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Isothermal section of the Gd-Mn-Ge ternary system at 1070 K

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Interaction between the components in the Gd–Mn–Ge ternary system was studied at 1070 K over the whole concentration range using scanning electron microscopy, energy dispersive X-ray microanalysis and X-ray diffractometry. At 1070 K Gd–Mn–Ge system is characterized by existence of six ternary compounds: $GdMn_4Ge_2$ ($ZrFe_4Si_2$ structure type, space group $P4_2/mnm$, a = 0.7645(4) nm, c = 0.3953(3) nm), GdMnGe (TiNiSi structure type, space group $Pnma$, a = 0.7129(2), b = 0.4166(2), c = 0.8201(2) nm), $Gd_3Mn_4Ge_4$ ($Gd_3Cu_4Ge_4$ structure type, space group $Immm$, a = 1.4023(4), b = 0.7121(3), c = 0.4212(3) nm), $GdMn_2Ge_2$ ($CeAl_2Ga_2$ structure type, space group $I4/mmm$, a = 0.40326(6), c = 1.0884(2) nm), $GdMn_6Ge_6$ ($MgFe_6Ge_6$ structure type, space group $P6/mmm$, a = 0.52377(5), c = 0.8182(1) nm) and $GdMn_{1-x}Ge_2$ ($CeNiSi_2$ structure type, space group $Cmcm$, a = 0.41612(8)-0.41590(7), b = 1.6123(3)-1.6119(1), c = 0.40336(8)-0.40290(6) nm). For the $GdMn_{1-x}Ge_2$ compound the homogeneity range was determined (x = 0.70-0.64).

Keywords: Intermetallics, ternary system, Phase equilibria, Crystal structure.

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Introduction

One of the stages on the way to the creation of new materials is the study of the component interaction in the metallic systems and the construction of appropriate phase equilibrium diagrams. This information makes it possible to establish the concentration and temperature limits of the stability of the intermetallic compounds, their crystal structure peculiarities, and to investigate some physical properties. Intermetallics contained rare earths, manganese and *p*-elements attract special attention as potential candidates for searching of magnetic materials. Several of the RMn_6Ge_6 compounds (R-rare earth metal) are characterized by two ordering temperatures associated with antiferromagnetic ordering of the R and Mn sublattices [1, 2].

Analysis of the studied R-Mn-Ge ternary systems (R = Ce, Nd, Gd, Y, Tb, Er, Yb) [3-6] showed that most of the ternary germanides of rare earths and manganese form isostructural series RMn_2Ge_2 (R = Ce, Nd, Gd, Y, Tb, Er, Yb, $CeAl_2Ga_2$-type), RMn_6Ge_6 (R = Gd, Tb, Er, Yb, $MgFe_6Ge_6$-type), $RMn_{1-x}Ge_2$ (R = Nd, Gd, Y, Tb, Er, Yb, $CeNiSi_2$-type), RMnGe (R = Ce, Nd, PbFCl-type; R = Y, Nd, Gd, Tb, Er, Yb, TiNiSi-type). The ternary system Gd–Mn–Ge was previously studied at 970 K [3]. The authors report the formation of nine ternary compounds, the crystal structure of two of them has not been established.

The subject of the presented work consists of the experimental investigation of the Gd-Mn-Ge ternary system at 1070 K.

I. Experimental

To study the Gd-Mn-Ge ternary system the weighed amount of constituent elements (gadolinium, purity of 99.9 wt.%; manganese, purity of 99.99 wt.%; and germanium, purity of 99.999 wt.%) were melted in electric arc-furnace under high purity Ti-gettered argon atmosphere on a water-cooled copper crucible. The pieces of the as-cast buttons were annealed at 1070 K for 720 hours in evacuated silica tubes and then water quenched. To determine the phase composition the annealed samples were examined using X-ray powder diffraction (the experimental X-ray powder patterns were

compared with reference powder patterns of initial elements, binary compounds and known ternary germanides). Composition analysis was carried out using Tescan Vega 3 LMU scanning electron microscopes with a Link EDX system operated at 20 kV and 60 μA. At least five measurements were done for each phase in each sample to obtain an average composition value.

The diffraction data for the crystal structure refinements were collected at room temperature using STOE STADI P powder diffractometer (graphite monochromator, Cu$K_{\alpha 1}$ radiation, 6 – 110.625° 2θ range with scanning step 0.015°). Calculations of the crystallographic parameters were performed using the FullProf Suite program package [7].

II. Results and discussion

To study the phase relations in the Gd-Mn-Sn system and, particularly, precise the phase equlibria with some Gd-Ge binary compounds which are not taken into account in Ref. [3] we prepared and examined by X-ray phase and EPM analyses 43 binary and ternary alloys. During the phase analysis the existence of all binary phases in the boundary binary systems Gd-Mn, Gd-Ge and Mn-Ge was confirmed according to reported phase diagrams [8] and literature data about Gd-Ge binaries. In the Gd-Mn binary system we confirmed the presence of $GdMn_2$ ($MgCu_2$-type), Gd_6Mn_{23} (Th_6Mn_{23}-type), and $GdMn_{12}$ ($ThMn_{12}$-type) binaries [8]. At 1070 K three compounds – Mn_3Ge (Mg_3Cd-type), Mn_5Ge_2 (Mn_5Ge_2-type), Mn_5Ge_3 (Mn_5Si_3-type) were formed in Mn-Ge system that corresponds to the reported phase diagram [8]. In the Gd-Ge binary system the existence of binaries Gd_5Ge_3 (Mn_5Si_3-type), Gd_5Ge_4 (Sm_5Ge_4-type), $Gd_{11}Ge_{10}$ ($Ho_{11}Ge_{10}$-type), GdGe (TlI-type), Gd_3Ge_4 (Er_3Ge_4-type), $GdGe_{1.5}$ (AlB_2-type), and $GdGe_{1.63}$ ($ThSi_2$-type), was confirmed [8, 9, 10].

Isothermal section of the Gd−Mn−Ge system was constructed at 1070 K based on the results of X-ray phase analysis and scanning electron microscopy (Fig. 1). Phase compositions of the selected samples are presented in Table 1, electron micrographs of some alloys are shown in Fig. 2.

At the temperature of investigation six ternary compounds was identified, crystallographic characteristics of which are given in Table 2. Ternary phases with compositions ~$GdMn_6Ge_3$ and ~$Gd_3Mn_2Ge_3$ reported in Ref. [3] was not observed under used conditions. Phase analysis and electron probe microanalysis showed that ~$GdMn_6Ge_3$ sample belongs to three phase field and contains $GdMn_4Ge_2$, $GdMn_2Ge_2$ and Mn_5Ge_2 compounds in equilibrium (Fig. 2c). Sample at composition ~$Gd_3Mn_2Ge_3$ contains three phases: GdMnGe, $Gd_3Mn_4Ge_4$ and Gd_5Ge_4 (Fig. 2b). To check the formation of $Gd_4Mn_{0.64}Ge_7$ compound ($Sm_4Co_{0.64}Ge_7$-type) [3] we prepared sample at composition $Gd_{34}Mn_6Ge_{60}$. According to performed phase analysis sample $Gd_{34}Mn_6Ge_{60}$ annealed at 1070 K contains main phase $GdMn_{1-x}Ge_2$ ($CeNiSi_2$-type) in equilibrium with $GdGe_{1.5}$ binary (AlB_2-type).

Solubility of Mn in $Gd_{11}Ge_{10}$ and Gd_5Ge_4 binaries extends up to ~3 and ~4 at. %, respectively. Significant solubility of the third component in the binary compounds of Mn-Ge and Gd-Mn systems was not observed under used conditions.

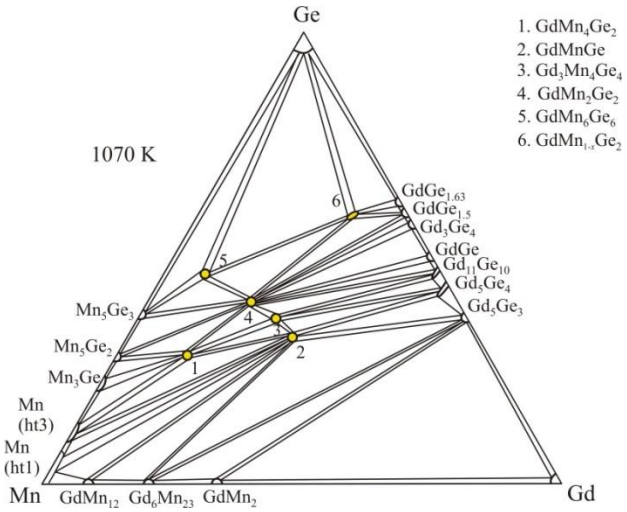

Fig. 1. Isothermal section of the Gd-Mn-Ge system at 1070 K.

An existence of the $GdMn_{1-x}Ge_2$ compound with $CeNiSi_2$-type was confirmed in our work according to Ref [11]. Data of X-ray phase and EPM analyses show a small homogeneity range within the content of Mn x = 0.70-0.64. The crystal structure refinements was performed for the $GdMn_{1-x}Ge_2$ compound, using the powder diffraction data of the $Gd_{30}Mn_{12}Ge_{58}$ sample (Fig. 3). The atomic coordinates and isotropic displacement parameters are gathered in Table 3.

The existence of the $GdMn_6Ge_6$ compound and its lattice parameters were reported in Ref. [12]. The detailed crystal structure refinements performed on $Gd_8Mn_{46}Ge_{46}$ sample confirmed a formation of $GdMn_6Ge_6$ compound with $MgFe_6Ge_6$-type (space group *P6/mmm*, a = 0.52377(5), c = 0.8182(1) nm) (Fig. 4). The presence of small amount (2.41 %) of $GdMn_{1-x}Ge_2$ compound was taken into account during crystal structure calculation. The final atomic parameters, refined to R_p = 0.0191, R_{wp} = 0.0244, R_{Bragg} = 0.0405 are listed in Table 4.

Analysis of the investigated in our work Gd-Mn-Ge system and known in the literature ternary germanides showed that the formation of the RMn_6Ge_6 compounds ($MgFe_6Ge_6$ structure type), RMn_2Ge_2 ($CeAl_2Ga_2$-type), RMnGe (TiNiSi, ZrNiAl structure types) and $RMn_{1-x}Ge_2$ with structure of $CeNiSi_2$ type are observed for all rare earths of Yttrium subgroup. Similarly to isotypic $RM_{1-x}Ge_2$ germanides with different *d*-metal (Cr, Fe, Co, Ni, Cu) the defect in crystallographic position of the transition metal was observed for $RMn_{1-x}Ge_2$ compounds. Equatomic compounds RMnGe where R = Tm, Yb are characterized by two polymorphic modifications – low-temperature ZrNiAl and high-temperature TiNiSi structure types [13, 14]. $R_3Mn_4Ge_4$ compounds with $Gd_3Cu_4Ge_4$-type are realized with Gd and Tb only.

Table 1.

Phase composition and EPMA data for individual samples of the Gd-Mn-Ge system

Nominal composition/EPMA data, at. %	Phase	Structure type	Lattice parameters, nm			EPMA, at. %		
			a	*b*	*c*	Gd	Mn	Ge
$Gd_{33}Mn_{62}Ge_5$	Gd_5Ge_3	Mn_5Si_3	0.8541(3)		0.6437(3)			
	$GdMn_2$	$MgCu_2$	0.7771(2)					
	Gd_6Mn_{23}	Th_6Mn_{23}	1.2527(4)					
$Gd_{20}Mn_{70}Ge_{10}$	$GdMn_{12}$	$ThMn_{12}$	0.8629(4)		0.4801(3)			
	Gd_6Mn_{23}	Th_6Mn_{23}	1.2526(4)					
	GdMnGe	TiNiSi	0.7125(4)	0.4164(3)	0.8187(4)			
$Gd_{20}Mn_{60}Ge_{20}$	GdMnGe	TiNiSi	0.7123(3)	0.4169(2)	0.8202(3)			
	(β-Mn)	Mn	0.6350(1)					
$Gd_{60}Mn_{13}Ge_{27}$ $Gd_{62.13}Mn_{12.09}Ge_{25.78}$	Gd_5Ge_3	Mn_5Si_3	0.8567(9)		0.6442(2)	66.52	0.35	33.13
	$GdMn_2$	$MgCu_2$	0.7769(2)			31.98	68.02	
	(Gd)	Mg	0.3568(3)		0.5774(5)	100.0		
$Gd_{15}Mn_{60}Ge_{25}$	GdMnGe	TiNiSi	0.7126(3)	0.4166(2)	0.8189(3)			
	$GdMn_4Ge_2$	$ZrFe_4Si_2$	0.7644(4)		0.3957(3)			
	(γ-Mn)	Cu	0.3722(3)					
$Gd_{15}Mn_{55}Ge_{30}$ $Gd_{13.65}Mn_{54.69}Ge_{31.66}$	$GdMn_2Ge_2$	$CeAl_2Ga_2$	0.4029(2)		1.0885(3)	20.48	40.98	38.54
	$GdMn_4Ge_2$	$ZrFe_4Si_2$	0.7643(3)		0.3958(4)	14.08	57.16	28.76
$Gd_{10}Mn_{60}Ge_{30}$ $Gd_{10.07}Mn_{59.78}Ge_{30.1}$	$GdMn_2Ge_2$	$CeAl_2Ga_2$	0.4030(3)		1.0883(4)	20.28	40.77	38.95
	$GdMn_4Ge_2$	Mn_5Si_3	0.7686(4)		0.3957(4)	14.08	57.97	27.95
	Mn_5Ge_2	Mn_5Ge_2	0.7195(4)		1.3073(5)	1.34	68.95	29.71
$Gd_{50}Mn_{15}Ge_{35}$ $Gd_{48.56}Mn_{14.67}Ge_{36.77}$	Gd_5Ge_3	Mn_5Si_3	0.8567(6)		0.6443(3)	64.91		35.09
	GdMnGe	TiNiSi	0.7124(4)	0.4166(3)	0.8188(4)	32.44	33.29	34.27
$Gd_{45}Mn_{20}Ge_{35}$ $Gd_{42.05}Mn_{20.05}Ge_{37.90}$	GdMnGe	TiNiSi	0.7124(4)	0.4166(3)	0.8192(4)	33.59	32.92	33.49
	Gd_5Ge_4	Sm_5Ge_4	0.7678(3)	1.4779(5)	0.7778(3)	54.68	2.98	42.34
$Gd_{20}Mn_{45}Ge_{35}$	$GdMn_2Ge_2$	$CeAl_2Ga_2$	0.4028(3)		1.0881(5)			
	$GdMn_4Ge_2$	$ZrFe_4Si_2$	0.7644(3)		0.3956(4)			
	$Gd_3Mn_4Ge_4$	$Gd_3Cu_4Ge_4$	1.4015(8)	0.7119(4)	0.4209(5)			
$Gd_{26}Mn_{36}Ge_{38}$ $Gd_{26.58}Mn_{36.45}Ge_{36.97}$	$GdMn_2Ge_2$	$CeAl_2Ga_2$	0.4029(3)		1.0883(5)	19.82	40.14	40.04
	$Gd_{11}Ge_{10}$	$Ho_{11}Ge_{10}$	1.0967(6)		1.6677(7)	46.70	2.59	50.71
	$Gd_3Mn_4Ge_4$	$Gd_3Cu_4Ge_4$	1.4019(7)	0.7120(4)	0.4207(5)	27.07	36.20	36.73
$Gd_{35}Mn_{30}Ge_{35}$ $Gd_{35.97}Mn_{30.50}Ge_{33.53}$	GdMnGe	TiNiSi	0.7124(3)	0.4168(3)	0.8193(4)	32.94	33.19	33.87
	$Gd_3Mn_4Ge_4$	$Gd_3Cu_4Ge_4$	1.4017(6)	0.7121(4)	0.4219(4)	27.12	37.62	35.26
	Gd_5Ge_4	Sm_5Ge_4	0.7678(3)	1.4779(5)	0.7778(3)	53.97	2.06	43.97
$Gd_{10}Mn_{50}Ge_{40}$ $Gd_{9.87}Mn_{49.04}Ge_{41.09}$	$GdMn_2Ge_2$	$CeAl_2Ga_2$	0.4031(3)		1.0898(5)	19.69	40.06	40.25
	$GdMn_6Ge_6$	$MgFe_6Ge_6$	0.5281(3)		0.8187(4)	8.12	45.87	46.01
	Mn_5Ge_3	Mn_5Si_3	0.7192(3)		0.5041(3)	0.21	66.08	33.71
$Gd_{30}Mn_{25}Ge_{45}$ $Gd_{30.68}Mn_{23.20}Ge_{46.12}$	$GdMn_2Ge_2$	$CeAl_2Ga_2$	0.4030(3)		1.0885(4)	19.82	40.57	39.61
	GdGe	TlI	0.4328(3)	1.0781(6)	0.3976(4)	49.34		50.66
	Gd_3Ge_4	Gd_3Ge_4	0.4102(2)	1.0741(5)	1.4343(5)	41.68	1.30	57.02
$Gd_{25}Mn_{28}Ge_{47}$ $Gd_{26.33}Mn_{27.43}Ge_{46.24}$	$GdMn_{1-x}Ge_2$	$CeNiSi_2$	0.4162(8)	1.6132(4)	0.4035(3)	30.40	11.32	58.28
	$GdMn_2Ge_2$	$CeAl_2Ga_2$	0.4028(3)		1.0889(4)	19.82	41.57	38.61
	$GdGe_{1.5}$	AlB_2	0.3969(2)		0.4204(3)	58.76	2.09	39.15
$Gd_{25}Mn_{25}Ge_{50}$ $Gd_{24.34}Mn_{27.00}Ge_{48.66}$	$GdMn_{1-x}Ge_2$	$CeNiSi_2$	0.4160(5)	1.6123(4)	0.4033(3)	29.49	11.73	58.78
	$GdMn_2Ge_2$	$CeAl_2Ga_2$	0.4027(3)		1.0888(4)	20.05	40.47	39.48
	$GdMn_6Ge_6$	$MgFe_6Ge_6$	0.5244(2)		0.8187(3)	7.86	46.43	45.71

Table 2.

Crystallographic characteristics of the compounds in the Gd−Mn−Ge system

Compound	Space group	Structure type	Lattice parameters, nm		
			a	*b*	*c*
$GdMn_4Ge_2$	*P*4$_2$/*mnm*	$ZrFe_4Si_2$	0.7645(4)	–	0.3953(3)
GdMnGe	*Pnma*	TiNiSi	0.7129(2)	0.4166(2)	0.8201(2)
$Gd_3Mn_4Ge_4$	*Immm*	$Gd_3Cu_4Ge_4$	1.4023(4)	0.7121(3)	0.4212(3)
$GdMn_2Ge_2$	*I*4/*mmm*	$CeAl_2Ga_2$	0.40326(6)	–	1.0884(2)
$GdMn_6Ge_6$	*P*6/*mmm*	$MgFe_6Ge_6$	0.52377(5)	–	0.8182(1)
$GdMn_{1-x}Ge_2$	*Cmcm*	$CeNiSi_2$	0.41612(8)-0.41590(7)	1.6123(3)-1.6119(1)	0.40336(8)-0.40290(6)

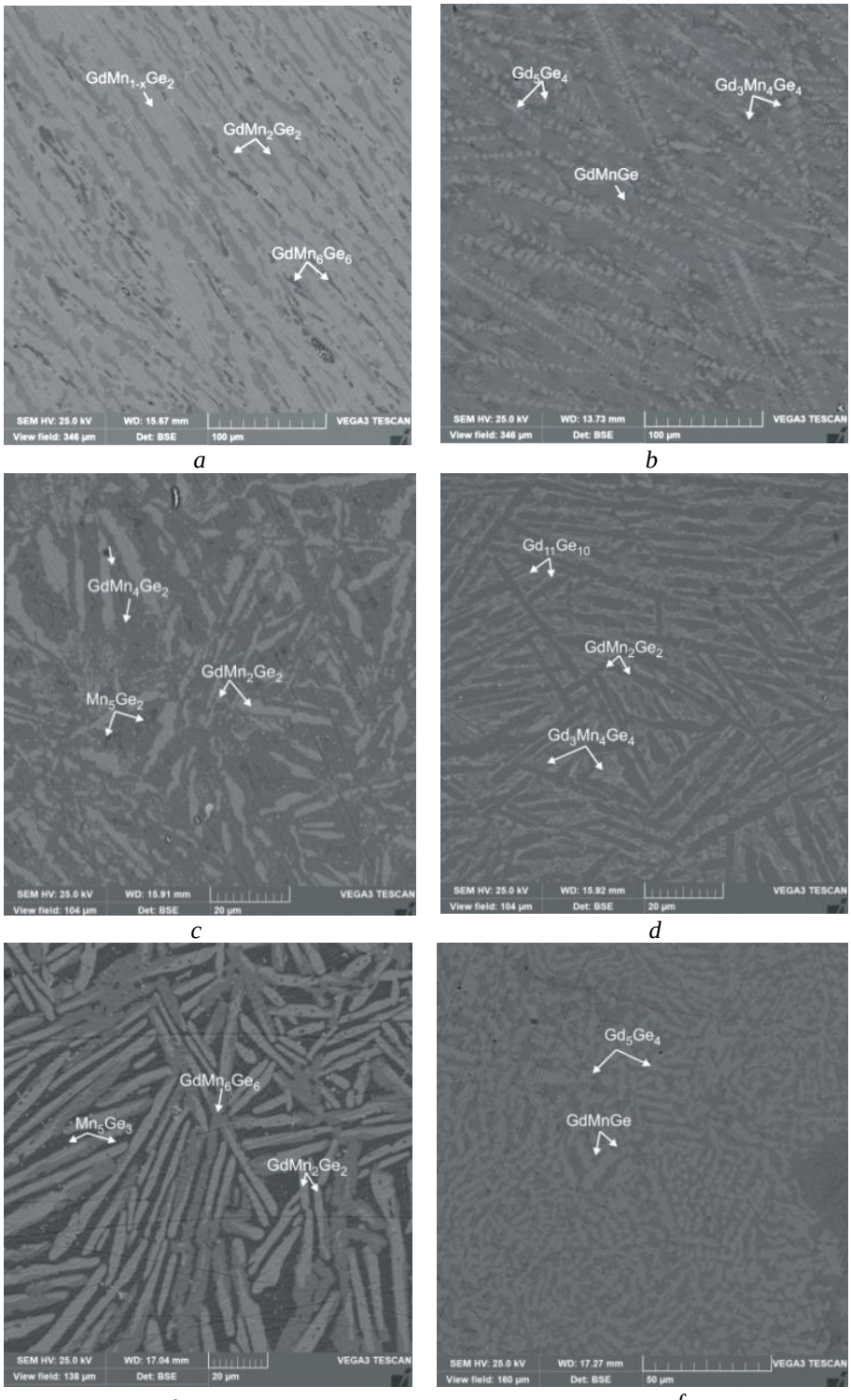

Fig. 2. Electron micrographs of the alloys: a) $Gd_{25}Mn_{25}Ge_{50}$; b) $Gd_{37}Mn_{26}Ge_{37}$; c) $Gd_{10}Mn_{60}Ge_{30}$; d) $Gd_{26}Mn_{36}Ge_{38}$; e) $Gd_{10}Mn_{50}Ge_{40}$; f) $Gd_{43}Mn_{20}Ge_{37}$.

Table 3.

Fractional atomic coordinates, site occupations (G) and isotropic displacement parameters B_{iso} for $GdMn_{0.36}Ge_2$ compound (R_p=0.0229, R_{wp}=0.0288, R_{Bragg}=0.0254)

Atom	Wyckoff position	*x*	*y*	*z*	G	$B_{iso.}\cdot 10^2$ (nm^2)
Gd	4*c*	0	0.1023(3)	1/4	1	0.14(7)
Mn	4*c*	0	0.3058(2)	1/4	0.36(1)	0.93(3)
Ge1	4*c*	0	0.4492(5)	1/4	1	1.62(3)
Ge2	4*c*	0	0.7493(5)	1/4	1	2.14(4)

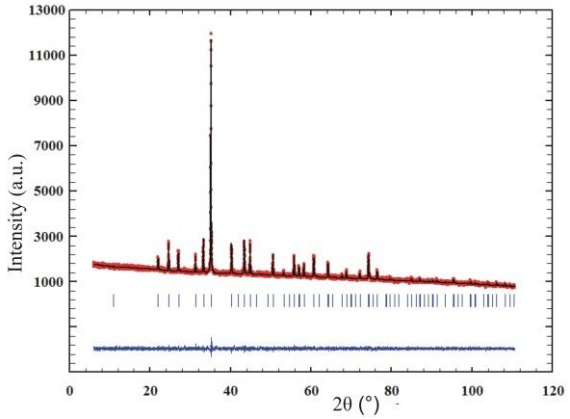

Fig. 3. The observed, calculated and difference X-ray patterns of $GdMn_{0.36}Ge_2$ compound.

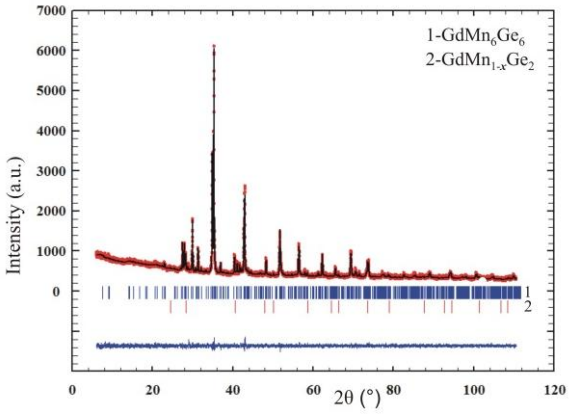

Fig. 4. The observed, calculated and difference X-ray patterns of $Gd_8Mn_{46}Ge_{46}$ sample.

Table 4.

Fractional atomic coordinates and isotropic displacement parameters B_{iso} for $GdMn_6Ge_6$ compound

Atom	Wyckoff position	*x/a*	*y/b*	*z/c*	$B_{iso.}\cdot 10^2$ (nm^2)
Gd	1*a*	0	0	0	0.87(6)
Mn	6*i*	1/2	0	0.2512(3)	0.66(3)
Ge1	2*e*	0	0	0.3470(4)	0.63(2)
Ge2	2*c*	1/3	2/3	0	0.46(1)
Ge3	2*d*	1/3	2/3	1/2	0.45(7)

Conclusions

Experimental study of the components interaction in the ternary Gd–Mn–Ge system at 1070 K over the whole concentration range showed the formation of six ternary germanides: $GdMn_4Ge_2$, GdMnGe, $Gd_3Mn_4Ge_4$, $GdMn_2Ge_2$, $GdMn_6Ge_6$ and $GdMn_{1-x}Ge_2$. It was found that all the ternary compounds are characterized by a point composition, except $GdMn_{1-x}Ge_2$. For $GdMn_{1-x}Ge_2$ germanide with $CeNiSi_2$ structure type a small homogeneity range was established, which is limited by the $GdMn_{0.30}Ge_2$ and $GdMn_{0.36}Ge_2$ compositions.

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Ізотермічний переріз діаграми стану потрійної системи Gd-Mn-Ge при 1070 K

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Взаємодію компонентів у потрійній системі Gd–Mn–Ge досліджено за температури 1070 K у повному інтервалі концентрацій методами скануючої електронної мікроскопії, енергодисперсійної рентгенівської спектроскопії і рентгенівського аналізу. За температури 1070 K система Gd–Mn–Ge характеризується існуванням шести тернарних сполук: $GdMn_4Ge_2$ (структурний тип $ZrFe_4Si_2$, просторова група $P4_2/mnm$, a = 0.7645(4), c = 0.3953(3) нм), GdMnGe (структурний тип TiNiSi, просторова група $Pnma$, a = 0.7129(2), b = 0.4166(2), c = 0.8201(2) нм), $Gd_3Mn_4Ge_4$ (структурний тип $Gd_3Cu_4Ge_4$, просторова група $Immm$, a = 1.4023(4), b = 0.7121(3), c = 0.4212(3) нм), $GdMn_2Ge_2$ (структурний тип $CeAl_2Ga_2$, просторова група $I4/mmm$, a = 0.40326(6), c = 1.0884(2) нм), $GdMn_6Ge_6$ (структурний тип $MgFe_6Ge_6$, просторова група $P6/mmm$, a = 0.52377(5), c = 0.8182(1) нм) і $GdMn_{1-x}Ge_2$ (структурний тип $CeNiSi_2$, просторова група $Cmcm$, a = 0.41612(8)-0.41590(7), b = 1.6123(3)-1.6119(1), c = 0.40336(8)-0.40290(6) нм). Визначено область гомогенності сполуки $GdMn_{1-x}Ge_2$ (x = 0.70-0.64).

Ключові слова: інтерметаліди, потрійна система, фазові рівноваги, кристалічна структура.