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**TERNARY SYSTEM Er–Zn–In  
IN THE REGION 0–33.3 AT. % OF Er AT 870 K**

**M. Dzevenko\*, I. Bigun, A. Poturay, Ya. Kalychak**

*Ivan Franko National University of Lviv,  
Kyryla i Mefodia Str., 6, UA-79005 Lviv, Ukraine  
e-mail: mashadzev@gmail.com*

The isothermal section of the Er–Zn–In system was constructed based on X-ray powder data and EDX-analyses at 870 K in the region up to 33.3 at. % Er. Three solid solutions:  $\text{ErZn}_{2-1.70}\text{In}_{0-0.30}$  (CeCu<sub>2</sub>-type; space group *Imma*;  $a = 4.448\text{--}4.594(3)$ ,  $b = 6.984\text{--}7.176(5)$ ,  $c = 7.610\text{--}7.463(5)$  Å);  $\text{Er}_2\text{Zn}_{17-16.54}\text{In}_{0-0.46}$  (Th<sub>2</sub>Zn<sub>17</sub>-type; space group *R-3m*;  $a = 8.9465\text{--}8.9704(3)$ ,  $c = 13.1199\text{--}13.1320(6)$  Å);  $\text{ErZn}_{0-0.88}\text{In}_{3-2.12}$  (AuCu<sub>3</sub>-type; space group *Pm-3m*;  $a = 4.563\text{--}4.504(1)$  Å) and one ternary compound with homogeneity region  $\text{ErZn}_{1.28-0.80}\text{In}_{0.72-1.20}$  (CaIn<sub>2</sub>-type; space group *P6<sub>3</sub>/mmc*;  $a = 4.513(2)\text{--}4.662(1)$ ,  $c = 7.023(3)\text{--}7.226(2)$  Å) are formed in the investigated part of this system. The crystal structure of the  $\text{ErZn}_{0-0.92}\text{In}_{3-2.08}$  solid solution was also determined using X-ray single crystal diffraction data on the sample with composition  $\text{ErZn}_{0.87}\text{In}_{2.13}$ .

*Keywords:* indium, zinc, crystal structure, homogeneity region, phase equilibrium.

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### 1. Introduction

The interaction of indium with rare earths and 3*d*-metals has complex character due to different electron structure of interacting components. Much information is available on the crystal structure and the physical properties of ternary compounds in the *R–T–In* (*R* = Rare Earth metals, *T* = Ni, Co or Cu) [1]. Recently, some ternary systems *R–Mn–In* (*R* = Gd, Dy) and *R–Fe–In* (*R* = Gd, Tb, Dy) [2–5] have been investigated as well. Meanwhile, the research on the ternary systems *R–Zn–In* is still missing. Only one partial section at 670 K of the phase diagram has been studied for Yb–Zn–In system [6]. This system is characterized by the presence of three extended homogeneity ranges, indium solubility in Yb<sub>13</sub>Zn<sub>58</sub> and YbZn<sub>2</sub> and of zinc solubility in YbIn<sub>2</sub>, and the existence of one ternary intermetallic compound, YbZn<sub>0.7</sub>In<sub>1.3</sub> (UHg<sub>2</sub> structure type, space group *P6<sub>3</sub>/mmm*). The few ternary compounds with equiatomic composition with CaIn<sub>2</sub>- (*R* = La–Gd, Dy–Er, Yb) or KHg<sub>2</sub>- (*R* = Eu) types have been found in the systems with zinc [7–9].

Therefore, in the present paper we describe the results of the investigations of the Er–Zn–In ternary system. The isothermal section in the region up to 33.3 at. % of Er at *T* = 870 K has been constructed, with the focus on the concentration range of phases existence.

### 2. Experimental

Starting materials for the preparation of the alloys were ingots of the rare earth metals, zinc, and indium, all with nominal purities better than 99.9 %. The 20 samples were synthesized by direct melting inside quartz ampoules under vacuum. The specimens were heated in a box furnace up to 1070 K for 5 hours and annealed at this temperature during 1 hour.

Then they were cooled to 870 K (at a rate of 20 K/hours) and annealed at this temperature during 168 hours. After that, the cooled ampoules were broken, the alloys were grinded with mortar and pestle and pressed into pellets. The pellets were sealed in evacuated silica tubes and annealed for a month at a temperature of 870 K. After annealing no reaction with silica was observed. The X-ray diffraction data for structure refinement were collected on diffractometers Bruker D8, STOE STADI P and Huber Imaging-Plate Guinier camera. The crystal structure refinements were carried out using the Fullprof software [10]. Single crystal X-ray diffraction was performed at room temperature on the Bruker SMART APEX CCD diffractometer with MoK $\alpha$  radiation. The structure was solved by direct methods, and refined by using the SHELXS97 program package [11]. The compositions of some samples were additionally studied by energy dispersive analyses of X-rays (EDX) using a PEMMA-102-02 scanning electron microscope.

### 3. Result and discussion

The isothermal section of the Er–Zn–In ternary system was built at  $T = 870$  K in the region 0–33.3 at. % Er (Fig. 1, Table 1) by means of X-ray powder diffraction data. It was confirmed that at 870 K the following binary compounds exist: ErZn<sub>2</sub> (CeCu<sub>2</sub>-type), ErZn<sub>3</sub> (YZn<sub>3</sub>-type), Er<sub>13</sub>Zn<sub>58</sub> (Gd<sub>13</sub>Zn<sub>58</sub>-type), ErZn<sub>5</sub> (ErZn<sub>5</sub>-type), Er<sub>2</sub>Zn<sub>17</sub> (Th<sub>2</sub>Zn<sub>17</sub>-type), ErZn<sub>12</sub> (ThMn<sub>12</sub>-type), Er<sub>3</sub>In<sub>5</sub> (Pu<sub>3</sub>Pd<sub>5</sub>-type), and ErIn<sub>3</sub> (AuCu<sub>3</sub>-type). The binary compounds ErZn<sub>2</sub>, Er<sub>2</sub>Zn<sub>17</sub> and ErIn<sub>3</sub> form the substitution type solid solutions across the sections of erbium. The rest binary compounds do not dissolve the third component. According to changes in the lattice parameters and cell volume the homogeneity regions of solid solutions based on ErZn<sub>2</sub> and ErIn<sub>3</sub> extend to 10.5 at. % In and 22 at. % Zn respectively (Fig. 2) and the composition of solid solutions can be described as ErZn<sub>2-1.70</sub>In<sub>0-0.30</sub> and ErZn<sub>0-0.88</sub>In<sub>3-2.12</sub> respectively.

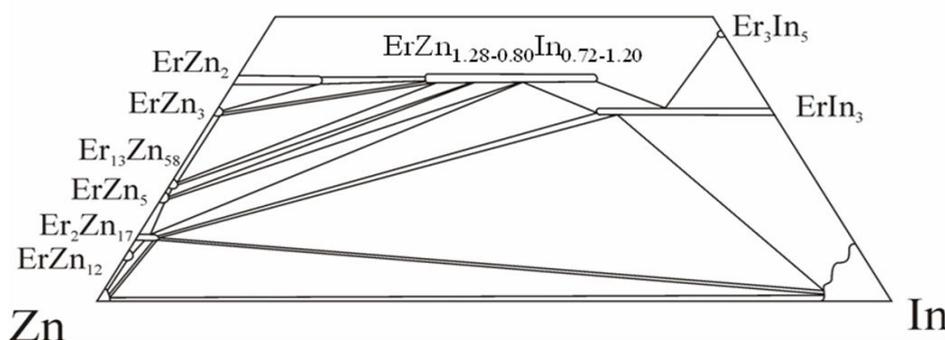


Fig. 1. The isothermal section of the Er–Zn–In system at  $T = 870$  K

Table 1

Crystallographic data of the phases in the Er–Zn–In system

Phase	Structure type	Space group	Lattice parameters, Å		
			<i>a</i>	<i>b</i>	<i>c</i>
ErZn <sub>2-1.70</sub> In <sub>0-0.30</sub>	CeCu <sub>2</sub>	<i>Imma</i>	4.448*–4.594(3)	6.984*–7.176(5)	7.610*–7.463(5)
Er <sub>2</sub> Zn <sub>17-16.54</sub> In <sub>0-0.46</sub>	Th <sub>2</sub> Zn <sub>17</sub>	<i>R-3m</i>	8.9465*–8.9704(3)	–	13.1199*–13.1320(6)
ErZn <sub>0-0.88</sub> In <sub>3-2.12</sub>	AuCu <sub>3</sub>	<i>Pm-3m</i>	4.563*–4.504(1)	–	–
ErZn <sub>1.28-0.80</sub> In <sub>0.72-1.20</sub>	CaIn <sub>2</sub>	<i>P6<sub>3</sub>/mmc</i>	4.513(2)–4.662(1)	–	7.023(3)–7.226(2)

\* Literature data.

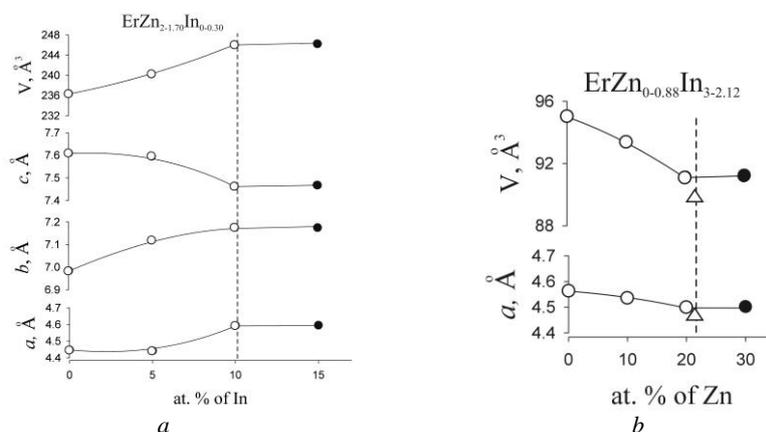


Fig. 2. Dependence of the lattice parameters and the unit cell volume of  $\text{ErZn}_{2-1.70}\text{In}_{0.30}$  vs. the In (a) and  $\text{ErZn}_{0-0.88}\text{In}_{3-2.12}$  vs. the Zn concentration (b) (triangle – single crystal data)

The crystal structure of  $\text{ErZn}_{0-0.88}\text{In}_{3-2.12}$  solid solution has been investigated by X-ray single crystal method. The single crystal suitable for intensity data collection was selected by mechanical fragmentation from the sample  $\text{Er}_{0.25}\text{Zn}_{0.20}\text{In}_{0.55}$ . Single crystal X-ray diffraction was performed on the Bruker diffractometer with  $\text{MoK}\alpha$  radiation ( $\lambda = 0.71073$  Å) in the whole reciprocal sphere at room temperature. All crystallographic data and details of the data collection are listed in the Table 2 and parameters of atoms are shown in Table 3. On the basis of the obtained data, it can be assumed that composition of single crystal is the limited composition of this solid solution.

Table 2

Experimental details and crystallographic data for  $\text{ErZn}_{0.87}\text{In}_{2.13}$  phase

Molar mass, g/mole	468.7
Structure type	AuCu <sub>3</sub>
Space group, $Z$	$Pm\bar{3}m$ , 1
Lattice parameters, $a$ , Å	4.474(4)
$V$ , Å <sup>3</sup>	89.5(3)
Calculated density(g/cm <sup>3</sup> )	8.57
Radiation, wavelength Å	$\text{MoK}\alpha$ $\lambda=0.71073$
Absorption coefficient (mm <sup>-1</sup> )	30.937
$F(000)$	196
Range $\theta$	4.56–37.60
Temperature ( $T$ , K)	296(2)
Range $hkl$	$-6 \leq h \leq 6; -7 \leq k \leq 7; -7 \leq l \leq 7$
Total no. of reflections	930
Independent reflections	73
$R_{\text{int}}$	0.0297
Reflections with $I > 2\sigma(I)$	73
Refinement method	Full-matrix least-squares on $F^2$
Goodness-of-fit on $F^2$	1.315
Extinction coefficient	0.027(4)
$R[I > 2\sigma(I)]$ ( $R1=$ ) ( $wR2=$ )	0.0159; 0.0364
$R[\text{all}]$ ( $R1=$ ) ( $wR2=$ )	0.0159
Largest diff. peak/hole, e/Å <sup>-3</sup>	1.768/–1.817

Table 3

Atomic coordinates and thermal displacement parameters ( $\text{\AA}^2$ ) for  $\text{ErZn}_{0.87}\text{In}_{2.13}$  phase

Atom	Wyckoff site	Occupation	x	y	z	$U_{\text{eq}}^*$	$U_{11}$	$U_{22}$	$U_{33}$
Er	1a	1	0	0	0	0.0061(2)	0.0061(2)	0.0061(2)	0.0061(2)
X	3c	0.71(2)In+0.29(2)Zn	0	1/2	1/2	0.0123(4)	0.0090(4)	0.0190(5)	0.0090(4)

\* $U_{\text{eq}}$  is defined as one third of the trace of orthogonalized  $U_{ij}$  tensor;  $U_{12}=U_{13}=U_{23}=0 \text{ \AA}^2$

The crystal structure of  $\text{ErZn}_{2-1.70}\text{In}_{0-0.30}$  solid solutions has been investigated by X-ray powder diffraction on the  $\text{Er}_{0.333}\text{Zn}_{0.617}\text{In}_{0.05}$  sample. Fig. 3, *a* shows the X-ray diffraction patterns of the respective sample. The results of the crystal structure investigation (Table 4, 5) revealed the occupancies of the smaller atomic positions by statistic mixtures (Zn/In).

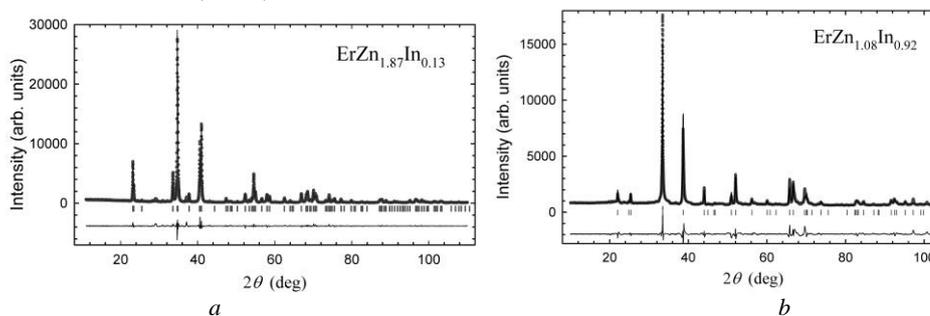


Fig. 3. Observed, calculated and difference X-ray diffraction patterns of the  $\text{ErZn}_{1.87}\text{In}_{0.13}$  (*a*) and  $\text{ErZn}_{1.08}\text{In}_{0.92}$  (*b*) phases

The phase and EDX analyses of a sample (Fig. 4, *b*) in the region near the  $\text{Er}_2\text{Zn}_{17}$  binary compound established the formation of the  $\text{Er}_2\text{Zn}_{17-16.54}\text{In}_{0-0.46}$  solid solution up to ~3 at. % In. The crystal structures of this solid solutions was investigated by X-ray powder diffraction on the  $\text{Er}_{10}\text{Zn}_{80}\text{In}_{10}$  sample (Fig. 4, *a*), which contained an additional In phase (weight fractions of phases = 5 %). The crystal structure data and details of structure refinements are given in Table 4 and 5. The one crystallographic position (6c) in the structure of this phase is occupied of by the mixtures of Zn and In atoms.

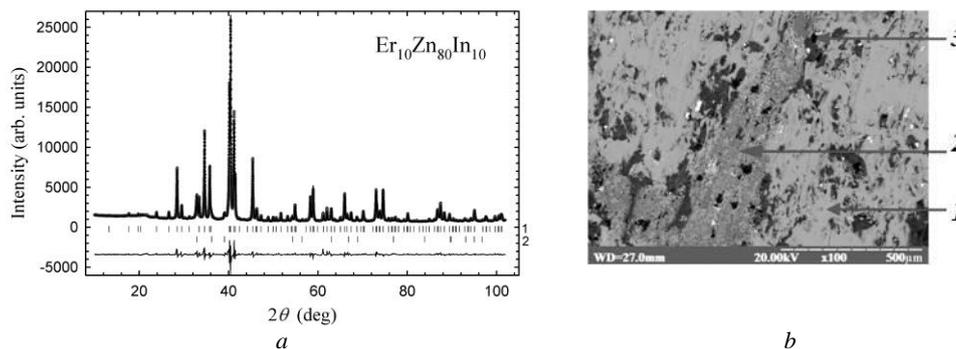


Fig. 4. Observed, calculated and difference X-ray diffraction patterns of the  $\text{Er}_{10}\text{Zn}_{80}\text{In}_{10}$  sample (*1* – phase  $\text{Er}_2\text{Zn}_{16.54}\text{In}_{0.46}$ ; *2* – In) (*a*) and EDX microphotographs of this sample (*b*) (*1* – phase  $\text{Er}_2\text{Zn}_{16.54}\text{In}_{0.46}$ ; *2* –  $\text{Er}_2\text{O}_3$ ; *3* – In)

The existence of earlier known ternary compound with  $\text{CaIn}_2$ -type was confirmed. The homogeneity region was determined for this compound across the section 33.3 at. % Er (Fig. 5), and the composition of compound can be described by formula  $\text{ErZn}_{1.28-0.80}\text{In}_{0.72-1.20}$ .

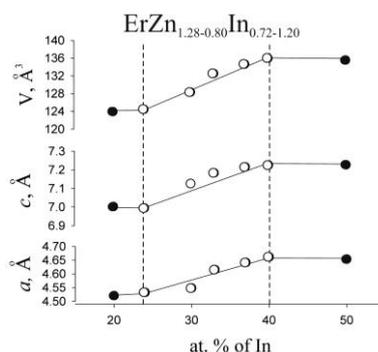


Fig. 5. Dependence of the lattice parameters and the unit cell volume  $V$  of  $\text{ErZn}_{1.28-0.80}\text{In}_{0.72-1.20}$  vs. the concentration of In

Additionally, the investigation of crystal structure of this compound was performed by X-ray powder diffraction on the  $\text{Er}_{0.333}\text{Zn}_{0.367}\text{In}_{0.30}$  sample. Fig. 3b shows the X-ray diffraction patterns. According to the results of the crystal structure investigation (Table 4, 5) the crystallographic position (4f) is occupied by mixtures (Zn/In).

Table 4

Experimental details and crystallographic data for  $\text{ErZn}_{1.87}\text{In}_{0.13}$ ,  $\text{Er}_2\text{Zn}_{16.54}\text{In}_{0.46}$ ,  $\text{ErZn}_{1.08}\text{In}_{0.92}$  phases

Refined composition	$\text{ErZn}_{1.87}\text{In}_{0.13}$	$\text{Er}_2\text{Zn}_{16.54}\text{In}_{0.46}$	$\text{ErZn}_{1.08}\text{In}_{0.92}$
$D_{\text{cal}}$ , g/cm <sup>3</sup>	8.41	7.94	8.50
$M_r$ , g/mole	390.5	1462	342.08
Structure type	$\text{CeCu}_2$	$\text{Th}_2\text{Zn}_{17}$	$\text{CaIn}_2$
Space group, $Z$	$Imam$ , 4	$R-3m$ , 19	$P6_3/mmc$ , 2
Lattice parameters			
$a$ , Å	4.4532(1)	8.9704(3)	4.6070(8)
$b$ , Å	7.1263(3)	–	–
$c$ , Å	7.5801(3)	13.1320(6)	7.162(1)
$V$ , Å <sup>3</sup>	240.55(2)	915.14(6)	131.16(6)
Equipment	STOE STADI P	Guinier Camera	Guinier Camera
Wavelength	$\text{CuK}\alpha_1$	$\text{CuK}\alpha_1$	$\text{CuK}\alpha_1$
$2\theta$ range	6.00°–110.00°	10.00°–102.00°	10.00°–102.00°
Step size in $2\theta$	0.015	0.005	0.005
$R_B$ , $R_r$ , %	3.00; 2.61	3.33, 4.09	6.51; 6.34
$R_p$ , $R_{wp}$ , %	6.36; 9.51	3.96, 5.65	5.83; 8.49

Therefore, the ternary system Er–Zn–In is characterized by formation of one ternary compound and three solid solutions based on  $\text{ErZn}_2$ ,  $\text{Er}_2\text{Zn}_{17}$  and  $\text{ErIn}_3$  binary compounds. The variations of lattice parameters in the homogeneity regions of mentioned above solid solutions and the ternary compound are due to the substitution of the larger In atoms ( $r = 1.626$  Å) by the smaller Zn atoms ( $r = 1.332$  Å) [12] or vice versa. It should be noted that for the solid solutions the variation of lattice parameters is slightly deviated from Vegard's rule.

Table 5

Atomic coordinates and thermal displacement parameters for  
 $\text{ErZn}_{1.87}\text{In}_{0.13}$ ,  $\text{Er}_2\text{Zn}_{16.54}\text{In}_{0.46}$  and  $\text{ErZn}_{1.08}\text{In}_{0.92}$  phases

Atoms	Wyckoff site	Occupation	x	y	z	$B_{\text{iso}}$ , Å <sup>2</sup>
$\text{ErZn}_{1.87}\text{In}_{0.13}$						
Er	4e	1	0	1/4	0.5280(3)	0.8(3)
X	8h	0.06(1)Zn+0.94(1)In	0	0.0408(6)	0.1675(6)	1.2(3)
$\text{Er}_2\text{Zn}_{16.54}\text{In}_{0.4}$						
Er	6c	1	0	0	0.3375(4)	0.6(1)
Zn1	18h	1	0.4945(5)	0.5055(5)	0.1530(5)	0.7(1)
Zn2	18f	1	0.2990(6)	0	0	0.6(1)
Zn3	9d	1	1/2	0	1/2	0.9(2)
X	6c	0.72(2)Zn+0.28(2)In	0	0	0.1016(6)	0.7(2)
$\text{ErZn}_{1.08}\text{In}_{0.92}$						
Er	2b	1	0	0	1/4	0.8(3)
X	4f	0.54(1)Zn+0.46(1)In	1/3	2/3	0.0383(8)	1.1(3)

Comparing the Er–{Mn, Fe, Co, Ni, Cu, Zn}–In systems with each other, it should be underlined that the ternary system with zinc has some similarity only with the Er–Mn–In system. In both systems, the ternary compounds exist on cross-section 33.3 at. % R and form the homogeneity regions. The main difference between Er–Zn–In and related systems is the formation of several solid solutions based on binary compounds. On the other hand, this feature is characteristic for the Yb–Zn–In [6] and the Yb–Zn–Ga [13] systems. Furthermore, small number of compounds is another similar feature of these systems. Regarding to the structural types of phases, it should be mentioned that the phases exist on cross-section 33.3 at. % R in the Er–{Mn, Cu}–In, {Er, Yb}–Zn–In systems and the Yb–Zn–Ga system as well as are crystallised in the hexagonal structure types; moreover the majority of them are related to  $\text{AlB}_2$ -type.

#### 4. Conclusion

The phase equilibria of Er–Zn–In system at 870 K are characterized by the formation of one ternary compound, namely  $\text{ErZn}_{1.28-0.80}\text{In}_{0.72-1.20}$  ( $\text{CaIn}_2$ -type). The some of the binary compounds, namely  $\text{ErZn}_2$  ( $\text{CeCu}_2$ -type),  $\text{Er}_2\text{Zn}_{17}$  ( $\text{Th}_2\text{Zn}_{17}$ -type) and  $\text{ErIn}_3$  ( $\text{AuCu}_3$ -type) reveal homogeneity ranges by Zn/In or In/Zn substitutions.

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**ВЗАЄМОДІЯ КОМПОНЕНТІВ У СИСТЕМІ Er–Zn–In  
В ОБЛАСТІ 0–0,333 АТ. % Er ПРИ 870 К**

**М. Дзевенко\*, І. Бігун, А. Потурай, Я. Каличак**

*Львівський національний університет імені Івана Франка,  
вул. Кирила і Мефодія, 6, 79005 Львів, Україна  
e-mail: mashadzev@gmail.com*

За результатами рентгенофазового та частково ЕДРС аналізів побудовано ізотермічний переріз діаграми стану потрійної системи Er–Zn–In при 870 К в області до 0,333 ат. часток Er. Зразки для дослідження виготовляли методом безпосередньої взаємодії компонентів у вакуумованих кварцових ампулах з використанням програмованої термічної обробки.

У дослідженій області системи Er–Zn–In утворюються три тверді розчини та одна тернарна сполука з областю гомогенності. Межі області гомогенності тернарної фази зі структурою типу  $\text{CaIn}_2$  простягаються від 0,24 до 0,40 ат. частки Індію, а її склад описують формулою  $\text{ErZn}_{1,28-0,80}\text{In}_{0,72-1,20}$  (просторова група  $P6_3/mmc$ ;  $a = 4,513(2)$ – $4,662(1)$ ,  $c = 7,023(3)$ – $7,226(2)$  Å). В межах області гомогенності відбувається збільшення параметрів комірки внаслідок заміщення менших атомів цинку на більші атоми індію. Твердий розчин на основі бінарної сполуки  $\text{ErZn}_2$  також утворюється вздовж ізоконцентрати Ербію 0,333 ат. частки, а склад фази може бути описаний формулою:  $\text{ErZn}_{2-1,70}\text{In}_{0-0,30}$  (структурний тип  $\text{CeCu}_2$ , просторова група  $Imma$ ;  $a = 4,448$ – $4,594(3)$ ,  $b = 6,984$ – $7,176(5)$ ,  $c = 7,610$ – $7,463(5)$  Å). Розчинність Індію в бінарній сполуці  $\text{Er}_2\text{Zn}_{17}$  (структурний тип  $\text{Th}_2\text{Zn}_{17}$ , просторова група  $R-3m$ ;  $a = 8,9465$ – $8,9704(3)$ ,  $c = 13,1199$ – $13,1320(6)$  Å) становить до 0,03 ат. часток. Твердий розчин на основі бінарної сполуки  $\text{ErIn}_3$  (структурний тип  $\text{AuCu}_3$ , просторова група  $Pm-3m$ ;  $a = 4,563$ – $4,504(1)$  Å) існує вздовж ізоконцентрати ербію 0,25 ат. часток до 0,22 ат. часток цинку. В межах цього твердого розчину відбувається заміщення атомів In на атоми Zn. Кристалічну структуру цього твердого розчину вивчали рентгеноструктурним методом монокристала на прикладі фази складу  $\text{ErZn}_{0,87}\text{In}_{2,13}$ .

*Ключові слова:* ербій, цинк, індію, фазові рівноваги, кристалічна структура, твердий розчин.

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