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Lu-V-{Ge, Sn} ternary systems

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The isothermal sections of the phase diagrams of the Lu–V–Ge and Lu-V-Sn ternary systems were constructed at 870 K over the whole concentration range using X-ray diffraction and EPM analyses. In the Lu-V-Ge system a formation of the substitutional solid solution $Lu_5Ge_{3-x}V_x$ based on the Lu_5Ge_3 binary compound (Mn_5Si_3 structure type) was found up to 6 at. % V. Insertion of the V atoms in the structure of the $LuGe_2$ binary germanide ($ZrSi_2$ structure type, up to 5 ат. % V) results in the formation of the $LuV_{0,15}Ge_2$ ternary phase ($CeNiSi_2$ structure type, space group *Cmcm*, $a = 0.40210(4)$, $b = 1.5661(1)$, $c = 0.38876(3)$ nm), which corresponds to the limit composition of the interstitial solid solution LuV_xGe_2. The interaction between the elements in the Lu-V-Sn system results in the formation of one ternary compound LuV_6Sn_6 ($SmMn_6Sn_6$-type, space group $P6/mmm$, $a = 0.5503(2)$, $c = 0.9171(4)$ nm) at investigated temperature.

Keywords: Intermetallics; Ternary system; Phase equilibria; Solid solution.

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Introduction

Study of the phase diagrams of the metallic systems can provides important information concerning the formation, temperature and concentration stability of the compounds for developing of new materials. Among R–M–{Ge, Sn} systems (M-*d*-metal) the most systems were studied with M = Mn, Fe, Co, Ni, or Cu [1, 2]. Analysis of the literature showed, that the higher number of the formed intermediate ternary phases was observed in the R-Ni-{Ge,Sn} systems. Passing from nickel to cobalt, iron and manganese the quite decreasing number of the formed ternary compounds up to one for the {Y, Gd, Dy}–Fe–Sn systems was observed [3, 4]. The R-V-Sn systems were studied with R = Gd, Er [5]. Both Gd–V–Sn and Er–V–Sn systems are characterized by formation of one ternary compound with stoichiometry RV_6Sn_6 ($SmMn_6Sn_6$-type). Isotypic compounds with the $SmMn_6Sn_6$-type were also found with Dy, Ho, Tm, and Lu, while YV_6Sn_6 stannide crystallizes in the $HfFe_6Ge_6$ structure type [5]. Among R-V-Ge systems the phase diagram was constructed for the Y-V-Ge system only at 870 K [6]. Interaction of yttrium with vanadium and germanium resulted in formation of one ternary compound with the $CeNiSi_2$ structure type. The crystal structure and magnetic property studies of intermetallics $RVGe_3$ (R=La, Ce, Pr, Nd) with perovskite-type $BaNiO_3$ structure were reported in Ref. [7]. With regard on the literature data in comparison to the intensively studied R–M–{Ge, Sn} where M are Fe, Co, Ni and Cu, much less information concerns the V-containing systems.

In this paper we present the results obtained in the investigation of the Lu-V-Ge (870 K) and Lu-V-Sn (870 K) ternary systems and analysis of an influence of *p*-element on the component interaction.

I. Experimental details

The alloys were prepared by a direct twofold arc melting of the constituent elements (lutetium, purity of 99.9 wt.%; vanadium, purity of 99.99 wt.%; tin and germanium, purity of 99.999 wt.%) under protected argon atmosphere on a water-cooled copper crucible. For better homogenization the samples were melted twice. The weight losses of the initial total mass after arc-melting were lower than 1 wt. %. The pieces of the as-cast buttons were homogenized for one month at 870 K in evacuated silica tubes and then water quenched. Phase analysis was performed using X-ray powder diffractions of the synthesized samples (DRON-4.0, Fe K_α radiation).

Table 1

Crystallographics characteristic of the binary phases in the Lu-Ge and V-Ge systems at 870 K

Phase	Space group	Structure type	Lattice parameters, nm			Reference
			a	*b*	*c*	
$LuGe_2$	*Cmcm*	$ZrSi_2$	0.3987	1.5558	0.3851	[14]
			0.3997(2)	1.5599(5)	0.3878(3)	this work
$LuGe_{1.5}$	*P6/mmm*	AlB_2	0.383		0.405	[15]
			0.3819(2)		0.4042(3)	This work
Lu_3Ge_4	*Cmcm*	Er_3Ge_4	0.3968	1.0438	1.4040	[16]
			0.3964(3)	1.0441(5)	1.4049(7)	This work
$Lu_{11}Ge_{10}$	*I4/mmm*	$Ho_{11}Ge_{10}$	1.0668		1.5980	[14]
			1.0651(8)		1.5967(7)	this work
Lu_5Ge_3	$P6_3/mcm$	Mn_5Si_3	0.8216		0.6158	[17]
			0.8233(1)		0.6161(2)	this work
$V_{11}Ge_8$	*Pnam*	$Cr_{11}Ge_8$	1.3398	1.6135	0.5017	[18]
			1.3395(4)	1.6096(8)	0.4999(2)	This work
V_5Ge_3	*I4/mcm*	W_5Si_3	0.9565		0.4867	[19]
			0.9569(4)		0.4861(3)	this work
V_3Ge	*Pm*-3*n*	Cr_3Si	0.4783			[20]
			0.4967(1)			this work

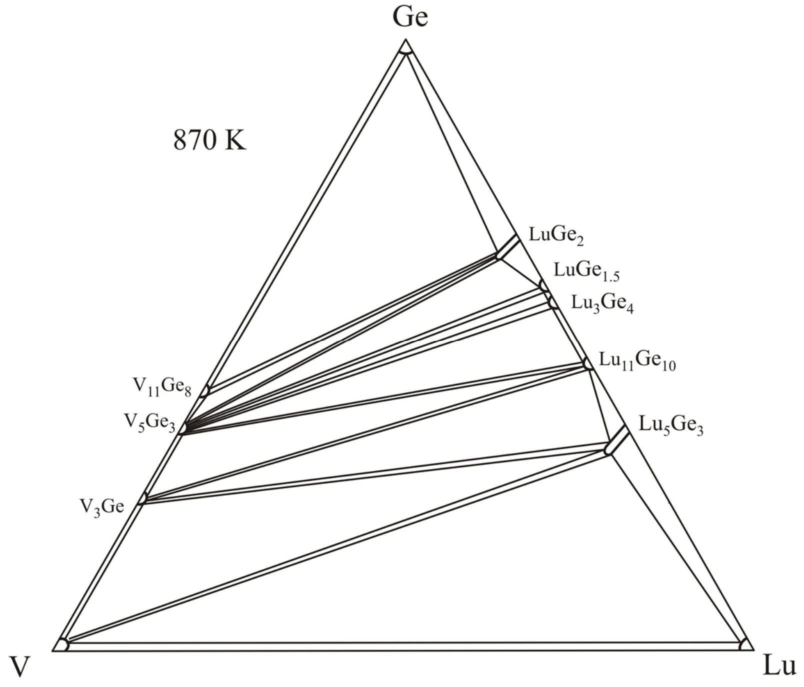

Fig. 1. Isothermal sections of the Lu-V-Ge phase diagram at 870 K.

The observed diffraction intensities were compared with reference powder patterns of the pure elements, binary and known ternary phases. The chemical and phase compositions of the obtained samples were examined by Scanning Electron Microscopy (SEM) using REMMA-102-02 scanning microscope. Quantitative electron probe microanalysis (EPMA) of the samples was carried out by using an energy-dispersive X-ray analyzer with the pure elements as standards (an acceleration voltage was 20 kV; *K*- and *L*-lines were used). The data for the crystal structure refinements were collected at room temperature using STOE STADI P diffractometer (Cu$K_{\alpha1}$ radiation). Calculations of the crystallographic parameters and theoretical diffraction patterns were performed using WinCSD program package [8]. Rietveld refinement was performed using the FullProf Suite program package [9].

Table 2

Phase composition of the selected Lu-V-Ge alloys according to EPMA data

Phase/Component content	Lu, at.%	V, at.%	Ge, at.%
$Lu_5V_{70}Ge_{25}$			
V_3Ge		76.17	23.83
$Lu_5V_xGe_{3-x}$	63.23	4.02	32.75
$Lu_{60}V_{10}Ge_{30}$			
$Lu_5V_xGe_{3-x}$	62.55	5.74	31.71
(V)		99.98	
(Lu)	99.96		
$Lu_{30}V_{30}Ge_{40}$			
$Lu_{11}Ge_{10}$	54.87		45.13
$Lu_5V_xGe_{3-x}$	62.45	3.78	33.77
V_3Ge		74.77	25.23
$Lu_9V_{44}Ge_{47}$			
$V_{11}Ge_8$		56.97	43.03
V_5Ge_3		63.56	36.44
LuV_xGe_2	31.87	4.86	63.27
$Lu_{25}V_{25}Ge_{50}$			
LuV_xGe_2	31.61	4.94	63.45
$LuGe_{1,5}$	58.21		41.79
V_5Ge_3		64.03	35.97
$Lu_{32}V_{10}Ge_{58}$			
LuV_xGe_2	31.74	4.88	63.38
$LuGe_{1,5}$	58.25		41.75
V_5Ge_3		63.83	36.17
$Lu_{20}V_{20}Ge_{60}$			
LuV_xGe_2	31.62	4.91	63.47
$V_{11}Ge_8$		56.83	43.17
(Ge)			99.98

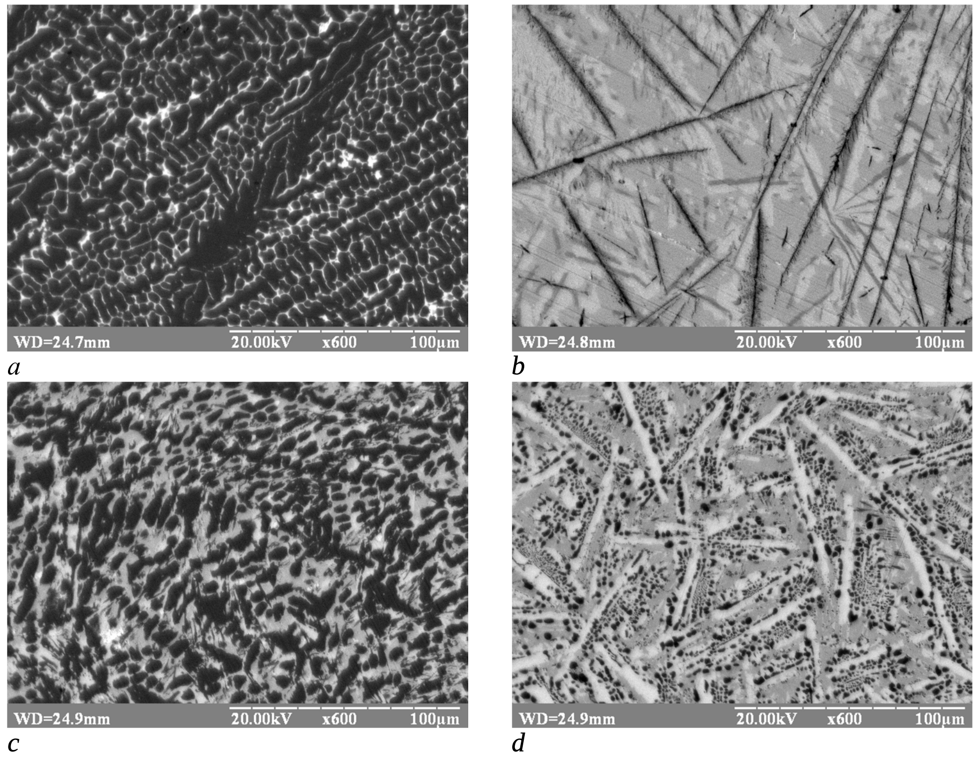

Fig. 2. Electron microphotographs of the Lu-V-Ge alloys:

a) $Lu_5V_{70}Ge_{25}$ – V_3Ge (dark phase); $Lu_5V_xGe_{3-x}$ (light phase);

b) $Lu_{32}V_{10}Ge_{58}$ – LuV_xGe_2 (grey phase); $LuGe_{1,5}$ (light grey phase); V_5Ge_3 (dark phase);

c) $Lu_9V_{44}Ge_{47}$ – $V_{11}Ge_8$ (dark grey phase); LuV_xGe_2 (light phase); V_5Ge_3 (grey phase);

d) $Lu_{30}V_{30}Ge_{40}$ – V_3Ge (dark phase); $Lu_{11}Ge_{10}$ (grey phase); $Lu_5V_xGe_{3-x}$ (light phase).

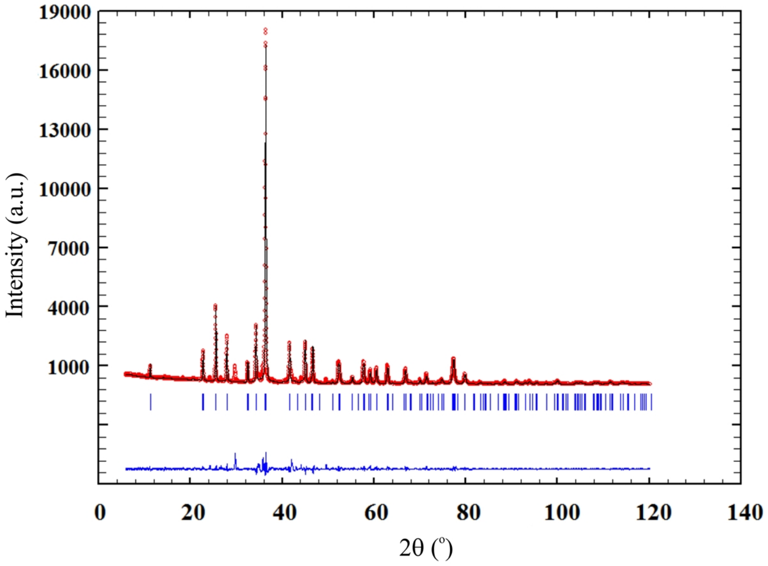

Fig. 3. The observed, calculated and difference X-ray patterns of the $Lu_{33}V_5Ge_{62}$ sample

Table 3

Crystallographics characteristic of the binary phases in the Lu-Sn and V-Sn systems at 870 K

Phase	Space group	Structure type	Lattice parameters, nm			Reference
			a	*b*	*c*	
$LuSn_2$	*Cmcm*	$ZrSi_2$	0.4343	1.5997	0.4273	[22]
			0.4341(2)	1.5990(4)	0.4269(3)	this work
$Lu_{11}Sn_{10}$	*I4/mmm*	$Ho_{11}Ge_{10}$	1.12953		1.6424	[21]
			1.1291(2)		1.6389(4)	this work
Lu_5Sn_3	$P6_3/mcm$	Mn_5Si_3	0.8700		0.6355	[23]
			0.8678(3)		0.6351(3)	this work
VSn_2	*Fddd*	$CuMg_2$	0.9488	0.5485	1.8667	[24]
			0.9487(4)	0.5486(1)	1.8675(3)	this work
V_3Sn	*Pm*-3*n*	Cr_3Si	0.4980			[25]
			0.4967(1)			this work

II. Results and discussion

2.1. Lu-V-Ge system

The binary boundary Lu-Ge, Lu-V and V-Ge systems have been investigated earlier and their phase diagrams are well known in the literature [10-13]. In the V-Ge system we confirmed an existence of the V_3Ge (Cr_3Si-type); V_5Ge_3 (W_5Si_3-type); and $V_{11}Ge_8$ ($Cr_{11}Ge_8$-type).binaries. Phase analysis of the sample, which corresponds to the $V_{17}Ge_{31}$ compound, showed two phases in equilibrium: $V_{11}Ge_8$ and Ge. In the Lu-Ge binary system the $LuGe_2$, $LuGe_{1.5}$, Lu_3Ge_4, $Lu_{11}Ge_{10}$, and Lu_5Ge_3 compounds were successfully identified in the course of our investigations. Crystallographic characteristics of the Lu-Ge and V-Ge binary compounds are listed in Table 1. Calculated lattice parameters are in agreement with literature data.

The phase equilibria in the Lu–V–Ge system have been established at 870 K using the X-ray and metallographic analyses of the examined binary and ternary alloys. The isothermal section of the Lu-V–Ge ternary system at corresponding temperature is illustrated in Fig. 1. The phase compositions of the selected alloys are presented in Table 2. The electron microphotographs of the some alloys are shown in Fig. 2.

No binary phases were observed in the Lu-V system at investigated temperature [11], the corresponding ternary samples in the region Lu-Lu_5Sn_3-V contain three phases in equilibrium – $Lu_5V_xGe_{3-x}$, Lu, and V.

The solubility of the third component in the V-Ge binaries and in the most of the Lu-Ge binary compounds is less than 1-2 at. % under our conditions. The solubility of V in the Lu_5Ge_3 compound (Mn_5Si_3 structure type) extends up to 6 at. % along isoconcentrate of 62 at. % Lu ($a = 0.82522(9)$, $c = 0.6171(2)$ nm for $Lu_{62}V_6Ge_{32}$

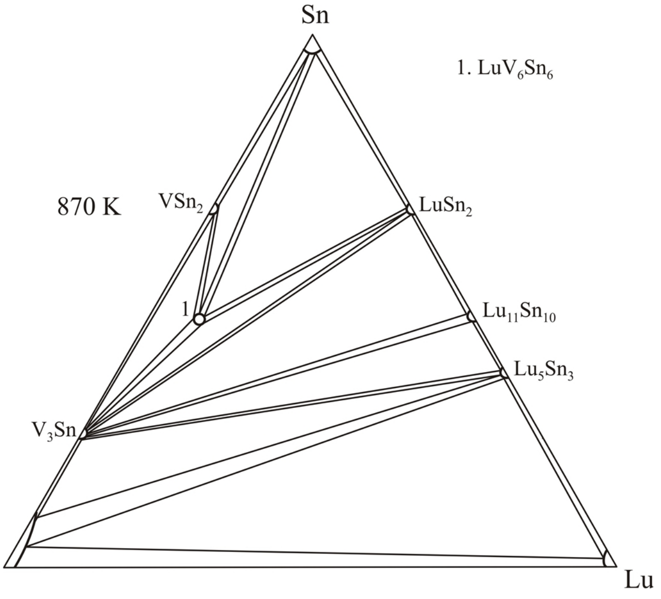

Fig. 4. Isothermal sections of the Lu-V-Sn phase diagram at 870 K.

Table4

Phase composition of the selected Lu-V-Sn alloys according to EPMA data

Phase/Cont. component	Lu, at.%	V, at.%	Sn, at.%
$Lu_{40}V_{40}Sn_{20}$			
Lu_5Sn_3	63.23		36.77
(Lu)	99.98		
(V)		99.99	
$Lu_{30}V_{35}Sn_{35}$			
$Lu_{11}Sn_{10}$	46.81		53.19
Lu_5Sn_3	62.45		37.55
V_3Sn		74.26	25.74
$Lu_{20}V_{40}Sn_{40}$			
$Lu_{11}Sn_{10}$	45.92		54.08
V_3Sn		76.56	23.44
$LuSn_2$	34.47		65.53
$Lu_5V_{50}Sn_{45}$			
V_3Sn		73.47	26.53
LuV_6Sn_6	8.21	45.43	46.36
VSn_2		34.23	65.77
$Lu_{30}V_5Sn_{65}$			
$LuSn_2$	34.66		65.34
V_3Sn		73.30	26.70
LuV_6Sn_6	7.87	45.57	46.56
$Lu_{20}V_{20}Sn_{60}$			
$LuSn_2$	33.87		66.13
LuV_6Sn_6	8.18	46.06	45.76
(Sn)			99.97

sample).

The formation of an interstitial solid solution LuV_xGe_2 (up to 5 at. % V) based on the $LuGe_2$ ($ZrSi_2$-type) binary compound was found. The limit composition of this solid solution was estimated from the systematic analysis of the cell parameters and by the results of electron microprobe analysis (Table 2, Fig. 2, b, c). Crystal structure refinements of the $Lu_{33}V_5Ge_{62}$ sample showed that insertion of the V atoms in the tetragonal-antiprismatic voids of the structure of the $LuGe_2$ binary germanide ($ZrSi_2$ structure type, crystallographic site 4(c) 0 y 1/4) results in the realization of the $CeNiSi_2$ structure type (space group Cmcm, a = 0.40210(4), b = 1.5661(1), c = 0.38876(3) nm; Lu in 4(c) y = 0.3965(1); V in 4(c) y = 0.190(2), G = 0.149(2); Ge1 in 4(c) y = 0.0525(2); Ge2 in 4(c) y = 0.7532(2); R_{Bragg}=0.0348, R_F=0.0234).

The observed, calculated and difference X-ray

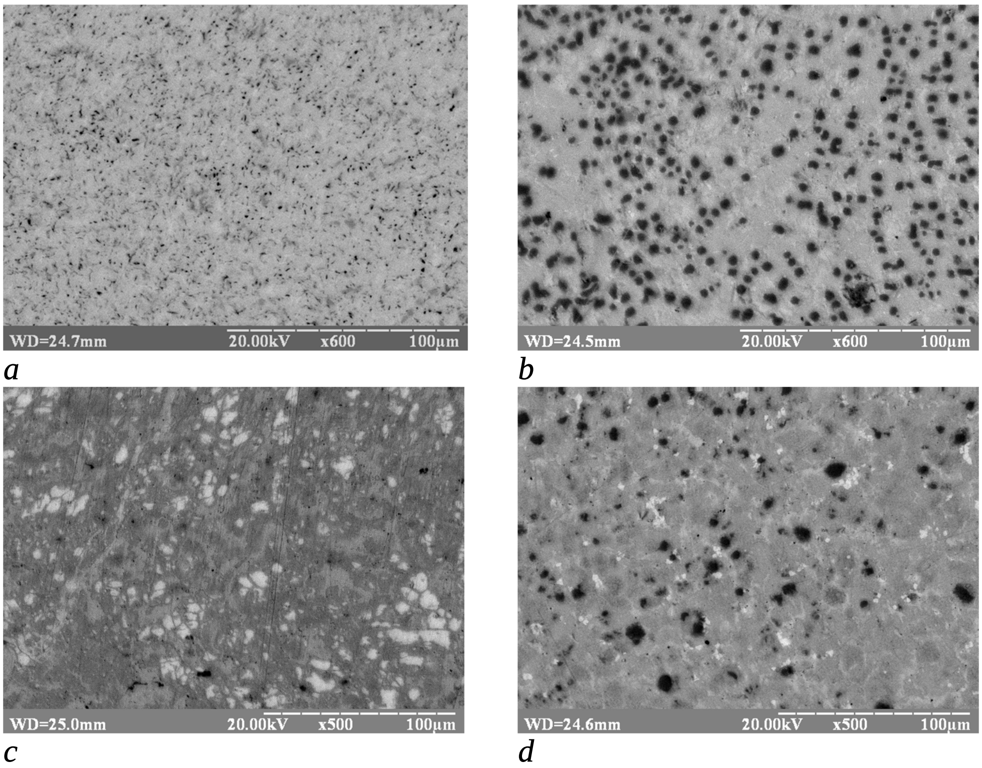

Fig. 5. Electron microphotographs of the Lu-V-Sn alloys:
a) $Lu_{40}V_{40}Sn_{20}$ – Lu_5Sn_3 (grey phase); Lu (light phase); V (dark phase);
b) $Lu_{20}V_{40}Sn_{40}$ – $Lu_{11}Sn_{10}$ (grey phase); V_3Sn (dark phase); $LuSn_2$ (light phase);
c) $Lu_{20}V_{20}Sn_{60}$ – LuV_6Sn_6 (dark grey phase); $LuSn_2$ (light phase); Sn (grey phase);
d) $Lu_{30}V_{35}Sn_{35}$ – $Lu_{11}Sn_{10}$ (grey phase); V_3Sn (dark phase); Lu_5Sn_3 (light phase).

patterns of the $Lu_{33}V_5Ge_{62}$ sample are shown in Fig. 3.

2.2. Lu-V-Sn system

To check the literature data of the binary boundary phases of the Lu-V-Sn system all known binary compounds in the Lu-Sn and V-Sn systems were synthesized and analyzed. In the V-Sn system the presence of the V_3Sn (Cr_3Si-type) and VSn_2 (Mg_2Cu-type) compounds was confirmed according to Ref. [10]. The data concerning the Lu-Sn binary system were used according to Refs. [10, 21]. In course of our study the $LuSn_2$ ($ZrSi_2$-type), Lu_5Sn_3 (Mn_5Si_3-type) and $Lu_{11}Sn_{10}$ ($Ho_{11}Ge_{10}$-type) binaries were confirmed. Crystallographic characteristics of the observed binary compounds are summarized in Table 3. The solubility of the third component in these compounds is less than 1-2 at. %.

The isothermal section of the Lu-V-Sn system at 870 K, presented in Fig. 4, was constructed based on the phase and Electron Microprobe analyses of the prepared ternary and binary samples. The results of the EPMA data for selected alloys are presented in Table 4. Electron microphotographs of the selected alloys are shown in Fig. 5.

According to reported phase diagram [11] no binary phases were formed in the Lu-V system. Performed phase analysis of the ternary samples in the region Lu-Lu_5Sn_3-V showed three phases in equlibrium– Lu_5Sn_3, Lu, and V. This result was confirmed by EPMA data (Fig, 3, a).

Under used in our work conditions one ternary compound LuV_6Sn_6 with the $SmMn_6Sn_6$ structure type was confirmed (space group P6/mmm, a = 0.5503(2), c = 0.9171(4) nm).

Conclusions

Experimental study of the Lu-V-Ge and Lu-V-Sn ternary systems was performed at 870 K. Analysis of the studied systems showed a significant influence of nature of *p*-element on interaction with lutetium and vanadium. Similarly to the R-M-Sn systems where M = Mn, Fe, and Co, the R-V-Sn systems (R=Gd, Er, Lu) are characterized by formation of the ternary RV_6Sn_6 intermetallics which crystallize in the $SmMn_6Sn_6$ type structure (space group *P*6/*mmm*, R=Gd, Dy, Ho, Er, Tm, Lu) or $HfFe_6Ge_6$-type (space group *P*6/*mmm*, R= Y) [5]. In contrast to the systems with Sn the germanides with stoichiometry RM_6Ge_6 are formed with M=Cr, Mn, Fe, and Co, while in the R-V-Ge systems were not found.

On the other hand the formation of the $RM_{1-x}Ge_2$ compounds is a feature of the R−{Mn, Fe, Co, Ni, Cu}−Ge systems. Germanides with the general composition $RM_{1-x}Ge_2$ ($CeNiSi_2$ structure type, space group *Cmcm*) have been reported to form with various *d*-electron transition metals [26]. The $CeNiSi_2$ type structure of these compounds can be described as the result of *d*-metal intercalations into the RGe_2 binaries with $ZrSi_2$-type. Intercalation of M-metal in square pyramidal sites of the $ZrSi_2$ type of the RGe_2 binaries results in the formation of an additional 4*c* position in the structure which corresponds to the ternary $CeNiSi_2$

structure type. This intercalation depends on the relative size of the M atoms resulting in non-stoichiometry with vacancies in M-metal sites or the statistical mixture of the transition metal and germanium atoms. In the Y–V–Ge system the ternary compound $YV_{0.16}Ge_{1.54}$ with $CeNiSi_2$-type was found [6]. In case of the studied Lu-V-Ge system the $LuGe_2$ binary directly belongs to the $ZrSi_2$-type and, consequently in the ternary region insertion of the V atoms results in the formation of the partially defect $LuV_{0,15}Ge_2$ phase which corresponds to the limit composition of the LuV_xGe_2 interstitial solid solution.

Similarly to the Lu-V-Ge system a formation of interstitial solid solutions based on the RSn_2 (R- Gd-Lu) binary compounds with $ZrSi_2$ structure was observed in the most studied R-M-Sn system where M = Fe, Co, Ni, and Cu [27-30]. In the case of the R-V-Sn systems the solubility of V in the RSn_2 binaries is only up to 2 at. % which may be caused by higher atomic radii of V (r_V= 0.134 nm) comparing with Fe, Co, Ni or Cu.

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Потрійні системи Lu-V-{Ge, Sn}

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Ізотермічні перерізи діаграм стану потрійних систем Lu–V–Ge і Lu-V-Sn побудовані за температури 870 K в повному концентраційному інтервалі методами рентгенофазового, рентгеноструктурного і мікроструктурного аналізів. В системі Lu-V-Ge на основі бінарної сполуки Lu_5Ge_3 (структурний тип Mn_5Si_3) встановлено утворення твердого розчину заміщення $Lu_5Ge_{3-x}V_x$ до вмісту 6 ат. % V. Включення атомів V в структуру бінарного германіду $LuGe_2$ (структурний тип $ZrSi_2$, до вмісту 5 ат. % V) приводить до утворення тернарної фази $LuV_{0,15}Ge_2$ (структурний тип $CeNiSi_2$, просторова група *Cmcm*, $a = 0{,}40210(4)$, $b = 1{,}5661(1)$, $c = 0{,}38876(3)$ нм), яка відповідає граничному складу твердого розчину включення LuV_xGe_2. Взаємодія компонентів у системі Lu-V-Sn за температури дослідження характеризується утворенням тернарної сполуки LuV_6Sn_6 (структурний тип $SmMn_6Sn_6$, просторова група *P*6/*mmm*, $a = 0{,}5503(2)$, $c = 0{,}9171(4)$ нм).

Ключові слова: інтерметаліди, потрійна система, фазові рівноваги, тверді розчини.