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THE Ho–Zn–Al SYSTEM AT 500 °C

O. Stelmakhovych¹, T. Krachan², B. Stelmakhovych¹

¹ Ivan Franko National University of Lviv,
Kyryla and Mefodiya Str., 6, 79005 Lviv, Ukraine;

² State Agrarian and Engineering University in Podillya,
Shevchenka Str., 13, 32300 Kamyanets-Podilskyy, Ukraine
e-mail: bstelmakhovych@gmail.com

Isothermal section of the phase diagram of the Ho–Zn–Al system at 500 °C in the range up to 50 at. % of Ho has been built by means of X-ray analysis. Existence of two earlier known ternary intermetallic compounds HoZn_5Al_3 (HoZn_5Al_3 -type structure, space group $I4/mmm$) and $\text{Ho}_3\text{Zn}_{4.4}\text{Al}_{6.6}$ ($\text{La}_3\text{Al}_{11}$ -type structure, space group $Immm$) has been confirmed. New ternary phases with AuCu_3 and CaIn_2 types of structure have been found for the first time and their crystal structures have been investigated. Limit compositions of the solid solutions on the basis of the binary compounds and homogeneity ranges for the ternary compounds have been determined.

Key words: intermetallic compound, crystal structure, phase equilibria.

Phase diagrams of the R -Zn–Al systems (R – rare earth metal) were constructed for the systems with $R = \text{La}$ [1], Ce [2], Pr [3], Eu [4], Nd [5], and, partially, with Yb [6]. Other systems were investigated only with the purpose of compounds with certain type of structure [7, 8, 9]. Ternary compounds which are formed in these systems, adopt 14 types of structure. According to classification by Krypyakevych these types belong to four classes: *ic* – icosahedric, *h* – hexahedric, *tp* – trigonal-prismatic, *ta* – tetragonal-antiprismatic (Table 1). Crystal structures of compounds in the R -Zn–Al systems are characterized by the statistical distribution of the small size atoms in the crystallographic positions, and, thus, by formation of the ternary compounds with significant homogeneity ranges. Binary compounds in the R -Zn and R -Al systems solve the third component, and form the substitutional solid solutions.

Existence of the two ternary compounds, namely, $\text{Ho}_3\text{Zn}_{4.4}\text{Al}_{6.6}$ ($\text{La}_3\text{Al}_{11}$ -type structure, space group $Immm$ (N 71), $oI28.0$, $a = 0.42107$, $b = 1.23881$, $c = 0.99514$ nm) [8], and HoZn_5Al_3 (HoZn_5Al_3 -type structure, space group $I4/mmm$ (N 139), $tI72.0$, $a = 0.8586$, $c = 1.6538$ nm) [9] has been reported in the Ho–Zn–Al system.

The main goals of our investigation are the construction of the isothermal section of the Ho–Zn–Al system phase diagram at 500 °C, searching for the new ternary compounds, determination of limit compositions of solid solutions on the basis of binary compounds and homogeneity ranges of ternary ones.

For the samples preparation the powders of metallic Ho, Zn and Al with the stated purity of 99.5, 99.9 and 99.99 mas. %, respectively, were used. Weighted components were mixed together, pressed into tablets, placed into alumina crucibles and sealed in silica ampoules under argon atmosphere. They were heated up to 600 °C with a rate of 10–20 °/min and held at this temperature for 24 hours. Then sintered samples were pulverized, carefully mixed, and pressed again with subsequent annealing in silica tubes at 500 °C

within 200 hours. For the samples with Ho content over 30 at. % the third homogenizing annealing during 200 hours was applied. The suggested synthesis method ensures quantitative interaction of the components and their full homogenization. In general 46 samples were synthesized for this investigation.

X-Ray structural analysis was performed using powder diffraction patterns recorded on DRON-3M diffractometer (CuK α radiation). All calculations were performed using WinCSD software [11] with multiple phases refinement method.

Table 1

Structure types (ST) of the ternary phases in R-Zn-Al systems

ST	R	Y	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
^{ic} SmZn ₁₁															+	
^{ic} Th ₂ Zn ₁₇								+								
^h Yb ₈ Cu ₁₇ Al ₄₉									+	+					+	
^{ic} Ce ₂ Zn ₂₂								+							+	
^h HoZn ₅ Al ₃		+									+	+	+	+		+
^{ic} Yb _{6,4} Zn _{46,8} Al _{3,4}															+	
^{ic} Yb _{12,4} Zn _{96,8} Al _{4,4}															+	
^{aa} BaAl ₄			+	+	+	+	+	+	+	+					+	
^{aa} La ₃ Al ₁₁		+							+	+	+	+	+	+	+	
^{ic} CeNi ₃															+	
^{ic} PuNi ₃															+	
^{ap} CaIn ₂															+	
^{ic} MgNi ₂															+	
^{ic} MgZn ₂															+	

As a result of our investigation the existence of several binary phases was confirmed (Figure 1) in the Ho-Zn-Al system. The HoAl₃ compound crystallizes in own structure type (space group *R-3m*, *a* = 0.60535(9), *c* = 3.5814(6) nm). We did not find any noticeable Zn-solubility in this compound. Binary compounds HoZn₁₂, Ho_{2-x}Zn₁₇, Ho₂Zn₁₇, HoZn₂, and HoZn solve up to 8.5, 4.7, 6.8, 5.0 and 24.0 at. % of Al, respectively, Laves phase HoAl₂ solves 10.0 at. % of Zn. Limit compositions of solid solutions and respective lattice parameters are shown in Table 6. Other binary compounds in the Ho-Al and Ho-Zn systems do not solve noticeable amounts of the third component at the temperature of investigation. Limit compositions of the solid solutions were refined by means of X-ray analysis of two-phase samples with the solid solution with the limit composition as the main phase.

Existence of earlier known ternary compounds HoZn₅Al₃ (own type structure, space group *I4/mmm*) and Ho₃Zn_{4,4}Al_{6,6} (La₃Al₁₁-type structure, space group *Immm*) was confirmed in the Ho-Zn-Al system. It was established that the phase Ho₃(Zn,Al)₁₁ has a homogeneity range and its composition can be defined as Ho₃Zn_{3.7-4.7}Al_{7.3-6.3}. Substitution

of the smaller Zn atoms ($r_{\text{Zn}} = 0.1332$ nm) by the Al atoms ($r_{\text{Al}} = 0.1431$ nm) causes increasing of the lattice parameters (Table 2). Lattice parameters for the phase $\text{Ho}(\text{Zn},\text{Al})_8$ were refined from X-ray patterns of the two- and three-phase samples, and are changed from $a = 0.8615(2)$, $c = 1.6352(4)$ nm to $a = 0.85952(8)$, $c = 1.6624(3)$ nm. This in turn causes the change in lattice volumes from $V = 1.2136$ nm³ to $V = 1.2281$ nm³, respectively. The phase composition can be described as $\text{HoZn}_{4.9-5.1}\text{Al}_{3.1-2.9}$.

New compound with the composition $\sim\text{Ho}_{25}\text{Zn}_{10}\text{Al}_{65}$ has been found during the systematic investigation of the components interaction in the Ho-Zn-Al system. This compound is in equilibria with binary compounds HoAl_3 , $\text{HoAl}_{2-x}\text{Zn}_x$ and with ternary phase $\text{Ho}_3(\text{Zn},\text{Al})_{11}$. X-Ray powder diffraction pattern was successfully indexed in the cubic system with lattice parameter $a = 0.4231(2)$ nm which may indicate the possible AuCu_3 -type structure for the compound. This suggestion was confirmed by the results of the crystal structure refinement using powder diffraction data of two-phase sample, contained new compound with AuCu_3 -type as a main phase, and a limit composition of the $\text{HoAl}_{2-x}\text{Zn}_x$ solid solution (MgCu_2 -type structure) as admixture. Experimental details and refined crystallographic data are listed in Table 3. Experimental, calculated and difference diffraction patterns of the $\text{Ho}_{25}\text{Zn}_{17}\text{Al}_{58}$ sample are shown in Fig. 1.

Table 2

Lattice parameters the compound composition*
within the homogeneity range of $\text{Ho}_3(\text{Zn},\text{Al})_{11}$ phase

Composition	Lattice parameters, nm			V, nm ³
	<i>a</i>	<i>b</i>	<i>c</i>	
$\text{Ho}_3\text{Zn}_{3.7}\text{Al}_{7.3}$	0.42033(2)	1.23992(6)	0.99842(5)	0.52036(7)
$\text{Ho}_3\text{Zn}_{4.2}\text{Al}_{6.8}$	0.42040(2)	1.23892(4)	0.99682(8)	0.51920(5)
$\text{Ho}_3\text{Zn}_{4.4}\text{Al}_{6.6}$	0.42051(3)	1.23732(7)	0.99581(6)	0.51775(9)
$\text{Ho}_3\text{Zn}_{4.7}\text{Al}_{6.3}$	0.42058(2)	1.23717(7)	0.99463(5)	0.51753(8)

* Compound compositions were refined from the X-ray structure refinement data.

Table 3

Experimental details and refined crystal data
for the ternary phases in the $\text{Ho}_{25}\text{Zn}_{17}\text{Al}_{58}$ sample

Compound	$\text{HoZn}_{0.54}\text{Al}_{2.46}$	$\text{HoAl}_{1.7}\text{Zn}_{0.3}$
Structure type	AuCu_3	MgCu_2
Space group	$Pm-3m$	$Fd3m$
Lattice parameters, nm	$a = 0.42365(2)$	$a = 0.78119(2)$
Cell volume, nm ³	0.076036(8)	0.47673(4)
Number of atoms in cell	4.0	24.0
$2\theta_{\text{max}} \sin\theta_{\text{max}}/\lambda$		120 5.62
Scale factor	0.18593	0.03034
Phase composition, mas. %	55.4	44.6
R_i, R_p	0.0682 0.2328	0.0843 0.2328

Table 3

Crystallographic data for the $\text{HoZn}_{0.54(6)}\text{Al}_{2.46(6)}$ compound					
Atoms	WP	Atomic coordinates			$B_{\text{iso}} \cdot 10^2, \text{nm}^2$
		<i>x</i>	<i>y</i>	<i>z</i>	
Ho1	1 <i>a</i>	0	0	0	1.44(11)
$X(2.46(6)\text{Al}+0.54(6)\text{Zn})$	3 <i>d</i>	0	1/2	1/2	2.4(2)
Crystallographic data for the $\text{HoAl}_{1.69(3)}\text{Zn}_{0.31(3)}$					
Ho1	8 <i>a</i>	3/8	3/8	3/8	1.05(10)
$X(13.5(2)\text{Al}+2.5(2)\text{Zn})$	16 <i>d</i>	0	0	0	1.5(2)

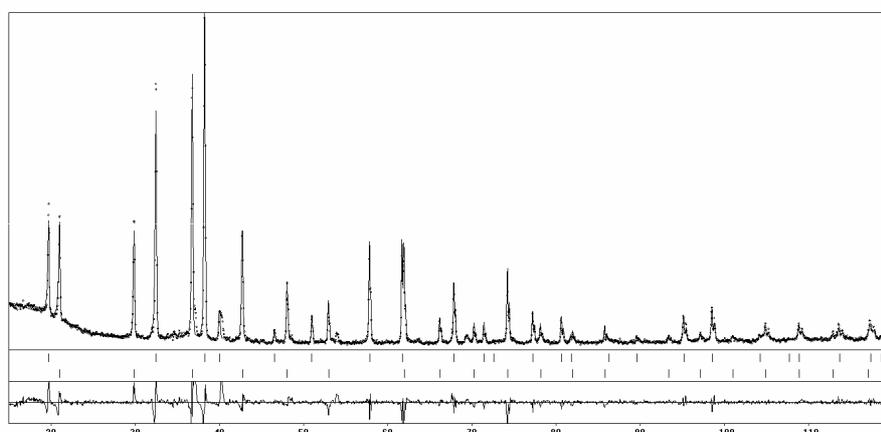


Figure 1. Experimental, calculated and difference diffraction patterns of the two-phase sample with the composition $\text{Ho}_{25}\text{Zn}_{17}\text{Al}_{58}$ (CuK α radiation)

Sample with the starting composition $\text{Ho}_{21}\text{Zn}_{25}\text{Al}_{54}$ contained two phases $\text{Ho}_3\text{Zn}_{4.4}\text{Al}_{6.6}$ ($\text{La}_3\text{Al}_{11}$ -type structure) and $\text{HoZn}_{1.7}\text{Al}_{0.3}$ (AuCu_3 -type structure), atomic parameters in these structures were also refined using powder diffraction data. Experimental details and refined crystallographic data of the compounds are listed in Table 4, experimental, calculated and difference diffraction patterns of the $\text{Ho}_{21}\text{Zn}_{25}\text{Al}_5$ sample are shown in Fig. 2.

The new ternary compound with equimolar composition was found in the sample with the starting composition $\text{Ho}_{33}\text{Zn}_{22}\text{Al}_{45}$. Respective X-ray pattern was indexed in hexagonal system with following lattice parameters: $a = 0.4510(1)$, $c = 0.7077(1)$ nm. Analysis of the literature data allowed us to assume the CaIn_2 -type structure (space group $P6_3/mmc$) for this compound. Results of the crystal structure determination of the new compound are given in Table 5 (refined composition $\text{HoZn}_{0.89}\text{Al}_{1.11}$, CaIn_2 -type structure). Taking into account a homogeneity range the compound composition could be described by the formula $\text{HoZn}_{0.89-1.07}\text{Al}_{1.11-0.93}$. The solid solution $\text{HoAl}_{2-x}\text{Zn}_x$ ($x = 0.3$ for the limit composition) with the MgCu_2 -type structure was the second phase in the $\text{Ho}_{33}\text{Zn}_{22}\text{Al}_{45}$ sample, atomic positional and occupation parameters have been refined too (see Table 5). X-Ray powder diffraction pattern of the $\text{Ho}_{33}\text{Zn}_{22}\text{Al}_{45}$ sample are shown in Fig. 3.

In the Zn-rich part of the Ho-Zn-Al system a new ternary compound with approximate composition $\sim\text{HoZn}_4\text{Al}$ has been found, however its crystal structure has not been established and requires additional investigations.

The isothermal section of the Ho-Zn-Al phase diagram at 500 °C in the region up to 50 at. % of Ho (Fig. 4) has been constructed as a result of our investigation. Crystallographic data of the ternary phases, which are formed in the system, are summarized in the Table 6.

Table 4

Experimental details and refined crystal data
for the ternary phases in the $\text{Ho}_{21}\text{Zn}_{25}\text{Al}_{54}$ sample

Compound	$\text{Ho}_3\text{Zn}_{4.4(1)}\text{Al}_{6.6(1)}$		$\text{HoZn}_{0.26(6)}\text{Al}_{2.74(6)}$		
Structure type	$\text{La}_3\text{Al}_{11}$		AuCu_3		
Space group	<i>Immm</i>		<i>Pm3m</i>		
Lattice parameters, nm	$a = 0.42051(3)$ $b = 1.23732(7)$ $c = 0.99581(6)$		$a = 0.42255(2)$		
Cell volume, nm ³	0.51775(9)		0.075448(9)		
Number of atoms in cell	26		3		
$2\theta_{\max}$, $\sin\theta_{\max}/\lambda$	108.50		5.26		
Scale factor	0.32221		0.08031		
Phase composition, mas. %	76.4		23.6		
R_I, R_P	0.086 0.211		0.097 0.211		
Crystallographic data for $\text{Ho}_3\text{Zn}_{4.4(1)}\text{Al}_{6.6(1)}$					
Atoms	WP	Atomic coordinates			$B_{iso} \cdot 10^2$, nm ²
		x	y	z	
Ho1	2a	0	0	0	0.79(2)
Ho2	4g	0	0.6871(5)	0	0.58(2)
X1(2Al)	2c	1/2	1/2	0	1.52(2)
X2(3.3(2)Al+0.7(2)Zn)	4j	1/2	0	0.300(2)	1.08(2)
X3(7.2(1)Al+0.8(1)Zn)	8l	0	0.851(2)	0.729(2)	0.74(2)
X4(0.7(2)Al+7.3(2)Zn)	8l	0	0.6588(9)	0.6243(8)	1.27(2)
Crystallographic data for $\text{HoZn}_{0.26(3)}\text{Al}_{2.74(3)}$ compound					
Ho1	1a	0	0	0	0.69(2)
X(2.74(3)Al+0.26(3)Zn)	3d	0	1/2	1/2	1.14(2)

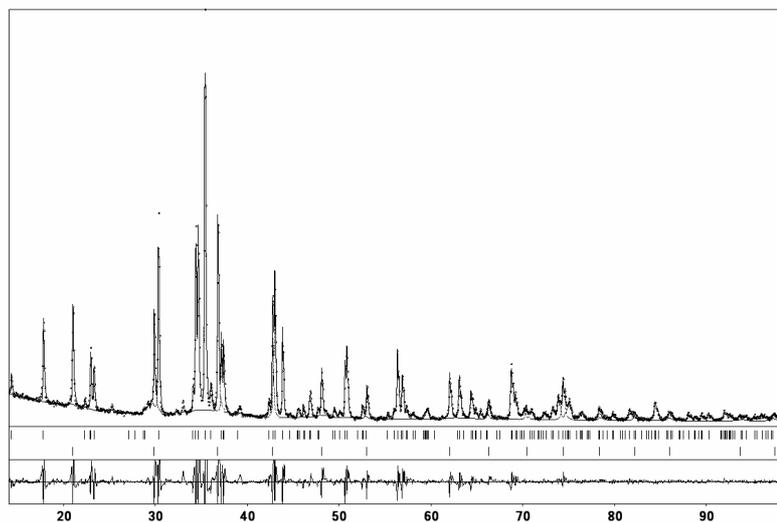


Figure 2. Experimental, calculated and difference diffraction patterns of the two-phase sample with the composition $\text{Ho}_{21}\text{Zn}_{25}\text{Al}_{54}$ ($\text{CuK}\alpha$ radiation)

Table 5

Experimental details and refined crystal data
 for the ternary phases in the $\text{Ho}_{33}\text{Zn}_{22}\text{Al}_{45}$ sample

Compound	$\text{HoZn}_{0.89}\text{Al}_{1.11}$		$\text{HoAl}_{1.66}\text{Zn}_{0.34}$		
Structure type	CaIn_2		MgCu_2		
Space group	$P6_3/mmc$		$Fd-3m$		
Lattice parameters, nm	$a = 0.44800(6)$ $c = 0.70322(9)$		$a = 0.78122(3)$		
Cell volume, nm^3	0.12223(5)		0.47678(6)		
Number of atoms in cell	6		24.0		
$2\theta_{\text{max}}, \sin\theta_{\text{max}}/\lambda$	111.0		5.35		
Scale factor	0.0701		0.2460		
Phase composition, mas. %	45.9		54.1		
R_I, R_P	0.087 0.214		0.054 0.214		
Crystallographic data for the $\text{HoZn}_{0.89(4)}\text{Al}_{1.11(4)}$ compound					
Atoms	WP	Atomic coordinates			$B_{\text{iso}} * 10^2, \text{nm}^2$
Ho	$2b$	x	y	z	
$X(2.22(8)\text{Al})+1.78(8)\text{Zn}$	$4f$	0	0	1/4	0.96(2)
		1/3	2/3	0.466(1)	0.99(2)
Crystallographic data for the $\text{HoAl}_{1.66(3)}\text{Zn}_{0.34(3)}$ compound					
Ho1	$8a$	3/8	3/8	3/8	0.49(2)
$X(13.3(2)\text{Al})+2.7(2)\text{Zn}$	$16d$	0	0	0	0.94(2)

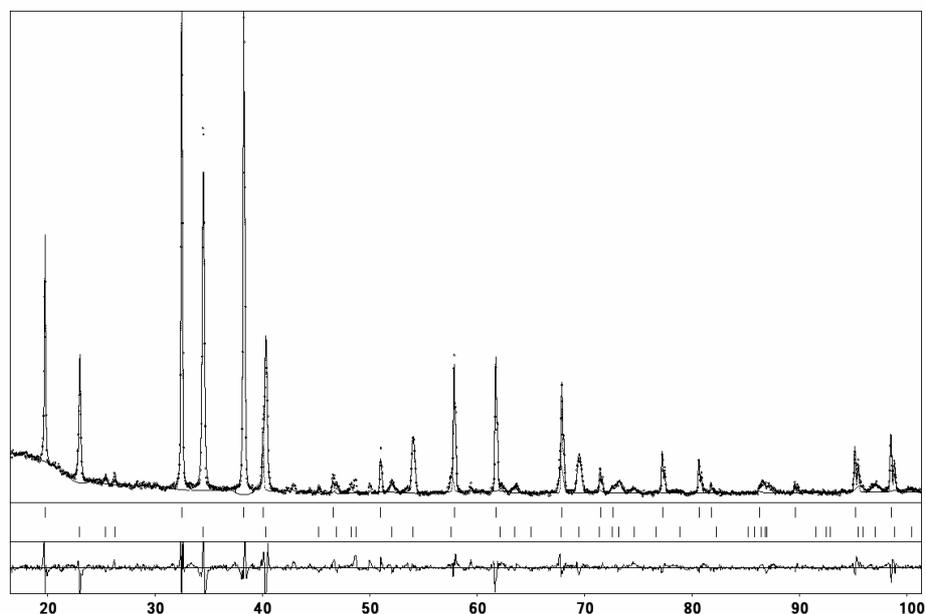


Figure 3. Experimental, calculated and difference diffraction patterns of the two-phase sample with composition $\text{Ho}_{33}\text{Zn}_{20}\text{Al}_{47}$ (Cu-K α radiation)

The Ho-Zn-Al system is significantly different from the previously studied *R*-Zn-Al systems, where *R* is light rare earth metal (La [1], Ce [2], Pr [3], Eu [4], and Nd [5]) by more complex type of the components interaction, formation of the large number of the ternary intermetallic compounds and existence of the substitutional solid solutions on the basis of some binary Ho-Zn and Ho-Al phases.

Formation of the Ho-Zn-Al ternary compounds in the concentration region up to 33.3 at. % of Ho is the typical feature for all the *R*-{Cu, Ag}-Al ternary systems and obviously this trend is typical for other *R*-Zn-Al ternary systems too.

Crystal structures of the almost all ternary phases in the Ho-Zn-Al system are characterized by the statistical distribution of the smaller size atoms (Zn and Al) in the crystallographic sites that causes formation of the homogeneity regions in many cases. Replacing of the smaller Zn atoms by the larger Al atoms leads to the increasing of the lattice parameters and cell volumes.

Interatomic distances in the crystal structures of the ternary compounds of the *R*-Zn-Al systems usually are the same as the respective sums of the metallic radii of the interacting components [5–9]. This indicates that the predominant contribution of metallic type bonding in the ternary rare earth and zinc aluminides.

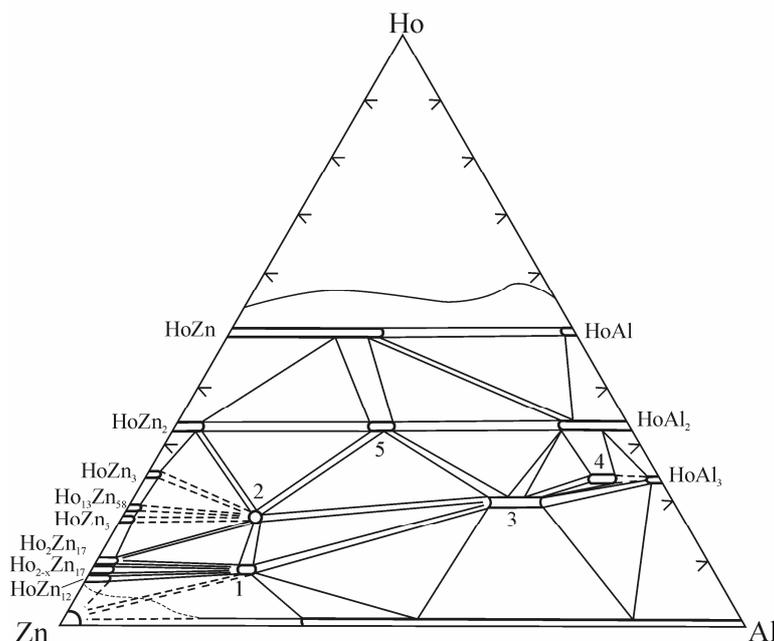


Figure 4. Phase diagram of the Ho–Zn–Al system at 500°C and 0–50 at. % Ho

Table 6

Crystallographic data of the ternary phases in Ho–Zn–Al system

N	Phase	ST	SG	Lattice parameters, nm		
				<i>a</i>	<i>b</i>	<i>c</i>
*	HoZn _{10.9} Al _{1.1}	ThMn ₁₂	<i>I4/mmm</i>	0.8873(1)	–	0.5199(1)
*	Ho _{2-x} Zn _{16.1} Al _{0.9}	Th ₂ Ni ₁₇	<i>P6₃/mmc</i>	0.89709(6)	–	0.87563(8)
*	Ho ₂ Zn _{15.7} Al _{1.3}	Th ₂ Zn ₁₇	<i>R-3m</i>	0.8959(1)	–	1.3139(2)
*	HoZn _{1.85} Al _{0.15}	KHg ₂	<i>Imma</i>	0.44627(7)	0.7039(1)	0.7605(1)
*	HoZn _{0.52} Al _{0.48}	CsCl	<i>Pm-3m</i>	0.3552(1)	–	–
*	HoAl _{1.69} Zn _{0.31}	MgCu ₂	<i>Fd3m</i>	0.78119(2)	–	–
1	HoZn _{4.9-5.1} Al _{3.1-2.9}	HoZn ₅ Al ₃	<i>I4/mmm</i>	0.8615(2)– 0.85952(8)	–	1.6352(4)– 1.6624(3)
2	~HoZn ₄ Al
3	Ho ₃ Zn _{3.7-4.7} Al _{7.3-6.3}	La ₃ Al ₁₁	<i>Immm</i>	0.42040(2)– 0.42058(2)	1.23892(4)– 1.23717(7)	0.99682(8)– 0.99463(5)
4	HoZn _{0.54-0.26} Al _{2.46- 2.74}	AuCu ₃	<i>Pm-3m</i>	0.42249(9)– 0.42379(4)	–	–
5	HoZn _{0.89-1.07} Al _{1.11- 0.93}	CaIn ₂	<i>P6/mmm</i>	0.44800(6)– 0.4471(1)	–	0.70322(9)– 0.7069(1)

* Limit compositions of the solid solutions on the basis of binary compounds.

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СИСТЕМА Ho–Zn–Al ПРИ 500 °С

О. Стельмахович¹, Т. Крачан², Б. Стельмахович¹

¹ Львівський національний університет імені Івана Франка,
вул. Кирила і Мефодія, 6, 79005 Львів, Україна;

² Подільський державний аграрно-інженерний університет,
вул. Шевченка, 13, 32300 Кам'янець-Подільський, Україна
e-mail: bstelmakhovych@gmail.com

Методами рентгеноструктурного та рентгенофазового аналізів побудовано ізотермічний переріз діаграми стану системи Ho-Zn-Al при 500 °С в області до 50 ат. % гольмію. Підтверджено існування двох тернарних інтерметалідів $HoZn_5Al_3$ (власний структурний тип, ПГ $I4/mmm$) та $Ho_3Zn_{4.4}Al_{6.6}$ (СТ La_3Al_{11} , ПГ $Immm$), вперше виявлено та досліджено кристалічну структуру двох нових сполук зі структурами типу $AuCu_3$ та $CaIn_2$. Сполуки характеризуються статистичним розподілом атомів меншого розміру (Zn та Al) і мають незначні області гомогенності, склади яких описують формули: $HoZn_{4.9-5.1}Al_{3.1-2.9}$ ($a = 0,8615(2)–0,85952(8)$, $c = 1,6352(4)–1,6624(3)$ нм),

$\text{Ho}_3\text{Zn}_{3,7-4,7}\text{Al}_{7,3-6,3}$ ($a = 0,42040(2)–0,42058(2)$, $b = 1,23892(4)–1,23717(7)$, $c = 0,99682(8)–0,99463(5)$ нм), $\text{HoZn}_{0,54-0,26}\text{Al}_{2,46-2,74}$, ($a = 0,42249(9)–0,42379(4)$ нм), $\text{HoZn}_{0,89-1,07}\text{Al}_{1,11-0,93}$ ($a = 0,44800(6)–0,4471(1)$, $c = 0,70322(9)–0,7069(1)$ нм). Виявлено існування нової сполуки приблизного складу $\sim\text{HoZn}_4\text{Al}$, кристалічна структура якої невідома.

Бінарні сполуки систем Ho-Zn та Ho-Al розчиняють алюміній та цинк, відповідно, утворюючи тверді розчини заміщення, граничні склади яких визначено методами рентгеноструктурного аналізу: $\text{HoZn}_{10,9}\text{Al}_{1,1}$ (СТ ThMn_{12} , $a = 0,8873(1)$, $c = 0,5199(1)$ нм), $\text{Ho}_{2,3}\text{Zn}_{16,1}\text{Al}_{0,9}$ (СТ $\text{Th}_2\text{Ni}_{17}$, $a = 0,89709(6)$, $c = 0,87563(8)$ нм), $\text{Ho}_2\text{Zn}_{15,7}\text{Al}_{1,3}$ (СТ $\text{Th}_2\text{Zn}_{17}$, $a = 0,8959(1)$, $c = 1,3139(2)$ нм), $\text{HoZn}_{1,85}\text{Al}_{0,15}$ (СТ KHg_2 , $a = 0,44627(7)$, $b = 0,7039(1)$, $c = 0,7605(1)$ нм), $\text{HoZn}_{0,52}\text{Al}_{0,48}$ (СТ CsCl , $a = 0,3552(1)$ нм), $\text{HoAl}_{1,69}\text{Zn}_{0,31}$ (СТ MgCu_2 , $a = 0,78119(2)$ нм).

Ключові слова: інтерметаліди, кристалічна структура, фазові рівноваги.

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