

Structure and Magnetism of REPtMg (RE = Pr, Nd, Sm)

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New magnesium based intermetallic compounds PrPtMg, NdPtMg and SmPtMg were synthesized from the elements by reaction in sealed tantalum tubes in a high-frequency furnace. The three compounds were investigated by X-ray powder and single crystal diffraction: ZrNiAl type, space group $P6_3m$, $a = 752.34(8)$, $c = 412.66(4)$ pm, $wR2 = 0.0668$, 341 F^2 values, 14 variables for PrPtMg, $a = 748.80(8)$, $c = 411.52(4)$ pm, $wR2 = 0.0521$, 196 F^2 values, 14 variables for NdPtMg and $a = 743.90(5)$, $c = 409.80(3)$ pm, $wR2 = 0.0489$, 248 F^2 values, 12 variables for SmPtMg. From a geometrical point of view these structures are composed of two types of platinum centered trigonal prisms, *i. e.* $[Pt1Mg_3RE_6]$ and $[Pt2Mg_6RE_3]$. These prisms are condensed via common edges and faces. Together the platinum and magnesium atoms build three-dimensional [PtMg] networks in which the rare earth atoms are located in distorted pentagonal channels. Magnetic susceptibility data of PrPtMg show Curie-Weiss behaviour with an experimental magnetic moment of $3.59(2) \mu_B$ and a paramagnetic Curie temperature of $7.5(5)$ K. Ferromagnetic ordering is detected at $T_C = 8.0(5)$ K with a magnetic moment of $1.75(5) \mu_B/Pr$ at 4.5 K and 5 T. SmPtMg orders ferromagnetically below $52(1)$ K with a presumably complex spin structure in the ordered state.

Introduction

The equiatomic ternary indides $REPtIn$ ($RE = Sc, Y, La - Nd, Sm - Lu$) have intensively been investigated in the past with respect to their crystal structures and greatly varying physical properties [1 - 15]. The rare earth atoms are trivalent in most $REPtIn$ compounds. They crystallize with the hexagonal ZrNiAl type structure [16 - 18], a ternary ordered variant of the well known Fe_2P type [19]. EuPtIn [10, 11] with the orthorhombic TiNiSi type structure, however, contains divalent europium. CePtIn [3 - 8] and YbPtIn [12, 13] have most intensively been investigated because of their intriguing heavy fermion behaviour. Susceptibility measurements showed paramagnetic behaviour down to 1.7 K for PrPtIn [14] while comparatively high ordering temperatures have been observed for compounds with the heavier rare earth metals, *i. e.* $T_C = 25$ K for SmPtIn [14], $T_N = 16$ K for EuPtIn [11], $T_N = 50$ K for TbPtIn, $T_C = 89$ K for GdPtIn and $T_C = 38$ K for DyPtIn [9]. Recently it has been demonstrated that the indium atoms in these indides can completely be substituted by magnesium or cadmium [20 - 28]. Although the electron count

is reduced, the magnesium compounds still are isotopic with the indides. In the series with platinum as transition metal component we already have synthesized LaPtMg and CePtMg [29]. We have now extended these studies to include other rare earth metals. The syntheses, structure refinements and the magnetic behaviour of PrPtMg, NdPtMg and SmPtMg are reported herein. The magnesium compounds show significantly higher ordering temperatures than the indides.

Experimental

Starting materials for the preparation of PrPtMg, NdPtMg and SmPtMg were ingots of the rare earth metals (Johnson Matthey, > 99.9%), platinum powder (Degussa-Hüls, 200 mesh, > 99.9%) and a magnesium rod (Johnson Matthey, \varnothing 16 mm, > 99.95%). The rare earth metal pieces were cut into smaller fragments under paraffin oil. These pieces were washed with *n*-hexane and arc-melted [30] to small buttons (about 400 mg) under an argon atmosphere and then kept in Schlenk tubes. The paraffin oil and *n*-hexane were dried over sodium wire. The argon was purified over titanium sponge (900 K), silica gel, and molecular sieves. The rare earth metal buttons were mixed with the platinum powder and magnesium pieces

Table 1. Crystal data and structure refinement for the hexagonal compounds PrPtMg, NdPtMg and SmPtMg (space group $P\bar{6}2m$, $Z = 3$).

	PrPtMg	NdPtMg	SmPtMg
Empirical formula	PrPtMg	NdPtMg	SmPtMg
Molar mass	360.31 g/mol	363.64 g/mol	369.75 g/mol
Lattice parameters (powder data)	$a = 752.34(8)$ pm $c = 412.66(4)$ pm $V = 0.2023$ nm ³	$a = 748.80(8)$ pm $c = 411.52(4)$ pm $V = 0.1998$ nm ³	$a = 743.90(5)$ pm $c = 409.80(3)$ pm $V = 0.1964$ nm ³
Calculated density	8.87 g/cm ³	9.07 g/cm ³	9.38 g/cm ³
Crystal size	10 × 20 × 25 μm ³	8 × 15 × 15 μm ³	6 × 10 × 12 μm ³
Transmission ratio (max/min)	2.41	–	2.03
Absorption coefficient	69.5 mm ⁻¹	71.6 mm ⁻¹	75.4 mm ⁻¹
$F(000)$	447	450	456
Detector distance	–	–	50 mm
Exposure time	–	–	25 min
ϕ Range; increment	–	–	0 - 200°; 1.4°
Profile / pixel	–	–	7 - 27
θ Range for data collection	3° to 34°	4° to 28°	3° to 31°
Range in hkl	±11, ±11, +6	±9, ±9, ±5	±10, ±10, ±5
Total no. reflections	1842	3639	2276
Independent reflections	341 ($R_{\text{int}} = 0.1268$)	196 ($R_{\text{int}} = 0.1017$)	248 ($R_{\text{int}} = 0.0720$)
Reflections with $I > 2(I)$	283 ($R_{\sigma} = 0.0671$)	194 ($R_{\sigma} = 0.0286$)	225 ($R_{\sigma} = 0.0374$)
Data / parameters	341 / 14	196 / 14	248 / 12
Goodness-of-fit on F^2	1.030	1.151	1.060
Final R indices [$I > 2(I)$]	$R1 = 0.0363$ $wR2 = 0.0631$	$R1 = 0.0216$ $wR2 = 0.0516$	$R1 = 0.0236$ $wR2 = 0.0478$
R Indices (all data)	$R1 = 0.0516$ $wR2 = 0.0668$	$R1 = 0.0221$ $wR2 = 0.0521$	$R1 = 0.0294$ $wR2 = 0.0489$
Extinction coefficient	0.003(1)	0.0033(8)	0.0019(5)
Flack parameter	-0.03(3)	-0.04(3)	0.01(3)
Largest diff. peak and hole	2.07 and -2.66 e/Å ³	1.28 and -1.57 e/Å ³	1.91 and -3.13 e/Å ³

in the ideal 1:1:1 atomic ratio and sealed in tantalum tubes (tube volume about 1 cm³) under an argon atmosphere of about 800 mbar.

The closed tantalum cylinders were put into a water-cooled quartz glass sample chamber in a high-frequency furnace (HÜTTINGER TIG 1.5 / 300, 1.5 kW) under flowing argon [31]. They were first heated for one minute at about 1500 K, cooled to about 1100 K and heated again to 1500 K. Subsequently the tubes were annealed at about 900 K for another two hours and then quenched by radiative heat loss. The reaction of the three elements was indirectly visible by a heat flash. The samples could easily be separated from the tantalum tubes. No reactions of the samples with the tubes could be detected. The compounds were obtained in about 1 g quantities. Compact pieces are light grey with metallic lustre. The samples are stable in moist air. No decomposition was observed after several months.

The purity of the samples was checked by X-ray powder diffraction. The powder patterns were recorded on a Stoe StadiP diffractometer with Cu-K α_1 radiation and 5 N silicon ($a = 543.07$ pm) as an external standard. The hexagonal lattice parameters (see Table 1) were obtained

from least-squares fits of the powder data. The correct indexing of the patterns was ensured through intensity calculations [32] using the atomic positions of the structure refinements. The lattice parameters determined from the powders and the single crystals agreed well. PrPtMg and SmPtMg were obtained as single phase materials, while a small, yet unknown impurity phase was detected in the NdPtMg sample.

Irregularly shaped single crystals of the three compounds were isolated from the samples by mechanical fragmentation. They were examined by Laue photographs on a Buerger precession camera in order to establish their quality. Single crystal intensity data of PrPtMg were collected at room temperature by use of a four-circle diffractometer (CAD4) with graphite monochromatized Mo-K α radiation (71.073 pm) and a scintillation counter with pulse height discrimination. The scans were performed in the $\omega/2\theta$ mode. Empirical absorption corrections were applied on the basis of Ψ -scan data. The SmPtMg crystal was investigated in the oscillation mode on a Stoe image plate system (IPDS) with graphite monochromatized Mo-K α radiation. A numerical absorption correction was applied to the data. The NdPtMg crystal was investigated on a

Table 2. Atomic coordinates and isotropic displacement parameters (pm^2) for PrPtMg, NdPtMg and SmPtMg. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	Wyckoff position	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
<i>PrPtMg</i> :					
Pr	3 <i>f</i>	0.5862(2)	0	0	102(3)
Pt1	2 <i>d</i>	2/3	1/3	1/2	109(3)
Pt2	1 <i>a</i>	0	0	0	113(3)
Mg	3 <i>g</i>	0.239(1)	0	1/2	82(17)
<i>NdPtMg</i> :					
Nd	3 <i>f</i>	0.5866(1)	0	0	139(3)
Pt1	2 <i>d</i>	2/3	1/3	1/2	128(3)
Pt2	1 <i>a</i>	0	0	0	130(3)
Mg	3 <i>g</i>	0.2414(8)	0	1/2	71(11)
<i>SmPtMg</i> :					
Sm	3 <i>f</i>	0.4124(2)	0	0	55(3)
Pt1	2 <i>d</i>	1/3	2/3	1/2	57(2)
Pt2	1 <i>a</i>	0	0	0	63(3)
Mg*	3 <i>g</i>	0.757(1)	0	1/2	53(15)

* The magnesium positions in SmPtMg has been refined with an isotropic displacement parameter.

Nonius KappaCCD (Mo- K_{α} radiation) by omega scans: 30 mm detector distance, 2.0° scan step, and 120 s scan time. All relevant crystallographic data and experimental details for the data collections are listed in Table 1.

The data sets showed no systematic extinctions and space group $P\bar{6}2m$ was found to be correct during the structure refinements in agreement with previous work [15, 23, 26]. The atomic positions of SmPtIn [14] were taken as starting values and the three structures were successfully refined using SHELXL-97 (full-matrix least-squares on F_o^2) [33] with anisotropic atomic displacement parameters for the rare earth metal and platinum sites. Only the magnesium position of the praseodymium compound could be refined anisotropically. The standard deviations of the isotropic displacement parameters of the magnesium positions of the samarium compound were too high (Table 2). Thus it was not reasonable to introduce anisotropic displacement parameters for these sites.

Refinement of the correct absolute structure was ensured through refinement of the Flack parameter [34, 35]. As a check for the correct composition, the occupancy parameters of all sites were refined in a separate series of least-squares cycles. All sites were fully occupied within two standard deviations. In the final cycles the ideal occupancies were assumed again. Final difference Fourier syntheses revealed no significant residual peaks (see Table 1). The positional parameters and interatomic distances are

Table 3. Interatomic distances (pm) in the structures of PrPtMg, NdPtMg and SmPtMg, calculated with the atomic positions refined from the single crystal data and the lattice parameters taken from X-ray powder data. Standard deviations are given in parentheses. All distances within the first coordination sphere are listed.

— PrPtMg —		— NdPtMg —		— SmPtMg —	
Pr:	4 Pt1 306.5(1)	Nd:	4 Pt1 305.4(1)	Sm:	4 Pt1 303.9(1)
	1 Pt2 311.3(2)		1 Pt2 309.5(1)		1 Pt2 306.8(1)
	2 Mg 332.8(7)		2 Mg 330.4(5)		2 Mg 328.2(6)
	4 Mg 340.4(1)		4 Mg 338.9(1)		4 Mg 336.6(1)
	4 Pr 392.6(1)		4 Nd 390.9(1)		4 Sm 388.7(1)
	2 Pr 412.7(1)		2 Nd 411.5(1)		2 Sm 409.8(1)
Pt1:	3 Mg 292.7(6)	Pt1:	3 Mg 290.2(4)	Pt1:	3 Mg 287.6(5)
	6 Pr 306.5(1)		6 Nd 305.4(1)		6 Sm 303.9(1)
Pt2:	6 Mg 273.7(6)	Pt2:	6 Mg 273.9(4)	Pt2:	6 Mg 273.2(5)
	3 Pr 311.3(2)		3 Nd 309.5(1)		3 Sm 306.8(1)
Mg:	2 Pt2 273.7(6)	Mg:	2 Pt2 273.9(4)	Mg:	2 Pt2 273.2(5)
	2 Pt1 292.7(6)		2 Pt1 290.2(4)		2 Pt1 287.6(5)
	2 Mg 312(2)		2 Mg 313(1)		2 Mg 313(1)
	2 Pr 332.8(7)		2 Nd 330.4(5)		2 Sm 328.2(6)
	4 Pr 340.4(1)		4 Nd 338.9(1)		4 Sm 336.6(1)

listed in Tables 2 and 3. Listings of the observed and calculated structure factors are available*.

The magnetic susceptibilities of polycrystalline, powdered samples of PrPtMg and SmPtMg were determined with a MPMS XL SQUID magnetometer (Quantum Design, Inc.) in the temperature range 4.2 to 300 K with magnetic flux densities up to 5 T. The samples were cooled to 4.2 K in zero magnetic field and slowly heated to room temperature in applied external fields.

Results and Discussion

Crystal chemistry and chemical bonding

Three new intermetallic magnesium compounds, *i. e.* PrPtMg, NdPtMg and SmPtMg have been synthesized and their structures refined from single crystal X-ray data. The intermetallics crystallize with the hexagonal ZrNiAl structure [16 - 18], a ternary ordered variant of the Fe_2P type [19]. From a geometrical point of view, these structures are built up from two different types of trigonal prisms, *i. e.* $[\text{Pt1Mg}_3\text{RE}_6]$ and $[\text{Pt2Mg}_6\text{RE}_3]$. In Fig. 1 we exemplarily present the SmPtMg structure as a projection along the short axis. The trigonal prisms are condensed *via* common edges and

*Details may be obtained from: Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen (Germany), by quoting the Registry No.'s. CSD-412100 (PrPtMg), CSD-412356 (NdPtMg) and CSD-412102 (SmPtMg).

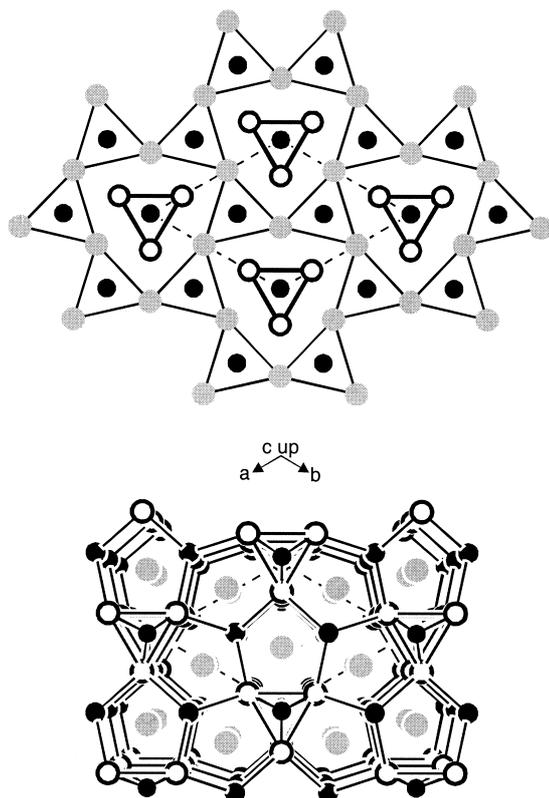


Fig. 1. The crystal structure of *SmPtMg*. In the upper drawing the platinum centered trigonal prisms are outlined. All atoms lie on mirror planes at $z = 0$ (thin lines) and $z = 1/2$ (thick lines). Samarium, platinum and magnesium atoms are drawn as grey, filled and open circles, respectively. At the bottom the three-dimensional [PtMg] network is emphasized.

triangular faces. The shortest interatomic distances occur between the platinum and magnesium atoms at Pt-Mg distances between 273 and 288 pm. Together the platinum and magnesium atoms build a three-dimensional [PtMg] network in which the samarium atoms are located in distorted pentagonal channels.

The Pt-Mg distances within the [PtMg] network are longer than the sum of the covalent radii of 266 pm [36] indicating only weak Pt-Mg interactions. The trigonal prisms at the origin of the unit cell have Mg-Mg distances of 313 pm within the triangular faces. These are shorter than the average Mg-Mg distance of 320 pm in *hcp* magnesium [37]. We can thus assume a significant degree of Mg-Mg interactions within the three-dimensional network. The crystal chemistry of the ZrNiAl type

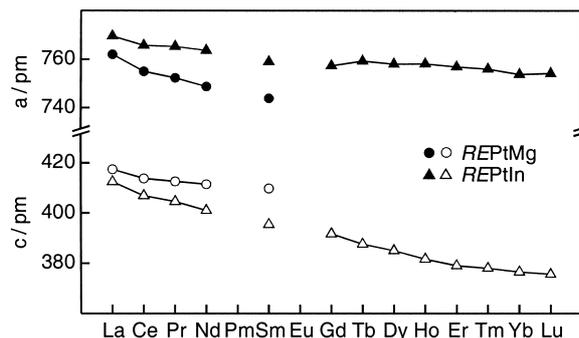


Fig. 2. Plot of the lattice parameters of the *REPtIn* [1 - 15] and *REPtMg* [29] compounds with hexagonal ZrNiAl type structure. The solid lines serve as a guide to the eye.

compounds has been discussed and reviewed in detail in various papers [38 - 40]. For further details we therefore refer to this literature.

At this point it is interesting to compare the magnesium compounds presented herein with the series of isotopic indides [1 - 15] reported in recent years. In Fig. 2 we present a plot of the lattice parameters vs. the rare earth metal component. If we consider the volumes of the unit cells, they are smaller for the magnesium compounds as could have been expected from the course of the metallic radii (160 pm for Mg and 163 pm for In) [36]. The course of the lattice parameters, however, is different. The *a* parameters are smaller for the magnesium compounds, while reversely the *c* parameters are smaller for the indides. This behaviour indicates that the *a* parameters are governed mainly by geometrical and the *c* parameters primarily by electronic factors. In a recent paper we compared the two series *RE*₂Cu₂In and *RE*₂Cu₂Mg [41]. The electronic structures of La₂Cu₂In and La₂Cu₂Mg were investigated by extended Hückel calculations. These studies showed higher overlap populations for the Cu-In than for the Cu-Mg contacts. This presumably is also the case for the equiatomic compounds reported herein.

Single crystal data are now available for *SmPtIn* [14] and *SmPtMg*. In both structures, the shortest interatomic distances occur for the Pt-In (277 pm) and Pt-Mg (273 pm) contacts. The Pt-In distances are shorter than the sum of the covalent radii (279 pm), while the Pt-Mg distances are longer (266 pm) [36]. Thus, the strength of chemical bonding within these trigonal prisms significantly governs the course of the lattice parameter *c*.

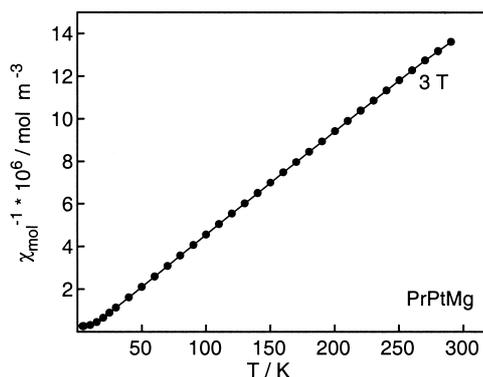


Fig. 3. Temperature dependence of the inverse magnetic susceptibility of PrPtMg measured at an external flux density of 3 T.

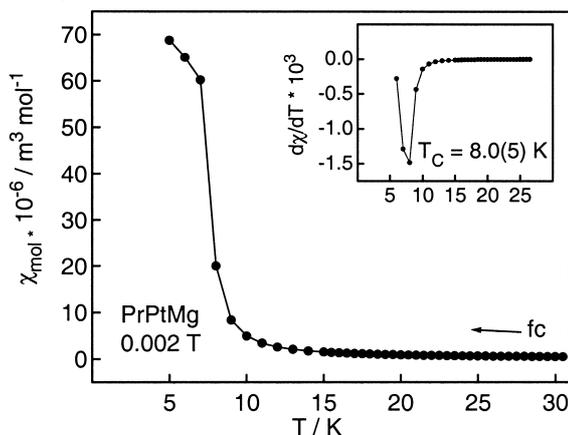


Fig. 4. Susceptibility of PrPtMg measured in field-cooling mode at an external flux density of 0.002 T. The Curie temperature was determined from the derivative $d\chi/dT$ (insert).

Magnetic properties

PrPtMg and SmPtMg were obtained as single phase materials. We could thus determine the magnetic properties of these two compounds. The temperature dependence of the inverse magnetic susceptibility of PrPtMg is shown in Fig. 3. PrPtMg shows Curie-Weiss behaviour above 50 K. A fit of these data according to a modified Curie-Weiss expression $\chi = \chi_0 + C/(T - \theta)$ resulted in a temperature independent contribution $\chi_0 = 1.63(3) \cdot 10^{-9} \text{ m}^3/\text{mol}$, an experimental magnetic moment of $3.59(2) \mu_B/\text{Pr}$ and a paramagnetic Curie temperature (Weiss constant) $\theta = 7.5(5) \text{ K}$. The experimental moment is close to the free ion value ($3.58 \mu_B$) of Pr^{3+} . The temperature independent contribution is in the order of magnitude of a Pauli para-

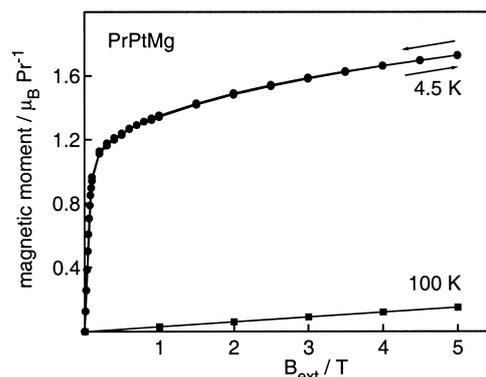


Fig. 5. Magnetization vs external magnetic flux density of PrPtMg measured at 4.5 and 100 K.

magnet and most likely results from the conduction electrons of this metallic compound.

Ferromagnetic ordering of the praseodymium magnetic moments was detected at low temperatures. The precise Curie temperature of $T_C = 8.0(5) \text{ K}$ was determined from the derivative $d\chi/dT$ of a kink-point measurement (insert of Fig. 4) at a magnetic flux density of 0.002 T. The magnetization vs. external magnetic field dependence is linear at 100 K (Fig. 5) as expected for a paramagnetic material. At 4.5 K we observe a strong increase of the magnetization, even at very low field strengths. The magnetization curve shows only a very small hysteresis with negligible coercivity and remanent magnetization classifying PrPtMg as a very soft ferromagnet. At the highest obtainable magnetic field of 5 T the magnetization amounts to $1.75(5) \mu_B/\text{Pr}$, significantly reduced when compared with the maximal value of $3.20 \mu_B/\text{Pr}$ according to $g \times J$ [42]. The reduced moment may be attributed to crystal field splitting. Similar reduced moments have also been observed for the ferromagnets PrCuAl ($1.30 \mu_B/\text{Pr}$) [43], PrRhIn ($1.60 \mu_B/\text{Pr}$) [44] and PrCuSi ($2.02 \mu_B/\text{Pr}$) [45].

The inverse magnetic susceptibility of SmPtMg shows no Curie-Weiss behaviour in the whole temperature range investigated (Fig. 6) as could be expected for a samarium containing compound. This behaviour (Van Vleck paramagnetism) results from the unusual electronic structure of trivalent samarium since the $J = 5/2$ ground state multiplet and the $J = 7/2$ excited multiplet levels are separated only by 1550 K [46]. The ferromagnetic nature of the magnetic phase transition at $T_C = 52(1) \text{ K}$ is evident from the field-cooling measurement at 0.002 T shown

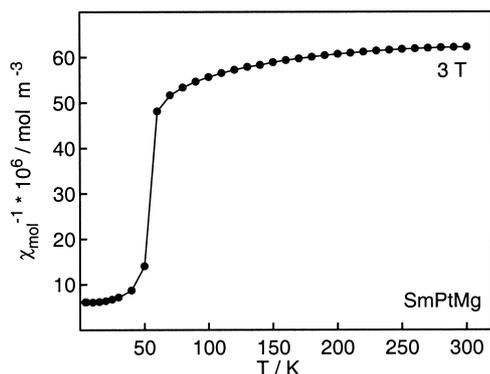


Fig. 6. Temperature dependence of the inverse magnetic susceptibility of SmPtMg measured at an external flux density of 3 T.

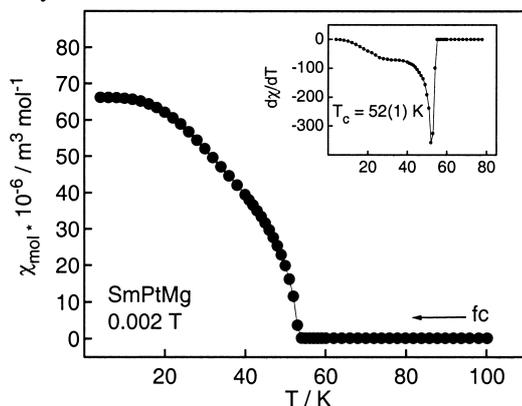


Fig. 7. Susceptibility of SmPtMg measured in field-cooling mode at an external flux density of 0.002 T. The Curie temperature was determined from the derivative $d\chi/dT$ (insert).

in Fig. 7. The magnetization vs. external magnetic field dependence is displayed in Fig. 8. At 100 K, well above the ordering temperature, we observe a linear increase. The M vs. B dependence at 4.5 K has the characteristics of a ferromagnetic material. The magnetization at the highest obtainable field of 5 T is $0.100(2) \mu_B/\text{Sm}$, significantly smaller than the possible saturation magnetic moment of $0.71 \mu_B/\text{Sm}$

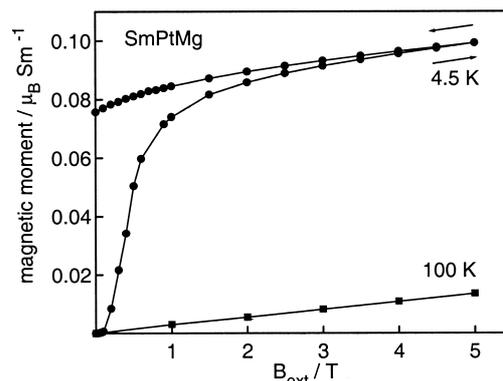


Fig. 8. Magnetization vs. external magnetic flux density of SmPtMg measured at 4.5 and 100 K.

according to $g \times J$ [42]. The reduced moment may be due to crystal field effects or a complex magnetic spin structure which certainly may not be of a simple collinear type. Neutron diffraction studies are planned in order to clarify the magnetic ground state of SmPtMg. Interestingly we observe a remanence magnetization of $0.076(2) \mu_B/\text{Sm}$. The magnetic behaviour of SmPtMg is very similar to that of the isotopic indide, however, with a significantly higher ordering temperature.

Summing up, we synthesized three new intermetallic magnesium compounds REPtMg ($RE = \text{Pr}, \text{Nd}, \text{Sm}$) and refined their ZrNiAl type crystal structures. Magnetic data revealed ferromagnetic ordering for the praseodymium and samarium compound with ordering temperatures of $8.0(5)$ and $52(1)$ K, respectively, much higher than for the isotopic indides.

Acknowledgements

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