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NEW PHASE Ho₃(Ag_{0.50-0.52}Al_{0.50-0.48})₁₄Ag_x (x = 0.67 – 0.77): CRYSTAL STRUCTURE AND MAGNETIC PROPERTIES Ho₃Ag_{7.67}Al_{7.01} AND Ho₃Ag_{8.10}Al_{6.76}

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The crystal structure of the Ho₃(Ag_{0.50-0.52}Al_{0.50-0.48})₁₄Ag_x compounds (x = 0.67–0.77) has been investigated by means of X-Ray analysis. It adopts DyAg_{2.4}Al_{2.6}-type of structure and crystallizes in the space group $P6_3/mmc$. Magnetic properties of two synthesised compounds were investigated revealing paramagnetic behaviour in whole investigated temperature range.

Keywords: Crystal structure, homogeneity range, partial occupation, coordination polyhedra, magnetic properties.

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1. Introduction

While investigating the phase equilibria diagram of the Ho–Ag–Al system [1], the existence of the ternary Ho(Ag_{0.5}Al_{0.5})₅ compound [2] has been confirmed (DyAg_{2.4}Al_{2.6}-type of structure [3], SG $P6_3/mmc$). This compound has insignificant homogeneity range with composition ~Ho₁₇Ag₄₀₋₄₅Al₄₃₋₃₈. Isostructural compounds are known in related *RE*–Ag–Al systems (*RE* = Y, Pr, Nd, Sm, Gd–Lu) [4]. However crystal structures have been investigated only for the following compounds: YAg_{2.5}Al_{2.7} [5], NdAg_{3.26}Al_{1.89} [6], SmAg_{2.6}Al_{2.5} [4], GdAg_{2.3}Al_{2.7} [7], and TbAg_{2.7}Al_{2.5} [8]. All compounds are characterized by insignificant homogeneity ranges, caused by the statistical distribution of smaller atoms (Ag and Al) and partial occupation of several crystallographic sites by Ag atoms [3-11]. A similar type of distribution of smaller atoms (Ag and Al) is observed in other ternary compounds of RE–Ag-Al systems. Partial occupation of crystallographic positions with Ag and/or Al atoms is characteristic of ternary aluminides with Th₂Ni₁₇- [12] and Yb₈Cu₁₇Al₄₉-type of structure [13].

2. Materials and Methods

Samples were synthesized by arc melting of the mixtures of initial components using metals of the following purity (wt. %): Ho - 99.7, Ag - 99.9, and Al - 99.95, which was followed by annealing of the alloys in sealed evacuated quartz ampoules at 870 K for 500 h.

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Phase analysis and crystal structure investigation were performed by means of X-Ray diffraction methods using DRON-3M diffractometer with Cu K α radiation. All calculations were performed using WinCSD software [14].

Composition of the compounds was confirmed by means of electron microscopy analysis using scanning electron microscope REMMA 102-02 (binary compounds HoAg and HoAl₂ were used as standard samples).

Quantum Design PPMS equipment was used for magnetic studies and heat-capacity measurements. Magnetic measurements were performed in the temperature range 2–300 K and fields up to 14 T. The grains of the sample for magnetic measurement were fixed in random orientation by acetone-soluble glue.

3. Results and discussion

Crystal structure determination for the samples with the starting compositions of $Ho_{17}Ag_{42}Al_{41}$ and $Ho_{17}Ag_{45}Al_{38}$ using the $DyAg_{2.4}Al_{2.6}$ (SG $P6_3/mmc$) model of structure confirmed that these compounds are isostructural. Conditions and results of the X-Ray investigations are shown in Table 1.

Table 1

Crystallographic data and results of the structure refinement of the Ho₃Ag_{7.67}Al_{7.01} and Ho₃Ag_{8.10}Al_{6.76} compounds

Compounds	H03Ag7.67Al7.01	H03Ag8.10Al6.76		
Structure type	DyAg _{2.4} Al _{2.6}			
Space group	P63/	mmc		
Lattice parameters, nm	a = 0.91470(3)	a = 0.91728(3)		
	c = 0.93860(4)	c = 0.93302(3)		
Cell volume, nm ³	0.68009(7)	0.67988(6)		
Number of atoms in cell	35.3	35.6		
Calculated density, g/cm ³	7.3750(7) 7.5634(7)			
Radiation and wavelength, nm	Cu K α , $\lambda = 0.154185$			
Diffractometer	Powder, DRON-3M			
Mode of refinement	Full profile			
Number of atom sites	8			
$2\theta_{\rm max}$ and $\sin \theta_{\rm max}/\lambda$	128.5, 0.584			
Scale factor	0.6019(2)	0.49804(8)		
RI, RP	0.068, 0.150	0.083, 0.158		

Crystal structure of the compounds is characterized by statistical distribution of Ag and Al atoms in the crystallographic positions and also by the presence of partially occupied sites 2(a), 2(b), and 4(e) (Tables 2, 3). Ultimate calculations lead to the compounds compositions as follows: Ho₃(Ag_{0.50}Al_{0.50})₁₄Ag_xAg_yAg_z (x = 0.09, y = 0.31, z = 0.27) and Ho₃(Ag_{0.52}Al_{0.48})₁₄Ag_xAg_yAg_z (x = 0.09, y = 0.35, z = 0.33), or Ho₃Ag_{7.67}Al_{7.01} and Ho₃Ag_{8.10}Al_{6.76}, respectively.

Ho atoms are located in the centers of 16-vertices polyhedra with *mm* symmetry (Table 4). The coordination polyhedra of X4–X6 atoms are the icosahedra, which differ from each other by their vertices occupation order and, as a result, by the type of deformation. X7 atoms are located in the centers of 12-vertices polyhedra with one apex alternatively filled by the atom X1 or X2, or one of two X3 atoms. The X1 atoms are located in trigonal prisms with 60° "twisted" parallel triangular sides formed by X7 atoms ($\delta_{X1-X7} = 0.2633(2)$ nm). This polyhedron can also be interpreted as tetragonal bipyramid. The distances to six Ho atoms which are centered the triangular sides are much larger ($\delta_{X1-H0} = 0.3903(1)$ nm).

Table 2

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Atomic	coordinates	and isot	ropic (displacement	parameters 1	for Ho3Ag7.67Al7.01
			1	1	1	0

Atoms	WD	Coordinates			$B_{iso}, \bullet 10^2$
Atoms	VV F	x	у	z	nm ²
Ho(6Ho)	6(<i>h</i>)	0.1968(2)	2x	1/4	1.24(1)
X1(0.18(2) Ag)	2(<i>a</i>)	0	0	0	1.02(2)
X2(0.62(2) Ag)	2(<i>b</i>)	0	0	1/4	0.99(2)
X3(0.53(3) Ag)	4(<i>e</i>)	0	0	0.318(3)	0.98(2)
X4(0.17(4) Ag+3.83(4) Al)	4(<i>f</i>)	1/3	2/3	0.0152(14)	0.91(2)
X5(3.61(5) Ag+2.39(5) Al)	6(<i>g</i>)	1/2	0	0	1.21(2)
X6(1.19(5) Ag+4.81(5) Al)	6(<i>h</i>)	0.5677(5)	2x	1/4	1.13(2)
X7(9.02(7) Ag+2.98(7) Al)	12(<i>k</i>)	0.8432(2)	2x	0.0929(3)	1.17(1)

Table 3

Atomic coordinates and isotropic displacement parameters for Ho₃Ag_{8.10}Al_{6.76}

Atoms	WD	Coordinates			$B_{iso}, \bullet 10^2$
Atoms	VV F	x	у	z	nm^2
Ho(6Ho)	6(<i>h</i>)	0.1976(2)	2x	1/4	1.02(1)
X1(0.18(2) Ag)	2(a)	0	0	0	1.00(1)
X2(0.70(2) Ag)	2(<i>b</i>)	0	0	1/4	1.07(1)
X3(0.66(3) Ag)	4(e)	0	0	0.336(3)	1.02(1)
X4(0.52(4) Ag+3.48(4) Al)	4(<i>f</i>)	1/3	2/3	0.0131(12)	0.95((1)
X5(2.76(5) Ag+3.24(5) Al)	6(<i>g</i>)	1/2	0	0	1.09(1)
X6(1.54(5) Ag+4.46(5) Al)	6(<i>h</i>)	0.5690(4)	2x	1/4	1.041)
X7(9.82(7) Ag+2.18(7) Al)	12(<i>k</i>)	0.8434(2)	2x	0.0903(3)	1.02(1)

The X2 and X3 atoms are located in trigonal prisms formed by X7 atoms. Tetragonal sides of the prisms are centered by three Ho atoms. Each of such prisms can be alternatively centered by one X2 atom or one of two X3 atoms. Thus, the peculiarity of the investigated structure is the existence of the void channels, located along **0Z** axis (x = 0, y = 0), successively formed by two types of trigonal prisms of X7 atoms. These prisms are connected together by their triangular sides. The unit cell contains one channel, formed by two prisms of X1 atoms and two prisms of X2 (or X3) atoms.

The structures closely related to the $DyAg_{2.4}Al_{2.6}$ type [3] are the EuMg₅ [15] and Sc₃Ni₁₁Si₄ [17] structure types with the different type of the **0Z** channel occupation by the smaller atoms (Table 5). The authors of Refs. [15–16] repeatedly investigated the crystal structure of the ~EuMg₅ compound. It was found that this compound has insignificant homogeneity range, composition of which can be described as Eu₃Mg₁₄Mg_x (1≤ x ≤1.7). In this structure Mg-atoms occupy the **0Z** channels in the same manner as Ag- atoms in the structure of DyAg_{2.4}Al_{2.6}.

The temperature dependence of magnetic susceptibility of the both Ho₃Ag_{7.67}Al_{7.01} and Ho₃Ag_{8.10}Al_{6.76} compounds exhibits the Curie-Weiss behavior with parameters $\mu_{eff} = 10.8$ μ_B/Ho , $\theta_p = 0.2$ K (for Ho₃Ag_{7.67}Al_{7.01}) and $\mu_{eff} = 10.8$ μ_B/Ho , $\theta_p = -1.5$ K (for Ho₃Ag_{8.10}Al_{6.76}). The effective moment close to the theoretical value 10.61 μ_B/Ho indicates that both compounds are paramagnetic down to low temperatures. The deviation of susceptibility in 3 and 6 T (Figs. 1–2), which is apparent below T = 50 K, is a natural consequence of Brillouin-type saturation of magnetization related to large Ho moments.

Table 4

	TT 4 4 1	TT 4 41	1 2000 000 1
Atoms	H03Ag7.67Al7.01	H03Ag8.10Al6.76	Coordinational polyhedra
$\mathbf{U}_{2} = 2\mathbf{V}_{1}$	0, 1111	0,1111	
$\Pi 0 = 2\Lambda 4$	0.3087(10) 0.2118(2)	0.3088(8) 0.2120(2)	
$-1\lambda 2$	0.3110(2) 0.2184(5)	0.3139(2) 0.2241(6)	
$-2\Lambda 3$	0.3164(3) 0.2212(2)	0.3241(0) 0.2222(6)	THAN AV
$-4\lambda/$	0.3213(2) 0.2221(5)	0.3233(0) 0.2255(4)	
-2X0	0.3231(5) 0.3280(2)	0.3255(4) 0.2241(2)	
$-2\lambda/$	0.3280(3)	0.3241(3) 0.2275(1)	
-4X5	0.3383(1)	0.3375(1)	U U
- 2Ho	0.3746(2)	0.3736(2)	[Ho 2X ₄ 1X ₂ 2X ₃ 4X ₇ 2X ₆ 2X ₇ 4X ₅ 2Ho]
X1 _*X3	0.170(2)	0.153(2)	
-2*X2	0.23465(1)	0.23326(1)	
- 6X7	0.2633(2)	0.2626(2)	
-2*X3	0.299(2)	0.314(2)	TOL
- 6*Ho	0.3903(1)	0.3911(1)	
0 110	010700(1)	010)11(1)	
			$[X_{1}6X_{7}]$
X2 - 2*X3	0.064(2)	0.081(2)	
-2X*1	0.23465(1)	0.23326(1)	
- 6X7	0.2890(2)	0.2900(2)	
- 3Ho	0.3118(2)	0.3139(2)	
W 2 1* W 2	0.0(4(2))	0.001(2)	[X ₂ 0X ₇ 3H0]
$X_3 = 1^*X_2$	0.064(2)	0.081(2)	
-1*X3	0.128(3)	0.161(3) 0.152(2)	
-1^*X1	0.170(2)	0.153(2)	
-3X/	0.2621(8)	0.2580(6)	
-1*X1	0.299(2)	0.314(2)	
- 3H0	0.3184(5)	0.3241(6)	
-3X/	0.3264(15)	0.339(2)	
			[X ₃ 3X ₇ 3Ho3X ₇]
X4 – 3X5	0.26444(8)	0.26508(6)	
- 3X6	0.2942(12)	0.2904(10)	
- 3X7	0.2975(5)	0.2969(4)	
– 3Ho	0.3087(10)	0.3088(8)	
			[X43X53X63X73H0]

Interatomic distances in $\mathrm{Ho_3Ag_{7.67}Al_{7.01}}$ and $\mathrm{Ho_3Ag_{8.10}Al_{6.76}}$ compounds

			Continued the table	
Atoms	H03Ag7.67Al7.01	H03Ag8.10Al6.76	Coordinational polyhedra	
Atoms	σ, nm	σ, nm	Coordinational polynedia	
X5 – 2X6	0.2580(2)	0.2577(2)		
- 2X4	0.26444(8)	0.26508(6)		
-4X7	0.2858(2)	0.2859(2)		
- 4Ho	0.3383(1)	0.3375(1)		
			[X 52X62X44X74H0]	
X6 – 2X5	0.2580(2)	0.2577(2)		
-2X6	0.2714(6)	0.2688(6)		
-4X7	0.2840(4)	0.2856(4)		
-2X4	0.2942(12)	0.2904(10)		
- 2Ho	0.3231(5)	0.3255(4)		
			[X 62X52X64X74H0]	
X7 – 1X3	0.2621(8)	0.2580(6)		
-1X1	0.2633(2)	0.2626(2)		
-2X6	0.2840(4)	0.2856(4)		
- 2X5	0.2858(2)	0.2859(2)		
-1X2	0.2890(2)	0.2900(2)		
-1X7	0.2949(4)	0.2981(4)		
-1X4	0.2975(5)	0.2969(4)		
-2X7	0.3036(3)	0.3004(3)	-	
- 2Ho	0.3213(2)	0.3233(2)		
- 1*X3	0.3264(15)	0.339(2)	[X ₇ 1X ₃ 1X ₁ 2X ₆ 2X ₅ 1X ₂ 1X ₇ 1X ₄ 2X ₇ 2Ho]	
- 1Ho	0.3280(3)	0.3241(3)		

* Alternative positions are shown which mutually exclude the possibility of simultaneous occupation by the atoms in one unit cell



Fig. 1 Temperature dependence of magnetic susceptibility of $Ho_3Ag_{7.67}Al_{7.01}$ and $Ho_3Ag_{8.10}Al_{6.76}$ measured in $\mu_0 H = 3$ and 6 T. The inset shows low temperature part of temperature dependence of magnetic susceptibility measured in various magnetic fields

As strong magnetic fields can modify the magnetic state, fingerprints of magnetic ordering have to be traced out in low fields. Ho₃Ag_{7.67}Al_{7.01} exhibits a cusp in the temperature range 25–30 K (Fig. 1), which indicates an anti-ferromagnetic order. But the fact that the cusp is superimposed on still increasing slope of $\chi(T)$ is suggestive of extrinsic (impurity) origin of this

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anomaly, which is progressively suppressed in magnetic field. The other reason is that there is no corresponding anomaly in the temperature dependence of heat capacity (Fig. 2). In the case of Ho there is a high entropy released at the magnetic phase transition (due to J = 8), such transition would have to be reflected in C(T). At low temperatures there is a high contribution of nuclear specific heat for Ho, and the fast increase of C/T vs. T masks possible magnetic entropy released in the T-range below 10 K.



Fig. 2 Temperature dependence of specific heat of Ho₃Ag_{7.67}Al_{7.01} (in C/T vs. T^2 representation) and Ho₃Ag_{8.10}Al_{6.76} (in C/T vs. T^2 representation). Low temperature part of specific heat of Ho₃Ag_{8.10}Al_{6.76} measured in various magnetic fields

Table 5

Peculiarities of the EuMg₅ [13], Sc₃Ni₁₁Si₄ [15] and DyAg_{2.4}Al_{2.6} [3] types of structure

WP	EuMg ₅	Sc ₃ Ni ₁₁ Si ₄	DyAg _{2.4} Al _{2.6}
6h(x 2x 1/4)	6Eu, $x = 0.1951$	6Sc, x = 0.1920	6Dy, <i>x</i> = 0.19538
4f(1/3 2/3 z)	4Mg, <i>z</i> =0.0031	4Ni, <i>z</i> = 0.0086	$4X^*1, z = 0.0131$
6g (1/2 0 0)	6Mg	6Si	6X2
6h(x 2x 1/4)	6Mg, x = 0.8385	6Ni, <i>x</i> = 0,5618	6X3, <i>x</i> = 0,5679
12k(x 2x z)	12Mg, x = 0,8385,	12Ni, x = 0,8386,	12X4, x = 0,8426,
	z = 0,0900	z = 0,0857	z = 0,092
2a (0 0 0)	2Mg	-	1.08Al
2b (0 0 1/4)	—	2Si	0.52Ag
4 <i>e</i> (0 0 <i>z</i>)	-	-	0.67Ag, <i>z</i> = 0.298

 X^* – statistical mixtures of the Ag and Al atoms.

Ho₃Ag_{8.10}Al_{6.76} has the spurious anomaly at 25–30 K much weaker, but still discernible. Besides that there is a rounded maximum at 5–6 K (Fig. 2). This is most likely intrinsic, reflecting magnetic order round 5 K. In this case there is magnetic entropy below 5 K, which is removed by applied magnetic field. The nuclear specific heat itself cannot be affected by fields of several Tesla at all (Fig. 2).

4. Conclusions

The crystal structure calculations for the samples with composition of $Ho_{17}Ag_{43}Al_{40}$ and $Ho_{17}Ag_{45}Al_{38}$ using the DyAg_{2.4}Al_{2.6} (SG *P*6₃/*mmc*) model of structure confirmed that these compounds are isostructural. Magnetic susceptibility and heat capacity measurements reveal paramagnetic behaviour for both compounds.

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НОВА ФАЗА Но3(Ад0.50–0.52Al0.50–0.48)14Адх (х = 0.67–0.77): КРИСТАЛІЧНА СТРУКТУРА ТА МАГНІТНІ ВЛАСТИВОСТІ Но3Ад7.67Аl7.01 ТА Но3Ад8.10Al6.76

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Методами рентгеноструктурного та рентгеноспектрального аналізів досліджено кристалічну структуру двох сполук у межах твердого розчину фази Ho₃(Ag_{0,50-0,52}Al_{0,50-0,48})₁₄Ag_x (x = 0,67–0,77): a = 0,91470(3), c = 0,93860(4) нм для складу Ho₃Ag_{7,67}Al_{7,01}, a = 0,91728(3), c = 0,93302(3) нм для складу Ho₃Ag_{8,10}Al_{6,76}. Сполуки належать до структурного типу DyAg_{2,4}Al_{2,6}, просторова група *P*6₃/*mmc*. Синтез проводили методом електродугового сплавляння шихти вихідних компонентів в атмосфері очищеного аргону з наступним гомогенізувальним відпалюванням при 870 К протягом 500 год. у вакуумованих кварцових ампулах. Рентгенофазовий та рентгеноструктурний аналізи проводили за рентгенограмами порошку, отриманими на автоматичному дифрактометрі ДРОН-3М (Си К α випромінювання, Ni β фільтр, режим крокової реєстрації інтенсивностей дифракційної картини, $\Delta \theta = 0,05$ град, час сканування — 20 с в кожній точці). Кількісний склад досліджених зразків додатково підтверджено методом локального рентгеноспектрального аналізу (електронний мікроскоп РЕММА 102-02, як стандарт використали бінарні сполуки НоАg і HoAl₂). Результати рентгенофазового і рентгеноспектрального аналізу задовільно корелюють. Поміри магнітних властивостей та температурної теплоємкості проводили на магнетометрі Quantum Design PPMS.

Кристалічна структура досліджених сполук є близькоспорідненою до структурних типів EuMg5 і Sc₃Ni₁₁Si4 і характеризується частковим заповненям кристалографічних позицій 2*a* $(0\,0\,0), 2b$ $(0\,0\,1/4)$ і 4e $(0\,0z)$ атомами аргентуму, які заповнюють тригонально- та тетрагональнопризматичні пустоти, розташовані вздовж каналу 0*Z*.

Дослідження магнітних властивостей вказують про парамагнітний характер обох сполук у дослідженій області температур з ефективними магнітними моментами $\mu_{eff} = 10.8 \ \mu$ в/Но (для Ho₃Ag_{7.67}Al_{7.01}) і $\mu_{eff} = 10.8 \ \mu$ в/Но (для Ho₃Ag_{8.10}Al_{6.76}), близькими до теоретичного значення 10.61 μ в/Но.

Ключові слова: Кристалічна структура, область гомогенності, часткове заповнення, координаційний многогранник, магнітні властивості.

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