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CRYSTAL STRUCTURE OF THE NEW SILICIDE $Lu_3Ni_{11.74(2)}Si_4$

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The new ternary silicide $Lu_3Ni_{11.74(2)}Si_4$ was synthesized from the elements by arc-melting and its crystal structure was determined by the single-crystal X-ray diffraction. The compound crystallizes in the $Sc_3Ni_{11}Ge_4$-type: Pearson symbol *hP*37.2, space group $P6_3/mmc$ (No. 194), a = 8.0985(16), c = 8.550(2) Å, Z = 2; R = 0.0244, wR = 0.0430 for 244 reflections. The silicide $Lu_3Ni_{11.74(2)}Si_4$ is new member of the $EuMg_{5.2}$-type structure family.

K e y w o r d s: intermetallics, silicide, crystal structure, single-crystal X-ray diffraction.

INTRODUCTION. Recently, ternary systems consisting of silicon with rare earth and transition metals attracted much attention mainly due to the phases with various crystals structures and interesting physical properties. The majority of such systems were studied only to identify isostructural series of compounds, for example the ternary system Lu-Ni-Si. The isothermal section of this system have been yet constructed, but during the exploration of it, the existence of nine ternary compounds has been found (Table 1) [1-3]. All known compounds in the Lu-Ni-Si system form in the region up to 33.3 at. % Lu. However, the formation of the greater number of ternary compounds should be expected considering the results of investigations of the related ternary system. Thus, recently the ternary compound $Dy_3Ni_{11.83}Si_{3.98}$ ($Sc_3Ni_{11}Ge_4$-type, Pearson symbol *hP*38, space group $P6_3/mmc$ (No. 194), a = 8.1990(7), c = 8.6840(7) Å) [4] has been found. This fact prompted us to search for an isostructural compound in the Lu-Ni-Si system. Our expectations were met and new phase with a composition ~ $Lu_3Ni_{11}Si_4$ was found.

Therefore, in the present paper we describe the results of the crystal structure investigation of the new rare-earth silicide using X-ray single-crystal diffraction data.

EXPERIMENT AND DISCUSSION OF THE RESULTS. A sample of nominal composition $Lu_{16.7}Ni_{61.1}Si_{22.2}$ was synthesized from high-purity elements (Lu ≥ 99.9 wt.%, Ni ≥ 99.999 wt.%, and Si ≥

Table 1
Crystallographic data of the ternary compounds in the system Lu-Ni-Si

Compound	Stucture type	Pearson symbol	Space group	Lattice parameters, Å			Ref.
				a	*b*	*c*	
$LuNi_{10}Si_2$	$Nd(Mn_{0.5}Fe_{0.5})Fe_8$	*tI*26	*I*4/*mmm*	8.164	-	4.650	1, 2
$LuNi_5Si_3$	YNi_5Si_3	*oP*36	*Pnma*	18.49	3.739	6.710	1, 2
$LuNi_2Si_2$	$CeAl_2Ga_2$	*tI*10	*I*4/*mmm*	3.905	-	9.495	1, 2
$LuNiSi_3$	$SmNiGe_3$	*oS*20	*Cmmm*	3.8827	20.8179	3.89111	3
$Lu_3Ni_6Si_2$	$Ce_3Ni_6Si_2$	*cI*44	*Im*-3*m*	8.659	-	-	1, 2
$Lu_2Ni_3Si_5$	$Lu_2Co_3Si_5$	*mS*40	*C*2/*c*	11.032	11.942	5.919	1, 2
					β=120.18°		1, 2
$LuNiSi_2$	$CeNiSi_2$	*oS*16	*Cmcm*	3.851	15.810	3.851	1, 2
LuNiSi	TiNiSi	*oP*12	*Pnma*	6.67857	4.09340	7.11618	3
$LuNi_{0.61}Si_{1.39}$	AlB_2	*hP*3	*P*6/*mmm*	3.94594	-	3.87276	3

99.999 wt.%) by arc-melting under a purified argon atmosphere, using Ti as a getter and a tungsten electrode. The alloy was remelted two times to ensure homogeneity. The ingot was annealed at 600°C in an evacuated quartz ampoule for 720 h and subsequently quenched in cold water. The weight loss during the preparation of the sample was less than 1 % of the total mass, which was 2 g.

The single crystals were selected by mechanical fragmentation from the sample. Laue and rotation diffraction patterns of selected single crystals showed hexagonal symmetry with lattice parameters $a \sim 8.2$ and $c \sim 8.7$ Å. Integrated intensities measured with graphite-monochroma-tized Mo $K\alpha$ radiation on KM-CCD diffractometer confirmed the hexagonal lattice and the systematic extinctions were in agreement with the space groups $P6_3mc$, P-62c, and $P6_3/mmc$. The structure type $Sc_3Ni_{11}Ge_4$ [5] was assigned and the structure was refined using the program SHELXL (full-matrix least-squares refinement on F^2) [6] with anisotropic displacement parameters for all of the atoms.

The chemical composition of the selected crystal was checked with a field-emission scanning electron microscope (FEINovaNano SEM 230) equipped with an EDS analyzer (EDAX GenesisXM4). The experimentally determined composition of the grain (19±2 at% La: 69±2 at% Ni: 12±2 at% Si) is rather close to the composition calculated from the structure refinements.

The crystallographic data and details of the data collection from the single crystal of new silicide are listed in the Table 2. The reasonable values of the residual factors of the structure reliability confirmed that these compounds indeed adopt the $Sc_3Ni_{11}Ge_4$-type, which belongs to the $EuMg_{5.2}$ family. A final electron-density difference map was flat and did not reveal any significant residual peaks. The coordinates and displacement parameters of the atoms are presented in Table 3 and 4.

It should be noted, that structures related to the $EuMg_{5.2}$-type are characterized

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Experimental details and crystallographic data for the $Lu_3Ni_{11.74(2)}Si_4$ compound

Refined composition	$Lu_3Ni_{11.74(2)}Si_4$
M_r, g/mole	2644.24
Structure type	$Sc_3Ni_{11}Ge_4$
Space group	$P6_3/mmc$
Z; Pearson symbol	2; $hP37.2$
Unit-cell parameters, Å	a = 8.0985(16), c = 8.550(2)
Cell volume, Å^3	485.65(18)
Calculated density, g cm^{-3}	9.041
Absorption coefficient μ, mm^{-1}	52.609
F (000)	1191
θ range for data collection	2.90-29.76
Limiting indices	$-11 \le h \le 10$, $-11 \le k \le 10$, $-8 \le l \le 11$
Reflections collected / unique / with $I > 2\sigma(I)$	5249 / 291 / 244
Refined parameters	28
Extinction coefficient	0.00028(6)
$R1$ / w$R2$ for $I > 2\sigma(I)$	0.0244 / 0.0430
$R1$ / w$R2$ (all data)	0.0380 / 0.0455
Goodness-of-fit on F^2	1.082

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Atomic coordinates, equivalent displacement parameters and site occupancies for the $Lu_3Ni_{11.74(2)}Si_4$ compound

Atom	Wyckoff position	Occupancy	x	y	z	U_{eq}, Å^2
Lu	6*h*	1	0.18984(4)	0.37968(4)	1/4	0.00729(19)
Ni1	12*k*	1	0.16195(4)	0.32390(17)	0.58555(14)	0.0065(3)
Ni2	6*h*	1	0.56331(12)	0.1266(2)	1/4	0.0065(4)
Ni3	4*f*	1	1/3	2/3	0.0016(2)	0.0057(4)
Ni4	2*b*	0.735(15)	0	0	1/4	0.0156(17)
Si1	6*g*	1	1/2	0	0	0.0049(7)
Si2	2*a*	1	0	0	0	0.028(2)

by channels along the *c* direction, the occupation of which varies from one structure to another (sites 4*e*, 2*b* and 2*a*) and generally presents some kind of disorder. Different models of occupations were tested for the investigated structure. The best result was obtained for fully occupied site 2*a* by Si atoms and 2*b* partially occupied by Ni atoms.

These positions are partially occupied by Ge atoms in the structure of the prototype $Sc_3Ni_{11}Ge_4$. Hence, the partial occupation of the 2*b* site and full occupations of all other sites in the structure of our silicide lead to the refined composition $Lu_3Ni_{11.74(2)}Si_4$ for the investigated crystal.

Interatomic distances (δ), reduced values of interatomic distances ($\Delta = 100(\delta - \sum r)/\sum r$, where $\sum r$ is the sum of the respective atomic radii), and coordination numbers (CN) of the atoms for $Lu_3Ni_{11.74(2)}Si_4$ are listed in Table 5 (values of the atomic radii are taken from [7]:

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Anisotropic displacement parameters (Å^2) for the $Lu_3Ni_{11.7}Si_4$ compound

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Lu	0.0064(2)	0.0117(3)	0.0056(3)	0	0	0.00585(17)
Ni1	0.0058(5)	0.0063(6)	0.0075(5)	0.0007(4)	0.0003(2)	0.0032(3)
Ni2	0.0067(6)	0.0058(9)	0.0068(8)	0	0	0.0029(4)
Ni3	0.0048(6)	0.0048(6)	0.0074(10)	0	0	0.0024(3)
Ni4	0.0088(18)	0.0088(18)	0.029(3)	0	0	0.0044(9)
Si1	0.0041(12)	0.0041(17)	0.0065(18)	0.0023(12)	0.0011(6)	0.0020(9)
Si2	0.006(2)	0.006(2)	0.071(6)	0	0	0.0030(11)

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Interatomic distances (*d*, Å), Δ values (Δ = 100(*d*-Σ*r*)/Σ*r*, where Σ*r* is the sum of the respective atomic radii [11]) and atomic coordination numbers (CN) for the $Lu_3Ni_{11.74(2)}Si_4$ compound

Atom		*d* (Å)	Δ (%)	Atom		*d* (Å)	Δ (%)
Lu	1 Ni4	2.6629(8)	-10.5	Ni3	2 Si1	2.3376(5)	-3.0
CN = 19	1 Ni2	2.8220(13)	-5.1	CN = 12	1 Si1	2.3383(3)	-3.0
	1 Ni2	2.8221(16)	-5.1		1 Ni1	2.5164(14)	1.5
	4 Ni1	2.8599(15)	-3.8		2 Ni1	2.5171(14)	1.5
	2 Ni1	2.8955(14)	-2.6		2 Ni2	2.5941(17)	4.6
	2 Ni3	2.9262(14)	-1.6		1 Ni2	2.5945(17)	4.6
	4 Si1	3.0627(5)	5.5		1 Lu	2.9258(14)	-1.6
	2 Si2	3.4147(7)	17.6		2 Lu	2.9262(13)	-1.6
	2 Lu	3.4862(7)	0.5	Ni4	2 Si2	2.1375(5)	-11.3
Ni1	1 Si2	2.3865(13)	-1.0	CN = 11	3 Lu	2.6629(7)	-10.5
CN = 13	2 Si1	2.4818(14)	3.0		6 Ni1	2.6716(9)	-1.0
	1 Ni3	2.5171(14)	1.5	Si1	2 Ni2	2.3146(7)	-4.0
	1 Ni2	2.5244(20)	1.8	CN = 12	2 Ni3	2.3376(5)	-3.0
	1 Ni2	2.5245(12)	1.8		4 Ni1	2.4818(14)	3.0
	1 Ni4	2.6716(13)	7.7		4 Lu	3.0627(6)	5.5
	2 Ni1	2.7020(12)	9.0	Si2	2 Ni4	2.1375(5)	-11.3
	1 Ni1	2.8121(18)	13.4	CN = 14	6 Ni1	2.3865(8)	-1.0
	2 Lu	2.8599(14)	-3.8		6 Lu	3.4147(7)	17.6

End of the Table 5

Atom		d (Å)	Δ (%)	Atom		d (Å)	Δ (%)
	1 Lu	2.8955(14)	-2.6	CN = 12	2 Ni1	2.5245(16)	1.8
Ni2	2 Si1	2.3146(7)	-4.0		2 Ni3	2.5941(17)	4.6
CN = 12	2 Ni2	2.5113(24)	1.3		1 Lu	2.8220(19)	-5.1
	2 Ni1	2.5244(14)	1.8		1 Lu	2.8221(10)	-5.1

r(Lu) = 1.734 Å, *r*(Ni) = 1.246 Å, *r*(Si) = 1.170 Å). Most of the interatomic distances are in good agreement with the atomic sizes. The interatomic distances between Lu-Si2 and Ni1-Ni1 are somewhat larger in comparison with the sum of the respective atomic radii. The largest decrease of interatomic distances is observed between Ni4 and Lu or Si2 atoms. It does not exceed 12 % of the sum of the atomic radii of respective atoms. Moreover, within $Lu_3Ni_{11.74(2)}Si_4$ structure the silicon atoms are not in contact with each other.

The projection of the unit cell of $Lu_3Ni_{11.74(2)}Si_4$ compound and the coordination polyhedrons (CP) of atoms are shown in Fig. 1. CP are similar to corresponding polyhedrons of atoms in the $Sc_3Ni_{11}Ge_4$ structure [5] and typical for the most of the ternary compounds in the *RE*-Ni-Si systems. The lutetium atoms have the largest coordination number (CN = 19) and its polyhedron [Lu($Lu_2Ni_{11}Si_6$)] can be described as pentagonal prisms with seven capping atoms. A distorted icosahedron with one additional atom [Ni1($Lu_3Ni_7Si_3$)] is the CP of the Ni1 atoms (CN = 13). The coordination polyhedrons of the Ni2 and Ni3 atoms (CN = 12 for both) are similar and can be described as distorted icosahedrons, [Ni2($Lu_2Ni_8Si_2$)] and [Ni3($Lu_3Ni_6Si_3$)], respectively. A trigonal prism [Ni4($Lu_3Ni_6Si_2$)] with lateral sides and bases capped by additional atoms is the coordination polyhedron of the Ni4 atom (CN = 11). Only lutetium and nickel atoms form the coordination polyhedrons of the Si1 and Si2 atoms. The Si1 atoms have 12 neighbors and the polyhedron [Si1(Lu_4Ni_8)] can be described as a distorted icosahedron. A distorted rhombicdo-decahedron is the CP of Si2 atoms [Si2(Lu_6Ni_8)] (CN = 14).

The $Lu_3Ni_{11.74(2)}Si_4$ structure can be considered as a packing of the Si1 polyhedrons [Si1(Lu_4Ni_8)] (Fig. 2). Such polyhedrons are connected to each other by com-

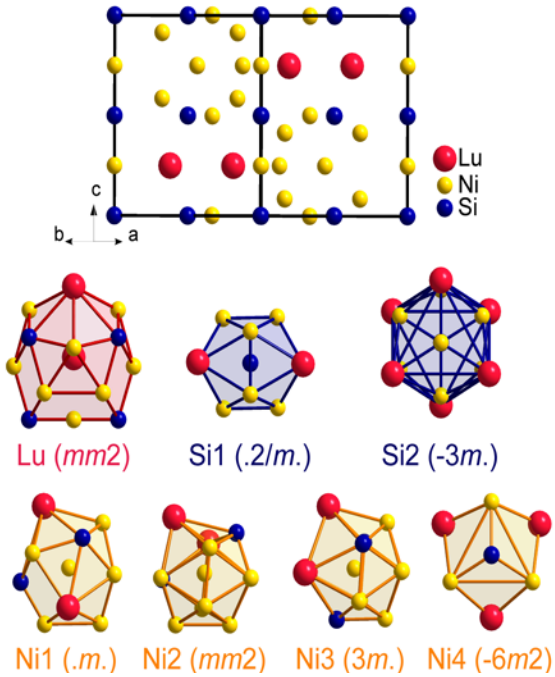

Fig. 1. The unit cell of the $Lu_3Ni_{11.74(2)}Si_4$ structure and coordination polyhedrons of the atoms. The site symmetries are indicated.

mon triangular faces and form column along the *c* direction with small holes between adjacent columns of icosahedrons from one unit cell. The columns from the neighboring unit cells, in turn, are combined into a network with twisted triangular cylinder, in the center of which the nickel and silicon atoms with coordination (0 0 *z*) are situated.

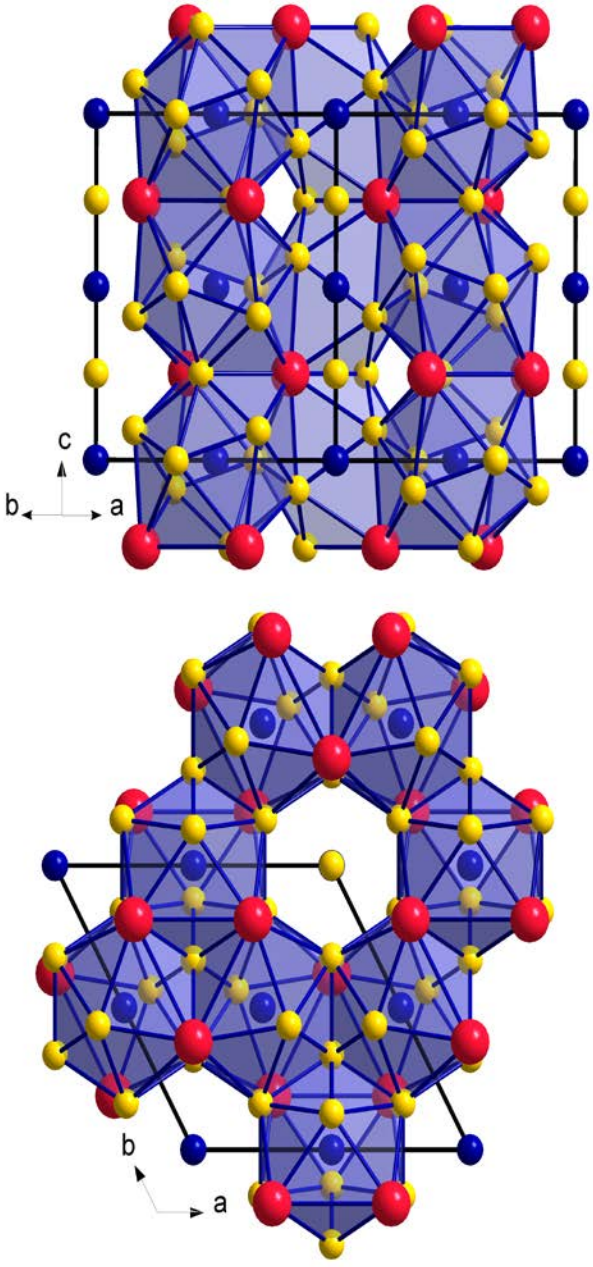

Fig. 2. The arrangement of the Si1 polyhedrons in the $Lu_3Ni_{11.74(2)}Si_4$ structure.

Alternatively, the structure of $EuMg_{5.2}$ type can be considered as a packing of clusters [Mg_{17}], which are dimers of face-sharing hypho-icosahedra with 14 vertices (Fig. 3) [8]. These dimers connect by common Mg atoms into the zigzag chain along *c* direction. The triangle from Eu atoms are located between the "links" of this chain. The space between zigzag chains are occupied by the linear chains from the Mg atoms. The similar units can be seen in the structure of $Sc_3Ni_{11}Ge_4$,-type as well as $Lu_3Ni_{11.74(2)}Si_4$ compound. The clusters of the first one are built from 11 Ni atoms and 6 Ge atoms, and 11 Ni atoms and 6 Si atoms form the clusters in the structure of our compound. The triangle from the Sc atoms in case of $Sc_3Ni_{11}Ge_4$ and the Lu atoms in case of $Lu_3Ni_{11.74(2)}Si_4$ are instead of triangle from the Eu atoms. The only difference in these two compounds are in the linear chains. In the structure of $Lu_3Ni_{11.74(2)}Si_4$ the Ni and Si atoms from such chain, while the atoms of only one type form the chain in the case of $Sc_3Ni_{11}Ge_4$ and $EuMg_{5.2}$. The difference in the occupation of channels along the *c* direction is as well observed in other related structures, for example, in the $Sc_3Ni_{11}Si_4$-type, which is realized in the ternary systems R-Cu-Si (*R* = Y, Gd-Lu) or in the $Gd_3Ru_4Al_{12}$-type. The latter one has four representatives among ternary silicides of nickel and rare-earth metals $R_3Ni_{12}Si_4$ (*R* = Gd, Ho, Er, Tm) [9, 10]. The Table 6 summarizes the data for the related structure types with space group $P6_3/mmc$ that belong to the family of the structure type $EuMg_{5.2}$.

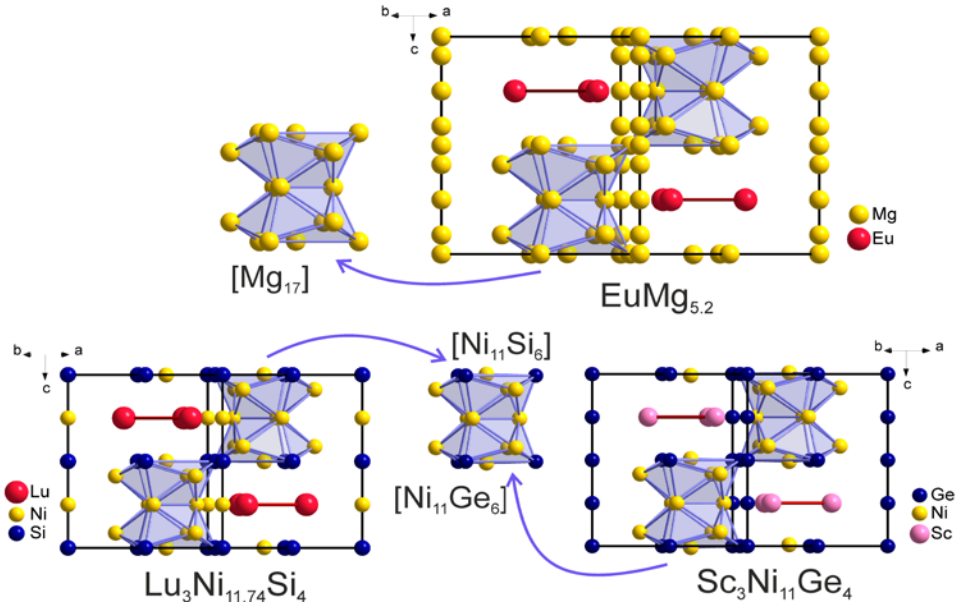

Fig. 3. The arrangement of the clusters in the $EuMg_{5.2}$, $Sc_3Ni_{11}Ge_4$ and $Lu_3Ni_{11.74(2)}Si_4$ structures.

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Atom distribution in related structure types with space group *P*6$_3$/*mmc*.

Structure type	Unit-cell parameters, Å	Wyckoff position								Refs
		6*h* (*x*2*x*1/4)	12*k* (*x* 2*x z*)	6*h* (*x* 2*x* 1/4)	6*g* (1/2 0 0)	4*f* (1/3 2/3 *z*)	4*e* (00*z*)	2*b* (0 0 1/4)	2*a* (0 0 0)	
$EuMg_{5.2}$	a = 10.395; c = 10.746	Eu	Mg	Mg	Mg	Mg	$Mg_{0.26}$	$Mg_{0.77}$	$Mg_{0.40}$	11
$ErZn_5$	a = 5.276; c = 4.233	Er	Zn	Zn	Zn	Zn	-	-	Zn	12
$Sc_3Cu_{7.5}Al_{7.5}$	a = 8.485; c = 8.859	Sc	$Al_{0.55}Cu_{0.45}$	$Al_{0.56}Cu_{0.44}$	$Cu_{0.74}Al_{0.26}$	$Al_{0.86}Cu_{0.14}$	-	$Cu_{0.61}$	$Cu_{0.389}$	13
$Gd_3Ru_4Al_{12}$	a = 8.814; c = 9.569	Gd	Al	$Al_{0.96}Ru_{0.04}$	Ru	Al	-	Al	Ru	14
$Ce_3Ag_{4.7}Mg_{11.3}$	a = 9.809; c = 10.448	Ce	$Mg_{0.94}Ag_{1.06}$	$Mg_{0.89}Ag_{0.12}$	$Ag_{0.97}Mg_{0.03}$	$Mg_{0.95}Ag_{0.05}$	-	$Mg_{0.95}Ag_{0.05}$	Ag	15
$Sc_3Ni_{11}Si_4$	a = 8.024; c = 8.429	Sc	Ni	Ni	Si	Ni	-	Si	-	16
$Sc_3Ni_{11}Ge_4$	a = 8.130; c = 8.505	Sc	Ni	Ni	Ge	Ni	-	$Ge_{0.72}$	$Ge_{0.28}$	5
$Dy_3Ni_{11.83}Si_4$	a = 8.199; c = 8.684	Dy	Ni	Ni	Si	Ni	-	$Ni_{0.828}$	$Si_{0.985}$	4
$Lu_3Ni_{11.74}Si_4$	a = 8.099; c = 8.550	Lu	Ni	Ni	Si	Ni	-	$Ni_{0.74}$	Si	

* this work

CONCLUSIONS. The new silicide $Lu_3Ni_{11.74(2)}Si_4$ has been synthesized and its structure has been investigated using single-crystal X-ray diffraction. The compound crystallizes in the $Sc_3Ni_{11}Ge_4$ structure type which belongs to the large family of structures related to the $EuMg_{5.2}$ type. The structural feature of $Lu_3Ni_{11.74(2)}Si_4$ compound is the partially occupied nickel position which leads to deviation of the composition of the compound from the composition of the structural type.

КРИСТАЛЛИЧЕСКАЯ СТРУКТУРА НОВОГО СИЛИЦИДА $Lu_3Ni_{11,74(2)}Si_4$

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Методом электро-дуговой плавки синтезировано новый тернарный силицид $Lu_3Ni_{11,74(2)}Si_4$. Кристаллическую структуру исследовано методом монокристальной рентгеновской дифракции. Соединение кристаллизируется в структурном типе $Sc_3Ni_{11}Ge_4$: символ Пирсона *hP*37.2, пространственная группа $P6_3/mmc$ (№ 194), a = 8,0985(16), c = 8,550(2) Å, Z = 2; R = 0,0244, wR = 0,0430 для 244 рефлексов. Атомы в исследованом соединении имеют следующие координационные числа: 19 для лютеция, 11-1для никеля, и 12, 14 для кремния.

К л ю ч е в ы е с л о в а: интерметаллиды, силицид, кристаллическая структура, монокристальные рентгеновские исследования.

КРИСТАЛІЧНА СТРУКТУРА НОВОГО СИЛИЦИДУ $Lu_3Ni_{11,74(2)}Si_4$

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Методом електродугового плавлення синтезовано новий тернарний силіцид $Lu_3Ni_{11,74(2)}Si_4$. Кристалічну структуру досліджено методом монокристальної рентгенівської дифракції. Сполука кристалізується в структурному типі $Sc_3Ni_{11}Ge_4$: символ Пірсона *hP*37.2, просторова група $P6_3/mmc$ (№ 194), a = 8,0985(16), c = 8,550(2) Å, Z = 2; R = 0,0244, wR = 0,0430 для 244 рефлексів. Новий силіцид характеризується частковим заповненням кристалографічного положення 2*b*, яке у структурі прототипу $Sc_3Ni_{11}Ge_4$ частково заповнено атомами германію. Міжатомні віддалі добре узгоджуються з сумою радіусів відповідних атомів. Атоми в дослідженій сполуці мають такі координаційні числа: 19 у лютецію, 11-1 у нікелю, та 12, 14 у кремнію. Координаційні многогранники є характерними для сполук такого типу. Атоми лютецію центрують пентагоналльну призму з сімома додатковими атомами. Атоми Ni1, Ni2 та Ni3 мають координаційні поліедри у вигляді спотворених ікосаедрів. Атом Ni4 центрує тригональну призму з п'ятьома додатковими атомами. До координаційної сфери атомів силіцію входять виключно атоми лютецію та нікелю, а координаційні поліедри мають фо-

рму спотвореного ікосаедра у випадку атомів Si1, та спотвореного ромбічного додекаедру – у Si2.

К л ю ч о в і с л о в а: інтерметаліди силіцид, кристалічна структура, монокристальні рентгенівські дослідження.

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