Novel behaviour in admixed Rare Earth systems: Exchange Bias, repeated magnetic compensation and self-compensation

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Plan

Highlights of ongoing work at TIFR on ‘zero magnetization’ systems

1. Brief recap. on the characteristics of magnetic Rare Earth (RE) elements, with special focus on Samarium.


3. Contemporary interest in alloys at ‘zero-magnetization’ stoichiometry.

4. Elucidation of notion of the Exchange Bias field in compensated admixed RE based magnets, along with other interesting features.

5. Identification of self-compensation in pristine Sm magnets via fingerprint of an Exchange Bias.


7. Discovery of novel Repeated Magnetic Compensation behavior in the admixed rare earth inter-metallics.
### Magnetic Rare Earth Elements: 58 - 70

The image shows a portion of the periodic table with a focus on elements in the 3d and 4f blocks. The highlighted elements are magnetic rare earths, specifically those in the range of atomic numbers 58 to 70.

- The 3d block includes elements like Sc, Ti, V, Cr, Mn, Fe, Co, Ni, and Zn.
- The 4f block includes elements like Ln, Tb, Dy, Ho, Er, Tm, Yb, and Lu.

These elements are important in various applications due to their magnetic and rare earth properties.
The ground state properties of the magnetic rare earth ions

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<th>$m_L$</th>
<th>+3</th>
<th>+2</th>
<th>+1</th>
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<th>-1</th>
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<th>-3</th>
<th>S</th>
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<th>$g_J$</th>
<th>$g_J J_z$</th>
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<td>$\frac{1}{2}$</td>
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Spin–Orbit coupling prevails and the ground state is given by:

$J = L + S$, for more than half filled

$J = L - S$, for less than half filled.

$\langle L_z \rangle = J_z (g_J - 2)$,

$\langle S_z \rangle = J_z (1 - g_J)$

$\mu_z = - \mu_B (\langle L_z \rangle + 2 \langle S_z \rangle)$

$\mu_J$ - orbital angular momentum,

$S$ - spin angular momentum and

$J$ - total angular momentum
Rare Earths Elements: ➢ The 4f- electrons give rise to magnetic moment.

\[
\langle L_z \rangle = J_z (g_J - 2),
\]

\[
\langle S_z \rangle = J_z (1 - g_J)
\]

\[
\langle \mu_z \rangle = g_J J_z
\]

For free \( \text{Sm}^{3+} \):

\[
\langle L_z \rangle = -30/7, \quad \langle S_z \rangle = 25/14,
\]

\[
\mu_z (\text{Sm}^{3+}) = \frac{5}{7} \mu_B
\]
The RKKY Interaction:

*Indirect Exchange*: mediated by local s-f interactions by conduction electrons.

\[ H = -J_{sf} S.s \]

S is local rare earth spin  
s is conduction electron spin.

\( J_{sf} \) is exchange coupling constant.

\[ H_{\text{eff}} \text{ (two rare earth ions)} = -j_{ff} S_1.S_2 \]

\[ j_{ff} \propto -J_{sf}^2 \sum F(x) \]

\[ F(x) = \frac{x \cos x - \sin x}{x^4} \]

\[ x = k_F r \]

Variation of coupling constant \( j_{ff} \), with distance \( (r) \) from rare earth ion. It is oscillatory and damped in space.
PrAl$_2$: $T_c \sim 35$ K
$\mu$/f.u. $\sim 2.8$ $\mu_B$
GdAl$_2$: $T_c \sim 170$ K
$\mu$/f.u. $\sim 7.7$ $\mu_B$

- Zero Magnetization stoichiometry realized at $x \sim 0.3$

*Pseudo-antiferromagnetism*

- Magnetic compensation phenomenon at $x \sim 0.2$  $T_c \sim 80$ K, $T_{\text{comp}} \sim 40$ K
- Magnetic correlation effects present even above nominal $T_c$ ($\sim 40$ K) at $x \approx 0.1$

Pr$_{1-x}$Gd$_x$Al$_2$

$x = 0.2$

Magnetic compensation phenomenon in $\text{Nd}_{1-x}\text{Ho}_x\text{Al}_2 : x \sim 0.25$

$\text{NdAl}_2 : T_c \sim 65 \text{ K}$

$\text{HoAl}_2 : T_c \sim 35 \text{ K}$

$\mu_{\text{Nd}} / \text{f.u.} \sim 2.5 \mu_\text{B}$

$\mu_{\text{Ho}} / \text{f.u.} \sim 7.7 \mu_\text{B}$

$\text{Nd}_{1-x}\text{Ho}_x\text{Al}_2$

$x = 0.25$

$T_c \sim 65 \text{ K}$

$T_{\text{comp}} \sim 25 \text{ K}$

RE based ‘Zero-magnetization spin -ferromagnets’

Schematic

Pr$_{1-x}$Gd$_x$Al$_2$

Pr

\[ \mu_{Pr}, J_{Pr}, L_{Pr}, S_{Pr} \]

Gd

\[ S_{Gd}, J_{Gd}, \mu_{Gd} \]

R$_{1-x}$R$'_x$X$_n$

R : First half

\[ \mu_R, J_R, L_R, S_R \]

R$'$ : Second half

\[ S_{R'}, L_{R'}, J_{R'}, \mu_{R'} \]

ZMSF (R$_{1-x}$R$'_x$X$_n$) : (1-x) $\mu_R \approx x \mu_{R'}$
‘Zero-magnetization System’

• Every site on a crystalline sub-lattice is randomly occupied by two species of magnetic ions, which are coupled antiferromagnetically.

• The relative compositions of two antiferromagnetically coupled magnetic ions is such that the net magnetization of the system is nearly ZERO.

• Conveniently realized in a Ferromagnetic Rare Earth (RE) Intermetallic Series

\[ RAl_2 : \text{Cubic } Cu_2Mg \text{ structure, } R \text{ atoms form a diamond lattice.} \]
Hyperfine fields in Sm$_{1-x}$Gd$_x$Al$_2$ alloys - Microscopic evidence for ferromagnetic coupling between rare earth spins


Tata Institute of Fundamental Research, Bombay 400 005, India
K. Shimizu
Faculty of Education, Toyama University, Toyama, Japan

Abstract: The results of hyperfine field studies on Sm$_{1-x}$Gd$_x$Al$_2$ alloys for 0<x<0.5 are reported. The hyperfine field at Al, H(Al), has been measured to be +32.5 kOe in SmAl$_2$ and -47 kOe in GdAl$_2$. In Sm$_{1-x}$Gd$_x$Al$_2$ alloys, we find that the magnitude of H(Al) increases with increasing x and further H(Al) becomes negative even with small replacement of Sm by Gd. H(Al) in these compounds is proportional to the average value of spin per rare earth ion. The observed behaviour can be understood in terms of a ferromagnetic coupling between the spins of Sm and Gd.

Hyperfine field at Al $\propto <S_z>_{\text{RE}}$
SmAl$_2$: Ferromagnet, $T_c \sim 125$ K

Magnetic Moment / formula unit of SmAl$_2$ $\sim 0.2 \mu_B$

GdAl$_2$: Ferromagnet, $T_c \sim 170$ K

Magnetic Moment / formula unit of GdAl$_2$ $\sim 7.7 \mu_B$
d) Magnetic Interaction Studies:

The magnetic hyperfine interaction of $^{111}$Cd in Sm$_{1-x}$Gd$_x$Al$_2$ compounds was studied at 80 K for Gd concentration of 2-10 at %. A least square fit program has been developed to fit the TDPAC patterns in case of such combined magnetic and quadrupole interactions. The results show that the hyperfine magnetic field $^{111}$Cd in these compounds reverses sign at a Gd concentration of 3 at %.
Can Admixed Rare Earth Intermetallics be described as Spin Glass Systems?

Proc. of DAE N.P. and SSP Symposium, 21C, 546 (1978)

Fig. 1. ac susceptibility in Sm$_{1-x}$Gd$_x$Al$_2$ alloys (GdAl$_2$ data is in a field of about 0.1Oe).
Magnetic compensation phenomenon in Sm$_{1-x}$Gd$_x$Al$_2$

- Magnetic reorientation (compensation) seen for 1% to 3% Gd doping in DC magnetization response
- Resistance data fingerprints drop of spin disorder contribution at $T_c$
  No fingerprint of compensation or reorientation in resistance data
- AC-susceptibility registers freezing of Gd moments as peaks

**RRh₃B₂ series: Crystal structure of the type CeCo₃B₂**

- **Space Group:** P6/mmm
- **a = 5.484 Å**
- **c = 3.087 Å**
- **T_c = 110 K,**
- **moment / f.u. = 0.4 μ_B**

- Hexagonal crystal structure of the type CeCo₃B₂ for the RRh₃B₂ compounds.
Effect of Gd substitution on anomalous ferromagnetism in CeRh$_3$B$_2$

Presented at International Conference on Valence Fluctuations, Bangalore, January, 1987

RRh$_3$B$_2$ $\quad T_c$

CeRh$_3$B$_2$ $\sim$ 110 K

GdRh$_3$B$_2$ $\sim$ 90 K

NdRh$_3$B$_2$ $\sim$ 10 K

$M$ versus $T$ in zero field cooled and field cooled states in Ce$_{0.95}$Gd$_{0.05}$Rh$_3$B$_2$

Comparison of magnetic compensation response in $\text{Sm}_{0.98}\text{Gd}_{0.02}\text{Al}_2$ and $\text{Nd}_{0.85}\text{Gd}_{0.15}\text{Rh}_3\text{B}_2$

(Grover and Dhar, 1986)
Contemporary Interest

“A ferromagnet having no magnetic moment”

Temperature dependence of Magnetization in (pseudo) Ferrimagnetic system
Sm$_{1-x}$Gd$_x$Al$_2$

‘Self-Compensation’?

A magnetic moment comprising nearly equal and opposite sub-parts, as for example, orbital and spin contributions to the total magnetic moment of Sm$^{3+}$.

Sm ion in a metallic matrix

Magnetization signal in a Sm system = Orbital contribution + Spin contribution + Cond. Electron Polarization contribution

Notionally: Orbital contribution > Contribution from spins

“Orbital Surplus” system

Occasionally: Contribution from spins > Orbital contribution

“Spin Surplus” system

- Admixture of higher excited states into the ground state due to strong Exchange field and crystalline electric field.

- Different temperature dependences for the ‘spin’ and ‘orbital’ parts of the Samarium moments
Adachi et al.:
Differences in M-T Curves in Ferromagnetic Samarium compounds

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<th>m_{total} (μB)</th>
<th>m_{4f} (μB)</th>
<th>-L_{z}</th>
<th>-2S_{z}</th>
<th>m_{cond} (μB)</th>
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<td>SmZn</td>
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<td>4.37</td>
<td>-3.87</td>
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Calculated T = 0 values

Notion of orbital and spin surplus moments in Sm systems

RECENT STUDIES AT TIFR

Magnetic compensation in a single crystal of Nd$_{0.75}$Ho$_{0.25}$Al$_2$

Nd$^{3+}$ and Ho$^{3+}$ ions randomly occupy the sites on a diamond lattice.

- Low field zero-crossover and the field induced reversal in magnetic moments

Prasanna D. Kulkarni et al., Europhysics Letters 86, 47003 (2009)
Magnetization hysteresis loops in pseudo-ferrimagnetic Nd$_{0.75}$Ho$_{0.25}$Al$_2$

- $M - H$ response is anti-ferromagnetic like very close to $T_{\text{comp}}$
Magnetization hysteresis loops in the very close vicinity of $T_{\text{comp}}$

- Note the visible shift in the $M-H$ loop at 23.75 K and 24 K and the symmetric $M-H$ loop at 23.85 K.
- (Left/Right) Shift in M-H loop is called Exchange Bias Field

Prasanna D. Kulkarni et al., Europhysics Letters 86,47003 (2009)
The notion of exchange bias is observed in the admixed rare earth intermetallics near $T_{\text{comp}}$

P. Kulkarni et al., Europhysics Letters 86, 47003 (2009)
EXCHANGE BIAS FIELD IN FM/AFM LAYERS (USED IN SPIN VALVES)

Comparison of the multi-layer structure and the admixed RE alloys

The soft conduction electron contribution could assume a role of the ferromagnetic layer near the magnetic compensation region

Prasanna D. Kulkarni et al., Europhysics Letters 86, 47003 (2009)
Magnetic compensation in a single crystal of Nd$_{0.75}$Ho$_{0.25}$Al$_2$

- Field induced reversal in magnetic moments of Ho/Nd imprints a peak in the specific heat data
- Oscillatory response in the magneto-resistance

Prasanna D. Kulkarni et al., Europhysics Letters 86,47003 (2009)
Magnetic compensation in (polycrystalline) Pr$_{1-x}$Gd$_x$Al$_2$

- Magnetization crosses $M = 0$ axis at a specific temperature, marked as $T_{\text{comp}}$, in nominal zero field.
- In higher values of the external magnetic field, a magnetic turn around behaviour is observed.

Fingerprint of Reorientation in AC-susceptibility, specific heat data and phase reversal in the exchange bias field

The electrical resistance data in admixed RE systems

- Spin-disorder resistivity drops out at the magnetic ordering temperature in $H = 0$ Oe
- The oscillatory response in the magneto-resistance data recorded upto 50 kOe

Prasanna D. Kulkarni et al., to be submitted
The electrical resistivity measurements in the presence of the magnetic field

For $H = 50 \text{ kOe}$:

- $T^* \approx 40 \text{ K}$
- $T_c \approx 64 \text{ K}$

For $H = 10 \text{ kOe}$:

- $T^* \approx 38 \text{ K}$
- $T_c \approx 64 \text{ K}$
Magnetic compensation in ‘spin surplus’ Sm$_{0.98}$Gd$_{0.02}$Al$_2$

- The ‘orbital-surplus’ SmAl$_2$ alloy is doped with Gd$^{3+}$ ions to aid the spins of the Sm$^{3+}$ ions.

- The temperature dependence of the net magnetization display a low field zero-crossover and a minimum at high fields.

Hysteresis loops of Sm$_{0.98}$Gd$_{0.02}$Al$_2$

S. Venkatesh et al., to be submitted
The notion of exchange bias is observed in the 'spin surplus' Gd doped SmAl$_2$ with near zero magnetization stoichiometry.

The generic temperature dependence of the effective coercive field and the exchange bias field in the admixed alloys.
Step change in the temperature dependence of magnetization, concomitant with the phase transition like attribute across $T_{\text{comp}}$
Fingerprint of Compensation phenomenon in Specific heat of Sm$_{1-x}$Gd$_x$Al$_2$ (x = 0.01 and 0.02)

Notion of field induced spin-orbit reorientation as a phase transition across compensation temperature

X. H. Chen …C. W. (Paul) Chu, 
Fingerprint of phase transition observed in the specific heat data as well as the magnetization data (TIFR data)
\[ M(\mu_B / \text{I.U.}) \]
\[ H_c (\text{kOe}) \]
\[ \chi_{\text{hf}} \text{ (arb. units)} \]
\[ H_{\text{exch}} (\text{Oe}) \]

Temperature (K)

S. Venkatesh et al., to be submitted
Examples of self-compensation
The temperature dependence of the magnetization and the hysteresis loops in ferromagnet SmPtZn

SmPtZn

$T_c \approx 48 \text{ K}$

$H = 10 \text{ kOe}$

$T = 10 \text{ K}$$\rightarrow$$M (\mu_B/\text{f.u.})$$H = 10 \text{ kOe}$$M (\mu_B/\text{f.u.})$$H (\text{kOe})$

$T = 30 \text{ K}$

$H_{\text{exch}} \approx 1 \text{ kOe}$

Sm$^{3+}$ ions are on a FCC lattice

P. D. Kulkarni, S. K. Dhar, et. al., to be submitted
The temperature dependence of the exchange bias field in SmPtZn compounds
Hysteresis loops in the ferromagnet SmCu$_4$Pd

Sm$^{3+}$ ions occupy a unique site in the cubic (AuBe$_5$) structure


P. D. Kulkarni, S. K. Dhar et. Al., to be submitted
Temperature dependence of the magnetization in Sm$_{1-x}$Gd$_x$Cu$_4$Pd series: Sm/Gd ions are on a FCC lattice.
Magnetic compensation in $\text{Sm}_{0.975}\text{Gd}_{0.025}\text{Cu}_{4}\text{Pd}$

(a) $T_c$ ($\approx 31$ K) and $T_{\text{comp}}$ ($\approx 21$ K) as a function of temperature and field.

(b) $H_c^\text{eff}$ and $H_{\text{exch}}$ as a function of temperature.

(c) $M$ as a function of field at $T = 24.5$ K and $T = 18.5$ K.
Temperature dependence of the exchange bias field in Sm$_{1-x}$Gd$_x$Cu$_4$Pd series
Exchange bias in ‘spin-surplus’ $\text{Sm}_{1-x}\text{Gd}_x\text{Al}_2$ ($x = 0.03, 0.04$)

- The pseudo-antiferromagnetic response in $\text{Sm}_{0.96}\text{Gd}_{0.04}\text{Al}_2$ alloy

- Note the monotonically increasing positive exchange bias field at lower temperatures in $\text{Sm}_{0.96}\text{Gd}_{0.04}\text{Al}_2$ in contrast to its sign reversal in $\text{Sm}_{0.97}\text{Gd}_{0.03}\text{Al}_2$

- No exchange bias in ‘spin-surplus’ $\text{Sm}_{0.94}\text{Gd}_{0.06}\text{Al}_2$
The pseudo-antiferromagnetic behavior in pristine SmScGe.

Note the magnetic ordering temperature $T_c \sim 270$ K, close to the ambient temperatures.


FAST TRACK COMMUNICATION

“New samarium and neodymium based admixed ferromagnets with near-zero net magnetization and tunable exchange bias field”
The magnetic compensation in ‘spin surplus’ SmScGe could be achieved with substitution of 9 at. % Nd$^{3+}$ ions.

Note the net magnetization values over the magnetically ordered state are $< 0.15 \mu_B$ even up to 50 kOe.

The magnetic ordering temperature is close to the ambient temperatures, most suitable for applications.
Tuning of the exchange bias in doped SmScGe alloy

P. Kulkarni et al., Unpublished
Effective coercive field in doped SmScGe alloy

P. Kulkarni et al., Unpublished
M-H loops in other ferromagnetic Samarium compounds
High field magnetization response in the admixed $\text{Nd}_{1-x}\text{Gd}_x\text{Rh}_3\text{B}_2$ series of alloys

- Note the paramagnetic state values of the net magnetization

Prasanna D. Kulkarni et al., PRB 78, 064426 (2008)
Exploration of Zero Magnetization stoichiometry:
Nd$_{0.75}$Gd$_{0.25}$Rh$_3$B$_2$

- Oscillatory magnetic response is observed in the thermomagnetic curves of Nd$_{0.75}$Gd$_{0.25}$Rh$_3$B$_2$.
- The specific heat data displays two underlying magnetic transitions (antiferromagnetic like).

Prasanna D. Kulkarni et al., PRB 78, 064426 (2008)
Magnetization curves in the single crystal $\text{Nd}_{0.8}\text{Tb}_{0.2}\text{Al}_2$

$M$ ($\mu_B$/f.u.) vs Temperature (K)

- $x = 0$
- $x = 0.1$
- $x = 0.15$
- $x = 0.175$
- $x = 0.25$
- $x = 0.225$

$H = 10$ kOe
Low field FCC curves in the single crystal $\text{Nd}_{0.8}\text{Tb}_{0.2}\text{Al}_2$: Repeated magnetic compensation

![Graph showing magnetic properties of $\text{Nd}_{0.8}\text{Tb}_{0.2}\text{Al}_2$.](image)

$T_{f1} (\approx 88 \text{ K})$ and $T_{f2} (\approx 32 \text{ K})$ are marked on the graph. The temperature range is from 0 to 120 K, and the magnetic moment is measured in $\mu_B / \text{f.u.}$. The applied magnetic fields are $H = 125 \text{ Oe}$ and $H = 150 \text{ Oe}$. The graph shows the magnetic behavior of the crystal under these conditions.
Repeated magnetic compensation phenomenon in polycrystalline Nd$_{0.75}$Gd$_{0.25}$Rh$_3$B$_2$ and Nd$_{0.8}$Tb$_{0.2}$Al$_2$ alloys: Comparative study
Summary

• New features:

(i) Exemplification of the existence of exchange bias field on approaching $T_{\text{comp}}$ and its phase reversal across $T_{\text{comp}}$ in admixed RE intermetallics

(ii) Evidence of the ‘self compensation’ behavior in the pristine ‘orbital-surplus’ and ‘spin-surplus’ Samarium intermetallics

(iii) Synthesis of alloys undergoing magnetic orderings close to the ambient temperatures and possessing large CEP, but, near-zero bulk magnetization, and permitting easy tuning of the exchange bias field for novel applications, etc.

(iv) Oscillatory character in the magneto-resistance response, including a change in its sign at $T_{\text{comp}}$.

(v) Sign reversal in Hall resistance across $T_{\text{comp}}$.

(vi) Identification of a step change in high field magnetization and its correlation with the fingerprint of field-induced entropic change in specific heat data.

(vii) Novel repeated magnetic compensation behavior in some specific alloys.
Publications:

Thank you.