Scandium-rich ternary coloring variants of the cubic $\text{Ag}_{7+x}\text{Mg}_{26-x}$ type

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Abstract

The scandium-rich intermetallic compounds $\text{Sc}_{50}\text{T}_{13}\text{In}_3$ ($T = \text{Ni}, \text{Ru}, \text{Pd}$) were synthesized from the elements in sealed tantalum crucibles in an induction furnace. The samples were studied through Guinier powder patterns and their structures were refined from single-crystal X-ray diffractometer data. The $\text{Sc}_{50}\text{T}_{13}\text{In}_3$ phases are site occupancy (coloring) variants of the aristotype $\text{Ag}_{7+x}\text{Mg}_{26-x}$ ($Fm\bar{3}$, $cF264$). Refinements of the occupancy parameters indicated one mixed occupied site for each crystal, leading to the refined compositions $\text{Sc}_{50}\text{Ni}_{13.16(1)}\text{In}_{2.84(1)}$, $\text{Sc}_{49.59(1)}\text{Ru}_{13.41(1)}$, and $\text{Sc}_{50}\text{Pd}_{13.65(2)}\text{In}_{2.35(2)}$. The complex structures can be explained by a condensation of cubes (CN 8), sphenocorona (CN 10), and icosahedra (CN 12). The samples with nickel and palladium are Pauli paramagnets.

Graphic abstract

Keywords Crystal structure · Lanthanoids · Metals · Solid state

Introduction

The rare earth (*RE*) transition metal (*T*) indium systems have intensively been studied with respect to phase formation, crystal structures and the broadly varying physical properties of the many $\text{RE}_x\text{T}_y\text{In}_z$ phases [1]. Most phases have been reported with the 3d transition metals and the early (light) rare earth elements [2]. Many complete isothermal sections of the nickel and copper containing systems have

been studied. Especially, the *RE*–Ni–In systems are rich in compounds. Typical examples are the Ce–Ni–In (12 ternary compounds) [3] and Gd–Ni–In (13 ternary compounds) [4] systems. One of the very few examples of phase analytical studies with 4d and 5d transition metals is the Ce–Pd–In system which also comprises 13 intermetallic phases [5].

The Sc–*T*–In systems are in contrast to the other *RE*–*T*–In systems. A general phase diagram with the so far-observed composition of ternary scandium indides is presented in Fig. 1. Hitherto only 29 $\text{Sc}_x\text{T}_y\text{In}_z$ phases are listed in the Pearson data base [2]. From Fig. 1 it is obvious that no indium-rich phases are known, in contrast to the other *RE*–*T*–In systems which exhibit several series of indium-rich compounds, e.g., YNiAl_4 type phases RETiIn_4 ($T = \text{Ni}, \text{Cu}, \text{Pd}, \text{Ir}, \text{Pt}, \text{Au}$, space group $Cmcm$) [2] or the series $\text{RERhIn}_5/\text{REIrIn}_5$ (HoCoGa_5 type, $P4/mmm$) and $\text{RE}_2\text{RhIn}_8/\text{RE}_2\text{IrIn}_8$ (Ho_2CoGa_8 type, $P4/mmm$) [6–8]. More

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Fig. 2 The two different substructures of $\text{Sc}_{50}\text{Pd}_{13.65}\text{In}_{2.35}$. The left-hand drawing emphasizes the MnCu_2Al (Heusler type) arrangement of the Pd3@Sc_8 , Pd2/In1@Sc_{12} , and In2@Sc_{12} cubes and icosahedra, while part of the condensed Pd1@Sc_{10} sphenocorona are presented at the right

Fig. 3 Temperature dependence of the magnetic susceptibility of the $\text{Sc}_{50}\text{Ni}_{13.5}\text{In}_{2.5}$ and $\text{Sc}_{50}\text{Pd}_{13.5}\text{In}_{2.5}$ samples measured with an applied field of 10 kOe

in *hcp* scandium (6×325 and 6×331 pm) [23], emphasizing the strong Sc–Sc bonding. This is also the case for other scandium-rich solids, e. g., Sc_3C_4 (314 pm) [24] or Sc_2RuMg (304 pm) [25].

Magnetic properties

Figure 3 shows the temperature dependence of the magnetic susceptibility of the $\text{Sc}_{50}\text{Ni}_{13.5}\text{In}_{2.5}$ and $\text{Sc}_{50}\text{Pd}_{13.5}\text{In}_{2.5}$ samples. They show weak, overall positive susceptibilities in the whole temperature range, classifying these indides as Pauli paramagnets with similar room temperature susceptibilities

of 4×10^{-3} emu mol⁻¹. The upturns at low temperature result from tiny amounts of paramagnetic impurities. The magnetic behavior of $\text{Sc}_{50}\text{Ni}_{13.5}\text{In}_{2.5}$ and $\text{Sc}_{50}\text{Pd}_{13.5}\text{In}_{2.5}$ is similar to $\text{Sc}_{50}\text{Co}_{12.5}\text{In}_{3.5}$ (12×10^{-3} emu mol⁻¹) [11], Sc_5CuIn_3 (5×10^{-4} emu mol⁻¹) [16], and $\text{Sc}_2\text{Ni}_2\text{In}$ (3.8×10^{-4} emu mol⁻¹) [26].

Conclusion

The series of $\text{Sc}_{50}T_{13}\text{In}_3$ phases with $T = \text{Ni, Ru, and Pd}$ nicely extends the family of scandium-rich intermetallics with pronounced Sc–Sc bonding. Their complex structures are composed of four different, highly symmetric polyhedra. The three compounds show small homogeneity ranges and manifest the electronic flexibility of such structure types, since they allow for a certain range in the valence electron count. $\text{Sc}_{50}\text{Ni}_{13.5}\text{In}_{2.5}$ and $\text{Sc}_{50}\text{Pd}_{13.5}\text{In}_{2.5}$ are Pauli paramagnets.

Experimental

Synthesis

The three scandium-rich samples were prepared using high-purity metals as starting materials: scandium pieces (Smart Elements; 99.999%), nickel wire (Goodfellow GmbH, 99.99%), ruthenium powder (Agosi; 99.99%), palladium sheets (Agosi; 99.99%), and indium ingots (Smart Elements; 99.995%). The metals were weighed in the atomic ratio of

75.7Sc:20.5 T:3.8In with a total weight of ~ 0.5 g, and sealed in tantalum crucibles under argon atmosphere (purified using titanium sponge (900 K), silica gel, and molecular sieves) by arc-welding [27]. The synthesis was conducted in a high-frequency furnace (Hüttinger Elektronik, Freiburg, Typ TIG 1.5/300), equipped with a water-cooled chamber [28]. The heating procedure consisted of three subsequent steps: (i) the ampoule was heated up to 1720 K and cooled down to 1170 K within 2 h; (ii) annealing at 1170 K for one hour; (iii) gradual cooling to 870 K and keeping this temperature for another 3 h. The gray products showed no reaction with the tantalum crucibles. The samples contained prismatic crystals, which exhibited metallic luster. All three compounds were stable in air over months.

X-ray diffraction on powders and single crystals

The polycrystalline $\text{Sc}_{50}\text{T}_{13}\text{In}_3$ samples were mortared to fine powders and examined by X-ray powder diffraction (Enraf–Nonius FR 552 Guinier camera, $\text{CuK}\alpha_1$ radiation, α -quartz ($a = 491.30$ and $c = 540.46$ pm) as internal standard). The diffraction patterns were collected with an imaging plate technique (Fuji film, BAS-READER 1800). The pattern of the $\text{Sc}_{50}\text{Pd}_{13}\text{In}_3$ sample is presented as an example in Fig. 4. The cubic lattice parameters were deduced from least-squares refinements. Intensity calculations (Lazy-Pulverix routine [29]) facilitated the correct indexing. The powder lattice parameters ($a = 1763.8(2)$ pm for $\text{Sc}_{50}\text{Ni}_{13}\text{In}_3$, $a = 1772.6(2)$ pm for $\text{Sc}_{50}\text{Ru}_{13}\text{In}_3$, and $a = 1785.26(9)$ pm for $\text{Sc}_{50}\text{Pd}_{13}\text{In}_3$) match with the single-crystal data listed in Table 1. Small differences account for the homogeneity ranges.

Small prismatic crystal splinters were selected from the crushed $\text{Sc}_{50}\text{T}_{13}\text{In}_3$ samples and glued to glass fibers with bees wax. The $\text{Sc}_{49.59}\text{Ru}_{13}\text{In}_{3.41}$ crystal is shown as an example in Fig. 5. For a first quality check, Laue patterns were collected on a Buerger precession camera (white Mo

radiation, Fuji-film imaging plate). Complete data sets were recorded at room temperature using a Stoe IPDS-II image plate system (graphite monochromatized Mo radiation; $\lambda = 71.073$ pm) in oscillation mode. Numerical absorption corrections were applied to the data sets. Details on the crystallographic data are given in Table 1.

EDX data

The three single crystals were semiquantitatively analyzed by EDX in a Zeiss EVO® MA10 scanning electron microscope (variable pressure mode (60 Pa) and W cathode) using Sc, Ni, Ru, Pd, and InAs as standards. The measurements (76 ± 1 at.% Sc: 19 ± 1 at.% Ni: 5 ± 1 at.% In, for the $\text{Sc}_{50}\text{Ni}_{13.16}\text{In}_{2.84}$, 75 ± 1 at.% Sc: 20 ± 1 at.% Ru: 5 ± 1 at.% In for the $\text{Sc}_{49.59}\text{Ru}_{13}\text{In}_{3.41}$ and 75 ± 1 at.% Sc: 21 ± 1 at.% Pd: 4 ± 1 at.% In for the $\text{Sc}_{50}\text{Pd}_{13.65}\text{In}_{2.35}$ crystal) confirmed the compositions refined from the single crystal X-ray data. The standard uncertainties account for the irregular crystal surface. No impurity elements (especially with respect to the tantalum containers) were detected.

Structure refinements

The three data sets showed face-centered cubic lattices with low Laue symmetry and space group $Fm\bar{3}$ was found to be correct in agreement with previous work on the isotypic compounds with $T = \text{Co, Rh, Ir, and Pt}$ [10–12]. The atomic parameters of the platinum compound were taken as starting values and the structures were refined on F^2 with the Jana 2006 [30] software package using anisotropic displacement parameters for all atoms. Since all representatives of this structure type show small homogeneity ranges, the occupancy parameters of all sites were refined in separate series of least-squares cycles to check the correct compositions. Each crystal showed one mixed occupied site. These site occupancies were refined as least-squares variables (the

Fig. 4 Experimental (bottom) and calculated (top) X-ray powder pattern ($\text{CuK}\alpha_1$ radiation) of $\text{Sc}_{50}\text{Pd}_{13}\text{In}_3$

Table 1 Crystallographic data and structure refinement details for $\text{Sc}_{50}\text{T}_{13}\text{In}_3$; $\text{Ag}_{7+x}\text{Mg}_{26-x}$ type, space group $Fm\bar{3}$, $Z=4$

Empirical formula	$\text{Sc}_{50}\text{Ni}_{13.16(1)}\text{In}_{2.84(1)}$	$\text{Sc}_{49.59(1)}\text{Ru}_{13}\text{In}_{3.41(1)}$	$\text{Sc}_{50}\text{Pd}_{13.65(2)}\text{In}_{2.35(2)}$
Molar mass/g mol ⁻¹	3346.1	3935.3	3970
Lattice parameters/pm	$a = 1765.16(9)$	$a = 1772.40(6)$	$a = 1784.46(6)$
Cell volume/nm ³	5.4999	5.5678	5.6823
Calculated density/g cm ⁻³	4.04	4.69	4.64
Crystal size/ μm^3	$15 \times 20 \times 40$	$70 \times 70 \times 165$	$30 \times 60 \times 100$
Diffraction type	IPDS-II (Stoe)	IPDS-II (Stoe)	IPDS-II (Stoe)
Detector distance/mm	70	70	70
Exposure time/min	30	5	5
ω -Range; step width/deg	0–180/1.0	0–180/1.0	0–180/1.0
Integr. Param. A, B, EMS	14.0, – 1.0, 0.030	14.0, – 1.0, 0.030	14.0, – 1.0, 0.030
Absorption coefficient/mm ⁻¹	11.2	10.3	10.6
$F(000)/e$	6230	7122	7172
θ Range/ $^\circ$	2.31–33.41	2.30–33.43	2.28–33.77
hkl range	$\pm 27, \pm 27, \pm 27$	$\pm 27, \pm 27, \pm 27$	$\pm 27, \pm 27, \pm 27$
Total no. reflections	46,460	20,114	36,097
Independent reflections/ R_{int}	986/0.2463	991/0.0427	1013/0.1075
Refl. with $I \geq 3\sigma(I)/R_\sigma$	559/0.0558	832/0.0108	774/0.0239
Data/parameters	986/34	991/34	1013/34
Goodness-of-fit on F^2	0.99	1.08	1.02
$R1/wR2$ for $I \geq 3\sigma(I)$	0.0294/0.0489	0.0146/0.0314	0.0169/0.0310
$R1/wR2$ for all data	0.0838/0.0606	0.0236/0.0337	0.0349/0.0402
Extinction coefficient	–	2050(90)	1010(70)
Largest diff. peak/hole/e $\text{\AA}^{-3}$	2.30/–2.13	0.81/–0.98	1.30/–1.54

Fig. 5 Scanning electron micrograph of the $\text{Sc}_{49.6}\text{Ru}_{13}\text{In}_{3.4}$ single crystal (ca. $70 \times 70 \times 165 \mu\text{m}^3$) mounted on a quartz fiber

enhanced standard deviation for the palladium-based crystal is a consequence of the close scattering power of palladium and indium, which differ by three electrons only). The final difference Fourier syntheses were flat. The final atomic coordinates, displacement parameters, and interatomic distances

(exemplarily for the palladium compound) are listed in Tables 2 and 3.

CCDC–2015860 ($\text{Sc}_{50}\text{Ni}_{13.16}\text{In}_{2.84}$), CCDC–2015859 ($\text{Sc}_{49.59}\text{Ru}_{13}\text{In}_{3.41}$), and CCDC–2015828 ($\text{Sc}_{50}\text{Pd}_{13.65}\text{In}_{2.35}$) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Magnetic characterization

The $\text{Sc}_{50}\text{Ni}_{13.5}\text{In}_{2.5}$ and $\text{Sc}_{50}\text{Pd}_{13.5}\text{In}_{2.5}$ samples were investigated using the Vibrating Sample Magnetometer (VSM) option of a Physical Property Measurement System (PPMS) by Quantum Design. The samples were ground to a fine powder and each was filled into a polypropylene capsule, which was then inserted into a brass sample holder, which was attached to the sample holder rod of the VSM. The measurements were conducted from 2.5 to 300 K and a field of 10 kOe.

Table 2 Atomic coordinates and displacement parameters [pm²] for Sc₅₀Ti₁₃In₃ (Ag_{7+x}Mg_{26-x} type, space group $Fm\bar{3}$, $Z=4$)

Atom	Wyck	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃	<i>U</i> _{eq}
Sc ₅₀ Ni _{13.16} In _{2.84}											
Sc1	96 <i>i</i>	0.09297(4)	0.25549(5)	0.15820(4)	116(3)	106(3)	127(3)	−15(3)	−19(3)	7(3)	116(2)
Sc2	48 <i>h</i>	0	0.09396(6)	0.35329(6)	86(4)	89(4)	115(5)	0	0	−15(4)	97(3)
Sc3	32 <i>f</i>	0.09018(4)	<i>x</i>	<i>x</i>	111(2)	<i>U</i> ₁₁	<i>U</i> ₁₁	−6(3)	<i>U</i> ₁₂	<i>U</i> ₁₂	111(1)
Sc4	24 <i>e</i>	0.21306(9)	0	0	99(6)	106(6)	135(7)	0	0	0	113(4)
Ni1	48 <i>h</i>	0	0.15263(5)	0.21650(5)	179(4)	136(4)	190(4)	0	0	64(3)	168(2)
0.84(2)In1/ 0.16(2)Ni2											
4 <i>b</i>	1/2	1/2	1/2	1/2	97(5)	<i>U</i> ₁₁	<i>U</i> ₁₁	0	0	0	97(3)
Ni3	4 <i>a</i>	0	0	0	100(5)	<i>U</i> ₁₁	<i>U</i> ₁₁	0	0	0	100(3)
In2	8 <i>c</i>	1/4	1/4	1/4	91(2)	<i>U</i> ₁₁	<i>U</i> ₁₁	0	0	0	91(1)
Sc _{49.59} Ru ₁₃ In _{3.41}											
Sc1	96 <i>i</i>	0.09393(2)	0.25607(2)	0.15569(2)	120(1)	94(1)	144(1)	−8(1)	−26(1)	10(1)	119(1)
Sc2	48 <i>h</i>	0	0.09353(3)	0.35444(3)	83(2)	86(2)	94(2)	0	0	−9(1)	87(1)
0.95(1)Sc3/ 0.05(1)In1											
32 <i>f</i>	0.08934(2)	<i>x</i>	<i>x</i>	<i>x</i>	88(2)	<i>U</i> ₁₁	<i>U</i> ₁₁	−4(1)	<i>U</i> ₁₂	<i>U</i> ₁₂	88(1)
Sc4	24 <i>e</i>	0.21226(4)	0	0	87(2)	127(3)	85(2)	0	0	0	100(1)
Ru1	48 <i>h</i>	0	0.15478(1)	0.21652(1)	169(1)	111(1)	163(1)	0	0	57(1)	147(1)
In2	4 <i>b</i>	1/2	1/2	1/2	89(1)	<i>U</i> ₁₁	<i>U</i> ₁₁	0	0	0	89(1)
Ru2	4 <i>a</i>	0	0	0	82(1)	<i>U</i> ₁₁	<i>U</i> ₁₁	0	0	0	82(1)
In3	8 <i>c</i>	1/4	1/4	1/4	100(1)	<i>U</i> ₁₁	<i>U</i> ₁₁	0	0	0	100(1)
Sc ₅₀ Pd _{13.65} In _{2.35}											
Sc1	96 <i>i</i>	0.09404(3)	0.25738(3)	0.15738(3)	107(2)	93(2)	107(2)	−13(2)	−2(2)	4(2)	102(1)
Sc2	48 <i>h</i>	0	0.09209(4)	0.35719(4)	78(3)	92(3)	96(3)	0	0	−15(2)	89(2)
Sc3	32 <i>f</i>	0.09119(3)	<i>x</i>	<i>x</i>	91(1)	<i>U</i> ₁₁	<i>U</i> ₁₁	1(2)	<i>U</i> ₁₂	<i>U</i> ₁₂	91(1)
Sc4	24 <i>e</i>	0.21372(5)	0	0	81(4)	91(4)	98(4)	0	0	0	90(2)
Pd1	48 <i>h</i>	0	0.15508(2)	0.21935(2)	122(1)	99(1)	136(1)	0	0	37(1)	119(1)
0.65(7)Pd2/ 0.35(7)In1											
4 <i>b</i>	1/2	1/2	1/2	1/2	84(3)	<i>U</i> ₁₁	<i>U</i> ₁₁	0	0	0	84(2)
Pd3	4 <i>a</i>	0	0	0	99(2)	<i>U</i> ₁₁	<i>U</i> ₁₁	0	0	0	99(1)
In2	8 <i>c</i>	1/4	1/4	1/4	92(1)	<i>U</i> ₁₁	<i>U</i> ₁₁	0	0	0	92(1)

The equivalent isotropic displacement parameter U_{eq} is defined as $U_{eq} = 1/3 (U_{11} + U_{22} + U_{33})$ (pm²). Coefficients U_{ij} of the anisotropic displacement factor tensor of the atoms are defined by: $-2\pi^2[(ha^*)^2U_{11} + \dots + 2hka^*b^*U_{12}]$. Standard deviations are given in parentheses

Table 3 Interatomic distances (pm) for $\text{Sc}_{50}\text{Pd}_{13.65}\text{In}_{2.35}$

Sc1:	1	Pd1	271.5	Sc3:	1	Pd3	281.8
	1	Pd1	308.8		3	Pd1	302.9
	1	Pd1	317.7		3	Sc4	317.5
	1	Sc2	317.8		3	Sc1	319.3
	1	Sc3	319.3		3	Sc3	325.4
	1	In2	323.9	Sc4:	2	Pd1	276.9
	2	Sc1	325.9		2	Sc2	304.2
	1	Sc1	331.5		4	Sc3	317.5
	1	Sc2	332.6		4	Sc1	336.4
	1	Sc1	335.6	Pd1:	1	Sc2	270.4
	1	Sc4	336.4		2	Sc1	271.5
	2	Sc1	359.9		1	Sc4	276.9
Sc2:	1	Pd1	270.4		2	Sc3	302.9
	1	Pd2/In1	303.3		2	Sc1	308.8
	1	Sc4	304.1		2	Sc1	317.7
	4	Sc2	316.5	Pd2/In1:	12	Sc2	303.2
	2	Sc1	317.8		8	Sc3	281.9
	1	Sc2	328.7	In2:	12	Sc1	323.9
	2	Sc1	332.6				

Standard deviations are equal or smaller than 0.1 pm

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