3+} , in the middle of the lanthanide series, has purely spin magnetism, while an isolated Sm^{3+} has opposing orbital and spin alignments with a net magnetic moment of $\sim 0.8 \mu_B$. Within the SmN context, that moment is reduced to $\sim 0.4 \mu_B$ by the crystal field, and reduced further

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to $\approx 0.035 \mu_B$ by the exchange interaction in the ferromagnetic state [17]. Crucially, that very small magnetic moment is orbital-dominated; the net magnetic moment is aligned in opposition to the spin contribution [17–20].

In $\text{Gd}_x\text{Sm}_{1-x}\text{N}$, the strong Gd-Sm exchange interaction, aligning the Sm^{3+} and Gd^{3+} spins, ensures that the net magnetic moments on the two cations are opposed [13]. The continuous development of the net magnetic moment of $\text{Gd}_x\text{Sm}_{1-x}\text{N}$ as x is varied between the orbital-dominated and spin-only magnetization of the parent materials constrains the net magnetization to pass through zero at some particular value of $x = x_c$. Within the limit that the magnetic moments on the two cations are independent of composition, the magnetic moment of a solid solution is taken as the weighted average of the magnetizations of the constituents [21]. From this and the near-zero moment of SmN , one expects a volume-averaged magnetization dominated by the Gd^{3+} ions and proportional to the composition parameter x :

$$\bar{M} = 7x - 0.035(1 - x) \mu_B/\text{cation}. \quad (1)$$

This simple treatment suggests the compensation point of the net magnetic moment of $\text{Gd}_x\text{Sm}_{1-x}\text{N}$ at $x_c \approx 0.5\%$.

This compensation of the magnetic moment is common within transition metal/rare-earth alloys (e.g., CoGd [22–24], CoTb [25], and CoFeGd [26–28]), Heusler alloys (Mn_2GaRu_x [29]), and the rare-earth iron garnets [30]. All of these exhibit ferrimagnetic ordering of the magnetic cations and compensation of the magnetic moments by tuning the temperature, strain, or chemical composition (as here). However, it is crucial to the compensation in this case that alignment of the spins in $\text{Gd}_x\text{Sm}_{1-x}\text{N}$ is **ferromagnetic**, as it is this ferromagnetic exchange coupled with the orbital-dominant behavior of the Sm^{3+} that drives the material towards a vanishing magnetization state.

Identifying the magnetic compensation point x_c with magnetization measurements is fraught; a material with a net zero magnetic moment is hard to identify using magnetometry. However, the strong f/d exchange interaction in the lanthanides [31] ensures spin-selected transport at n-type doping up to 0.01 electrons per formula unit [32]. Thus, we have turned to the anomalous Hall effect (AHE), which follows directly the spin imbalance ($n_\uparrow - n_\downarrow$) in the conduction channel. The AHE sign is positive or negative depending on whether the $4f$ (and thus the $5d$ conduction-channel) spins are aligned parallel or antiparallel to the net magnetization, leading to opposite signs of the AHE in GdN and SmN [20,32]. This dependence can be exploited to locate the composition at which the magnetization crosses over from being dominated by the Sm^{3+} moments (giving a positive AHE) to being dominated by the Gd^{3+} moments (giving a negative AHE).

The films used in this study were grown at the Centre de Recherche sur L'Hetero-Epitaxie et ses Applications (CRHEA) in France in a Riber ultrahigh vacuum chamber with a base pressure of 10^{-10} torr. High-purity rare-earth metals were evaporated at rates controlled to achieve the desired alloy composition and grown to a thickness of ~ 100 nm in an atmosphere of N_2 . Films were grown on substrates of $\text{Si}(111)$ buffered with 100 nm of AlN . Growing cubic crystals on a hexagonal net, as here, leads to two rotational variants of the crystal [14,33,34], but films can also be epitaxially grown

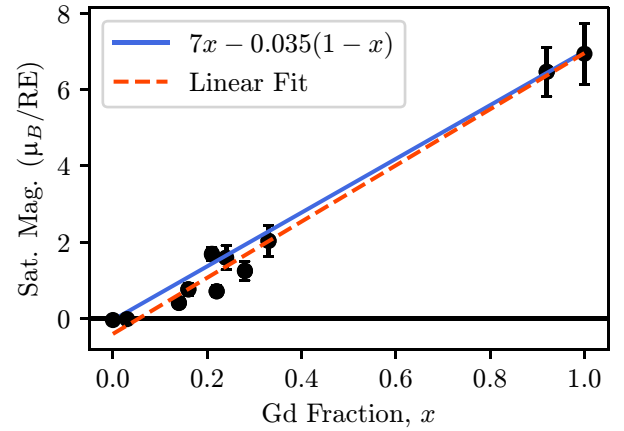


FIG. 1. Saturated magnetic moments of $\text{Gd}_x\text{Sm}_{1-x}\text{N}$ measured at 5 K. The blue trace follows $7x - 0.035(1 - x)$, as in (1), suggesting a magnetic compensation point at 0.5% Gd incorporation. The dashed red trace is a linear fit to the experimental data, which points to a magnetic compensation point closer to $\sim 4\%$. Data points at $x = 0$ and $x = 1$ are averages over multiple samples.

without this effect on Lanthanum Aluminate (LAO) [35,36]. The growth of the films was undertaken at temperatures above $\sim 450^\circ\text{C}$ to ensure good crystal quality. The variation of the growth temperature and partial pressures of nitrogen ($10^{-5} - 10^{-4}$ mbar) across the range of compositions gave a range of nitrogen vacancy concentrations (and thus conductivities). After growth, the films were capped with a 100 nm layer of GaN to prevent oxidation, to which the rare-earth nitrides are known to be prone.

Magnetometry measurements were performed in a Quantum Design Magnetic Property Measurements System (MPMS) capable of temperatures as low as 5 Kelvin and fields up to 7 Tesla. Electrical transport measurements were performed in a Quantum Design Physical Property Measurements System (PPMS) capable of temperatures as low as 2 Kelvin and fields up to 9 Tesla. Resistivity and Hall effect measurements were performed in the Van der Pauw geometry. In these films, special care is required to separate the three components to the Hall measurement: parasitic resistivity, the ordinary Hall effect, and anomalous Hall effect. These methods are described in Ref. [20]. Composition, structural, and magnetic experimental data are included in the Supplemental Material Ref. [37].

Magnetometry measurements covering the full range of compositions are shown in Fig. 1. The near-zero moment of Sm^{3+} [18] ensures that the volume-averaged magnetization in these solid solutions is dominated by the Gd^{3+} ions and, to the first order, proportional to the composition parameter x . However, fitting a linear model to the data across the series suggests a greater Sm^{3+} moment than that given by Eq. (1).

A similar treatment of the $4f$ angular momenta to that of the magnetic moment in Eq. (1) gives a cation-averaged angular momentum:

$$\bar{J} = 3.5x - 1(1 - x)\hbar/\text{cation}$$

placing the angular momentum critical point of $\text{Gd}_x\text{Sm}_{1-x}\text{N}$ at $x = 0.222$. However, x-ray magnetic circular dichroism

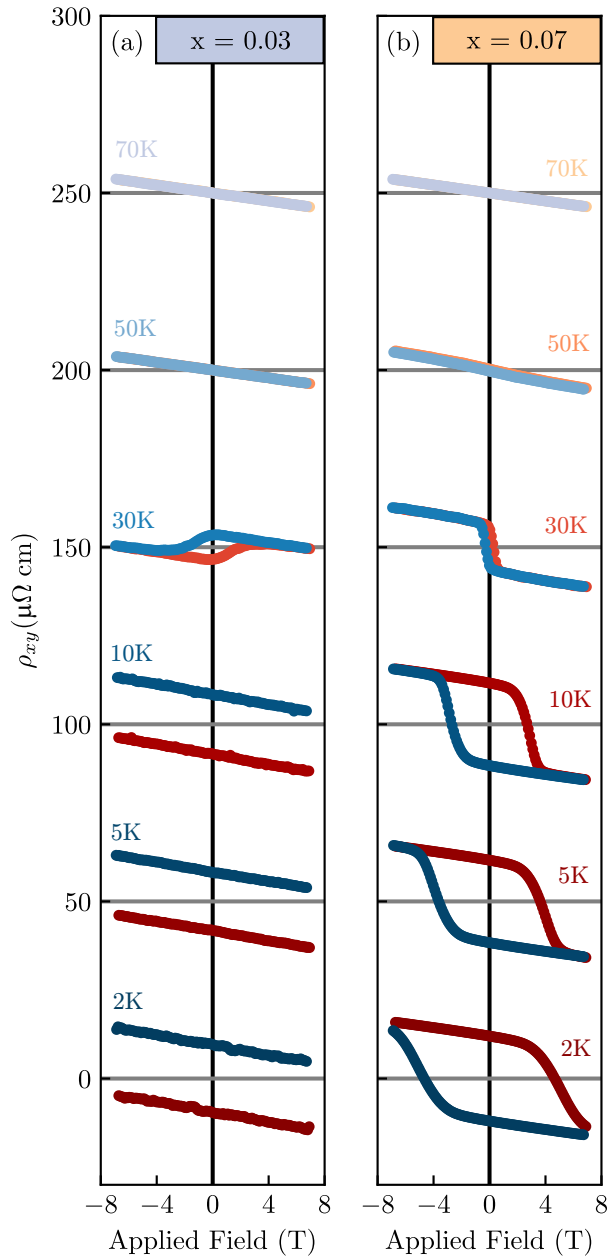


FIG. 2. Hall effect measurements for a $\text{Gd}_x\text{Sm}_{1-x}\text{N}$ films with (a) $x = 0.03$ and (b) $x = 0.07$ taken at 2, 5, 10, 30, 50, and 70 K. Each dataset is artificially offset by $50 \mu\Omega \text{ cm}$, and the color is used to indicate both sweep direction and temperature.

(XMCD) for both $\text{Gd}_x\text{Sm}_{1-x}\text{N}$ solid solutions [13] and GdN/SmN multilayers [17,38] shows a strong influence of neighboring Gd ions, enhancing the magnetic moment of SmN. This enhancement shifts the angular momentum compensation point modestly from a first-order approximation, and it lies close to $x \approx 0.24$ [13]. We show below that the magnetization compensation point is also shifted from the first-order expectation of magnetization compensation at $x \approx 0.005$ by this enhancement.

Figure 2 shows the Hall effect measured at temperatures ranging from 70 K, above the Curie temperature, down to 2 K,

deep in the ferromagnetic phase, for two films differentiated by the Gd fraction $x = 0.03, 0.07$. For low temperatures, a simple sweep up and down between 7 and -7 T is clearly insufficient to reverse the magnetization. To obtain a more accurate characterization of the full hysteresis loop, the blue data were taken, sweeping the field from positive to negative. From here, the samples were heated to 30 K and then cooled in -7 T to reverse the magnetization direction. The orange/red were then taken, sweeping the field from negative to positive. Care must be taken to separate the longitudinal and transverse components of the Hall voltage. This process is described in [20].

The ordinary Hall effect, which is linear at the highest fields, is negative, confirming the usual n-type doping that is found throughout the entire RN series and understood as due to nitrogen vacancies (V_N). The AHE magnitude of the two films is closer to that of SmN [20], a factor of more than 5 larger than seen in GdN [32], which relates, respectively, to their $4f$ vs $5d$ conduction channels [16,39]. The coercive field is also closer to that of SmN, largely as a result of the weak coupling between the small magnetic moment and the magnetic field. The picture that emerges is of a material with the inter-ion interactions—the energetics that determine the ferromagnetic response—related to the majority SmN forming the solid solution, with only the stronger Gd^{3+} coupling to a magnetic field determining the universal $4f$ spin alignment. For the film in Fig. 2(b), the sign of the AHE clearly indicates that the spin alignment of the conduction band follows that of GdN. The net magnetization lies still in the 7% of Gd cations; clearly, one must seek magnetization compensation at $x < 0.07$.

A clear contrast is found in the AHE of a 3% Gd film in Figure 2(a). At 30 K, the hysteresis loop of the AHE opens up with a clearly positive sign; the $5d$ conduction band is now occupied by electrons whose spin magnetic moment is aligned antiparallel to the applied field, as in SmN. The 5 K data show the same alignment, now with a coercive field that is larger than the maximum 7 T available in the measurements. As discussed previously, these measurements require that the net magnetization is prepared in the fully positive and negative magnetized state by lowering the temperature from above the Curie temperature in the maximum available field. The implication, however, is clear: the cation spin magnetic moments now lie *antiparallel* to the net magnetization. The point at which the spin moments reverse lies in the range $0.03 < x_c < 0.07$, at $5 \pm 2\%$ Gd. This shift of the compensation point to $\sim 10\times$ the value given by Eq. (1) is consistent with the enhancement of the Sm^{3+} moment that appears in XMCD measurements [13] and is implicit in the fit shown in Fig. 1.

The AHE here offers strong evidence corroborating the existence of an ordered magnetic state. The coercive field of SmN at 10 K is typically between 2 and 3 Tesla [20]. Here, there is no indication that the AHE has begun to turn over even up to 7 T, let alone reached the coercive field where the magnetization is likely to have reversed. These results strongly indicate an ordered magnetic state with a minuscule moment, even smaller than that of SmN ($0.035 \mu_B$ per ion), robust up to 10 K, and not relying on a single compensation

temperature. This is a strong sign of being in the neighborhood of the magnetic compensation point.

An orbital-dominated magnetic moment is a characteristic found among the majority of light lanthanide ions. This is thus not the only $(R, R')\text{N}$ compound that shows promise for achieving a compensation like the one exhibited here. As the mechanism for compensation relies on the ferromagnetic alignment of the orbital-dominant/spin-dominant combination of cations, one expects similar zero-magnetization facility to occur with most light-lanthanide/heavy-lanthanide solid solutions within the nitride series. Moreover, the combination of zero-magnetization with the extended $4f$ bands that form at the Fermi level in doped rare-earth nitrides [39] makes these materials a possible host for unconventional, heavy fermion-type superconductivity, as in SmN [40].

In summary, we have presented evidence for magnetic compensation occurring within solid solutions of the form $\text{Gd}_x\text{Sm}_{1-x}\text{N}$. Within these solid solutions, the magnetization

compensation occurs at a point where the dominant magnetization changes from spin- to orbital-dominated, accompanied by a reversal of the spin magnetic moment relative to an applied magnetic field. The anomalous Hall effect sign reverses with the spin, providing an unambiguous signal of magnetic compensation. This material is not the first to display magnetic compensation alongside ferromagnetic alignment [7] but is the first to be electrically dopable and to observe the effects of compensation over a wide temperature range.

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