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L. Zinko, G. Nychyporuk, O. Matselko, R. Gladyshevskii The ternary system Hf–Re–Al at 1000°C

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The interaction of the components in the Hf-Re-Al system was investigated by X-ray powder diffraction and scanning electron microscopy with energy-dispersive X-ray spectroscopy. The isothermal section of the phase diagram at 1000 °C was constructed in the full concentration range. A new ternary compound ~HfsRe₂Al₂, isostructural with Ti₅Ga₄ (*hP*18, *P*6₃/*mcm*), and two extended solid solutions Hf(Re,Al)₂ were found.

Keywords: hafnium, rhenium, aluminum, X-ray powder diffraction, energy-dispersive X-ray spectroscopy, phase diagram, isothermal section, crystal structure.

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Introduction

Ternary systems involving *d*-elements of groups IV and VII and *p*-elements of group III of the periodic table have been studied for more than 50 years. Isothermal sections of the phase diagrams have been constructed for the Ti-Mn-B [1], Zr-Mn-B [2], Ti-Re-B [3], Hf-Re-B [4], Ti-Mn-Al [5-8], Ti-Mn-Ga [9], Zr-Mn-Ga [9, 10], Hf-Mn-Ga [10, 11], and Zr-Mn-In [12, 13] systems. $T^{IV}-T^{VII}-M^{III}$ systems are characterized by no or few ternary compounds [14-17]. Some of the systems have been investigated for the formation of compounds of particular compositions, isostructural to known types. Information about the compounds found in the systems {Ti, Zr, Hf}-{Re, Mn}-{B, Al, Ga, In} is summarized in Table 1. Other $T^{IV} - T^{VII} - M^{III}$ systems, probably due to the

Table 1.

Ternary compounds in $T^{IV} - T^{VII} - M^{III}$ systems [17]						
System	Phase	Structure type	Pearson symbol	Space group		
Ti–Re–B	Ti–Re–B Ti ₂ ReB ₂		<i>tP</i> 10	P4/mbm		
Zr–Re–B	Zr ₉ Re ₄ B	Hf9M04B	hP28	P6 ₃ /mmc		
Hf–Re–B	Hf9Re4B	Hf9M04B	hP28	P6 ₃ /mmc		
Ti-Mn-Al	Ti _{7.25} Mn _{7.25} Al _{14.5}	Mg ₆ Cu ₁₆ Si ₇	<i>cF</i> 116	Fm-3m		
Zr-Mn-Al	ZrMn ₆ Al ₆	ThMn ₁₂	<i>tI</i> 26	I4/mmm		
Ti–Mn–Ga	TiMn _{0.5} Ga _{2.5}	Cu ₃ Au	cP4	Pm-3m		
	Ti _{2.75} Mn _{3.25} Ga ₅	Ti ₆ Sn ₅	hP22	P6 ₃ /mmc		
	Zr ₆ Mn _{10.5} Ga _{12.5}	Th ₆ Mn ₂₃	<i>cF</i> 116	Fm-3m		
7. M. C.	ZrMn _{0.5} Ga _{2.5}	Cu ₃ Au	cP4	Pm-3m		
Zr–Mn–Ga	Zr ₃ Mn ₂ Ga ₆	Hf ₃ Mn ₂ Ga ₆	oP22	Pmmn		
	Zr ₃ Mn ₂ Ga ₄	Zr ₃ V ₂ Ga ₄	oP72	Pnma		
Hf–Mn–Ga	HfMnGa ₂	HfFeGa ₂	oP48	Pnma		
	Hf ₃ Mn ₂ Ga ₆	Hf ₃ Mn ₂ Ga ₆	oP22	Pmmn		
	Hf ₃ Mn ₂ Ga ₄	Zr ₃ V ₂ Ga ₄	oP72	Pnma		
Zr–Mn–In	ZrMn _{0.7} In _{2.3}	Cu ₃ Au	cP4	Pm-3m		

difference between the melting temperatures of the components and the complexity of the synthesis, remain little studied.

The aim of this work was to investigate the interaction of the components in the Hf–Re–Al system at 1000 °C.

It should be noted that the binary systems Hf–Re, Hf– Al, and Re–Al, which border the ternary system under investigation, have been studied quite thoroughly. The phase diagrams have been constructed [18] and the crystal structures of several compounds have been determined [17].

I. Experimental

The interaction of the components in the Hf-Re-Al system was investigated on 2 binary and 35 ternary samples. The alloys were synthesized from high-purity metals (Hf \ge 99.9 mass %, Re \ge 99.9 mass % (pressed pellets), Al \geq 99.999 mass %) by arc melting with a tungsten electrode, a water-cooled copper hearth and a Ti getter, under an argon atmosphere. After the synthesis, the ingots were sealed in quartz ampoules under vacuum, annealed at 1000 °C for 1 week and guenched into cold water. Phase analysis and structure refinements were carried out using X-ray powder diffraction (XRPD) data collected on diffractometers DRON-2.0M (Fe Ka radiation) and STOE Stadi P (Cu $K\alpha_1$ radiation). The phase analysis was performed using Powder Cell [19] and STOE WinXPOW [20] programs. The profile and structural parameters were refined by the Rietveld method, using the WinCSD [21] program packages. The overall compositions of the samples and of the individual phases, in particular for the determination of the solubilities of the third component in the binary phases, were investigated by means of energy-dispersive X-ray spectroscopy (EDXS; scanning electron microscope Tescan Vega 3 LMU equipped with an X-MaxN²⁰ silicon drift detector).

II. Results and discussion

As a result of the phase analysis by X-ray diffraction and EDXS of the samples of the Hf–Re–Al system at 1000°C, we confirmed the existence of 13 of the 20 known binary compounds, the compositions and crystallographic parameters of which are given in Table 2. The results are in good agreement with literature data on the interaction of the components and phase diagrams of the binary systems Hf–Re, Hf–Al, and Re–Al [18, 22-25].

According to the results of the local X-ray spectral analysis, the binary compounds $HfAl_3$, Hf_2Al_3 , HfAl, Hf_4Al_3 , Hf_3Al_2 , Hf_2Al , and $Re_{14}Al_{54.75}$ do not dissolve significant amounts of the third component. The maximum solubility of Hf in the compounds Re_4Al_{11} and Re_2Al is ~5 at.%. Approximately the same amount of Al is soluble in the compound $Hf_{21}Re_{25}$.

The formation of two extended solid solutions based on the compounds HfRe₂ (a certain region of homogeneity exists) and HfAl₂ is expected, given the isostructurality of the parent compounds (see Table 2) and the similar sizes of the rhenium and aluminum atoms ($r_{\rm Hf} = 0.1564$, $r_{\rm Re} = 0.1370$, $r_{\rm Al} = 0.1431$ nm [26]). A continuous series

Table 2.

(binary phases stable at 1000 °C are highlighted)						
Compound	Structure	Pearson	Space	Cell parameters, nm		
Compound	type	symbol	group	a	b	С
$Hf_{21}Re_{25}$	$Zr_{21}Re_{25}$	hR276	R-3c	2.5773	—	0.8760
HfRe ₂	MgZn ₂	hP12	P6 ₃ /mmc	0.5239	—	0.8584
Hf ₅ Re ₂₄	Ti ₅ Re ₂₄	<i>cI</i> 58	I-43m	0.9708	—	_
Re ₂ Al	CuZr ₂	tI6	I4/mmm	0.29802	—	0.95796
ReAl	CsCl	cP2	Pm-3m	0.288		
$Re_{1.8}Al_{22}$	$Re(Re_{0.4}Al_{0.6})_2Al$	tP4	P4/mmm	0.30785		0.59515
Re_4Al_{11}		aP15	<i>P</i> -1	0.5172	0.5154	0.8959
Ke4Al11	Mn_4Al_{11}		Γ-1	$\alpha = 74.83^\circ; \beta = 90.43^\circ; \gamma = 80.24^\circ$		
D . 41	Do 41	<i>aP</i> 71	<i>P</i> -1	0.5159	0.9106	2.3755
Re ₁₄ Al _{54.75}	Re ₁₄ Al _{54.75}		Γ-1	$\alpha = 100.94^{\circ}; \beta = 90.30^{\circ}; \gamma = 93.00^{\circ}$		
D. 41	Re ₈ Al _{32.07}	aP43	<i>P</i> -1	0.5153	0.90782	1.3727
Re ₈ Al _{32.07}				$\alpha = 96.852^\circ; \beta = 95.521^\circ; \gamma = 92.392^\circ$		
ReAl ₆	MnAl ₆	oS28	Cmcm	0.7599	0.6606	0.9029
ReAl ₁₂	WAl ₁₂	<i>cI</i> 26	Im-3	0.7527	—	_
HfAl ₃ rt	ZrAl ₃	<i>tI</i> 16	I4/mmm	0.3989	_	1.7155
HfAl ₃ ht	TiAl ₃	<i>tI</i> 8	I4/mmm	0.3893	—	0.8925
$HfAl_2$	MgZn ₂	hP12	$P6_3/mmc$	0.525	—	0.868
Hf_2Al_3	Zr_2Al_3	oF40	Fdd2	0.9529	1.3763	0.5522
HfAl	TlI	<i>oS</i> 8	Cmcm	0.3253	1.0822	0.4280
Hf ₄ Al ₃	Zr ₄ Al ₃	hP7	P6/mmm	0.5334	—	0.5429
Hf ₃ Al ₂	Zr ₃ Al ₂	<i>tP</i> 20	$P4_2/mnm$	0.7535	—	0.6906
Hf ₅ Al ₃	Mn ₅ Si ₃	hP16	P6 ₃ /mcm	0.8052	-	0.5690
Hf ₂ Al	CuAl ₂	<i>tI</i> 12	I4/mcm	0.6776	-	0.5372

Crystallographic parameters of binary compounds in the Hf–Re, Re–Al, Hf–Al systems [17] (binary phases stable at 1000 °C are highlighted)

Compositions of the samples shown in Fig. 1

of solid solutions is likely to form at higher temperatures.

At the temperature of the study, in the region of high hafnium content in samples with the element ratio Hf:Re:Al = 9:4:1 according to the EDXS analysis, the existence of a new compound of approximate composition

~Hf₅Re₂Al₂, isostructural with the compound Ti_5Ga_4 (*hP*18, *P*6₃/*mcm*) [27], was discovered. A detailed description of this compound will be the subject of a separate publication.

Table 3 shows the compositions, and Fig. 1 presents

Table 3.

	Composition Composition			Composition		
Sample	nominal	from EDXS	Sample	nominal	from EDXS	
1	$Hf_{10}Re_{80}Al_{10}$	Hf _{11.0(5)} Re _{86.2(5)} Al _{2.8(7)}	6	Hf ₂₅ Re ₃₀ Al ₄₅	Hf _{28.0(4)} Re _{33.7(4)} Al _{38.3(9)}	
2	$Hf_{15}Re_{40}Al_{45}$	$Hf_{13.4(3)}Re_{40.5(2)}Al_{46.1(8)}$	7	$Hf_{40}Re_{15}Al_{45}$	Hf _{42.1(5)} Re _{10.6(5)} Al _{47.4(8)}	
3	$Hf_{20}Re_{40}Al_{40}$	$Hf_{22.1(6)}Re_{49.5(6)}Al_{28.4(9)}$	8	$Hf_{50}Re_{30}Al_{20}$	Hf _{53.3(8)} Re _{28.8(8)} Al _{17.9(9)}	
4	$Hf_{25}Re_{10}Al_{65}$	Hf _{22.2(2)} Re _{8.4(2)} Al _{69.4(8)}	9	$Hf_{75}Re_{10}Al_{15}$	$Hf_{73.4(7)}Re_{11.3(7)}Al_{15.3(6)}$	
5	$Hf_{20}Re_{65}Al_{15}$	$Hf_{25.9(7)}Re_{67.4(6)}Al_{6.7(9)}$	9			

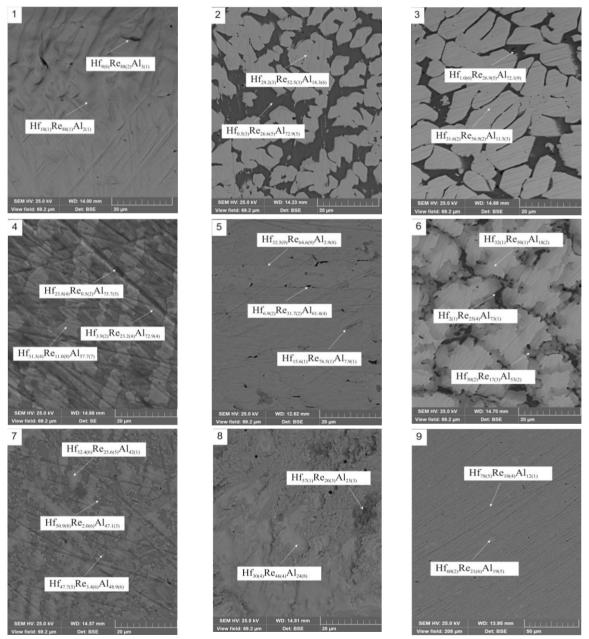


Fig. 1. Microstructures of selected samples and compositions of the detected phases.

Table 4.

ole		Pearson symbol, space	Cell parameters, nm					
Sample	Phase	group	а	b	с			
1	Hf5Re24 Hf10(1)Re88(1)Al2(1)	cI58, I-43m	0.96621(3)	-	_			
	Hf ₉₍₆₎ Re ₈₈₍₂₎ Al ₃₍₁₎	not detected by XRPD						
2	Hf(Re,Al) ₂ Hf _{29.2(3)} Re _{52.5(3)} Al _{18.3(6)}	hP12, P6 ₃ /mmc	0.52294(3)	_	0.85248(9)			
	Hf5Re24	cI58, I-43m	0.96694(9)	—	—			
	Re ₄ Al ₁₁	aP15, P-1	0.5213(2)	0.5132(2)	0.9010(3)			
	Hf0.5(3)Re26.6(5)Al72.9(5)		$\alpha = 74.98(2)^{\circ}; \beta = 91.28(3)^{\circ}; \gamma = 80.32(3)^{\circ}$					
3	$\frac{Hf(Re,Al)_2}{Hf_{31.6(2)}Re_{56.9(2)}Al_{11.5(3)}}$	hP12, P6 ₃ /mmc	0.52327(2)	_	0.85345(4)			
	Re4Al 11 Hf1.0(6)Re26.9(5)Al72.1(9)	not detected by XRPD						
	Hf(Al,Re) ₂ Hf _{31.3(4)} Re _{11.0(8)} Al _{57.7(7)}	hP12, P6₃/mmc	0.52573(3)	-	0.85710(7)			
4	HfAl3 rt Hf23.8(4)Re0.5(2)Al75.7(5)	tI16, I4/mmm	0.39907(3)	_	1.7164(2)			
	Re4Al11 Hf _{3.9(2)} Re _{23.2(4)} Al _{72.9(4)}	<i>aP</i> 15, <i>P</i> -1, >5 % by XRPD						
5	Hf(Re,Al) 2 Hf _{32.5(9)} Re _{64.6(9)} Al _{2.9(8)}	hP12, P6 ₃ /mmc	0.52450(3)	_	0.85774(9)			
	Hf ₅ Re ₂₄ Hf _{15.6(1)} Re _{76.5(1)} Al _{7.9(1)}	cI58, I-43m	0.97154(8)	-	-			
	Hf _{6.9(2)} Re _{31.7(2)} Al _{61.4(4)}	not detected by XRPD						
6	Hf(Re,Al) ₂ Hf ₃₂₍₁₎ Re ₅₀₍₁₎ Al ₁₈₍₂₎	hP12, P6 ₃ /mmc	0.52485(3)	-	0.85404(8)			
	Hf(Al,Re) ₂ Hf ₃₀₍₂₎ Re ₁₇₍₃₎ Al ₅₃₍₂₎	not detected by XRPD						
	Re4Al ₁₁ Hf ₂₍₁₎ Re ₂₅₍₄₎ Al ₇₃₍₁₎	not detected by XRPD						
7	Hf(Re,Al) ₂ Hf _{32.4(6)} Re _{25.6(5)} Al ₄₂₍₁₎	hP12, P63/mmc	0.52592(4)	-	0.8554(1)			
	Hf4Al3 Hf47.7(5)Re3.4(6)Al48.9(6)	hP7, P6/mmm	0.53282(8)	-	0.5435(2)			
	HfAl Hf50.9(8)Re2.0(6)Al47.1(3)	oS8, Cmcm	0.3318(6)	1.116(2)	0.4185(7)			
8	Hf(Re,Al) ₂ Hf ₃₀₍₄₎ Re ₄₆₍₄₎ Al ₂₄₍₈₎	hP12, P6 ₃ /mmc	0.52747(7)	_	0.8603(2)			
	~Hf5Re2Al2 Hf57(1)Re20(3)Al23(3)	hP18, P6 ₃ /mcm	0.8091(2)	_	0.5689(2)			
	Hf	hP2, P63/mmc	0.3170(1)	-	0.5038(3)			
9	$\begin{array}{c} \textbf{Hf} \\ Hf_{78(5)}Re_{10(4)}Al_{12(1)} \end{array}$	hP2, P6 ₃ /mmc	0.31599(3)	-	0.50208(9)			
У	~Hf5Re2Al2 Hf60(2)Re21(6)Al19(5)	hP18, P6 ₃ /mcm	0.8076(1)	_	0.5730(2)			

Results of the phase analysis of the samples shown in Fig. 1, compositions from EDXS, cell parameters from XRPD

photographs of the surfaces of individual samples. Table 4 summarizes the results of the phase analyses. Fig. 2 shows the isothermal section of the Hf–Re–Al phase diagram at 1000 °C. The ternary phase ~Hf₅Re₂Al₂ forms equilibria with Hf₃Al₄, Hf₂Al, (Hf), Hf₂₁Re₂₅ and the extended solid solution of HfRe₂.

If we compare the Hf–Re–Al system with previously studied $T^{IV}-T^{VII}-M^{III}$ systems, we can see that they differ both in the nature of the phase fields and in the number and types of ternary compounds that exist in the systems [1-17]. In most of the systems involving boron, compounds are either absent or a single compound is formed. The systems with aluminum and indium are characterized by the presence of one or two compounds.

Slightly more compounds (2-4) are formed in the systems with gallium. The Ti–Mn–Al system has been studied at various temperatures.

Similarly to the system studied by us, hexagonal phases isostructural to the MgZn₂ type with regions of homogeneity of different size are also known in the systems {Ti, Zr, Hf}–Mn–Al and {Zr, Hf}–Mn–Ga, whereas in the system Hf–Re–B the binary phase HfRe₂ does not dissolve significant amounts of the third component. In contrast to the Hf–Re–Al system, no ternary compound with a structure of the Ti₅Ga₄ or Hf₅CuSn₃ types [28] has been reported in the systems considered here.

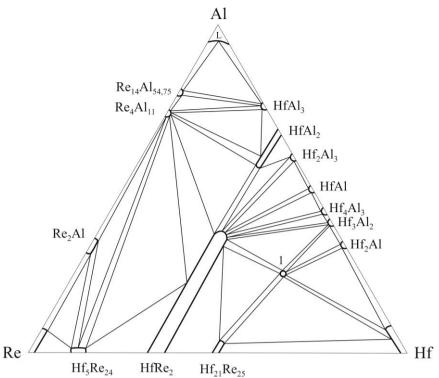


Fig. 2. Isothermal section of the Hf–Re–Al phase diagram at 1000 °C (1 – ~Hf₅Re₂Al₂).

Conclusions

Ternary systems involving *d*-elements of groups IV and VII and *p*-elements of group III of the periodic system are characterized by either the absence, or a low number of ternary compounds. The isothermal section of the phase diagram of the Hf-Re-Al system at 1000 °C was investigated in the full concentration range using X-ray powder diffraction, scanning electron microscopy, and energy-dispersive X-ray spectroscopy. Under the conditions of the study, the existence of 13 previously reported binary compounds confirmed. was Approximately 5 at.% of Hf can be dissolved in the compounds Re₄Al₁₁ and Re₂Al, and about the same amount of Al in Hf₂₁Re₂₅. A new ternary compound of the approximate composition ~Hf₅Re₂Al₂, adopting a Ti₅Ga₄-type structure (hP18, $P6_3/mcm$), and two extended solid solutions based on the hexagonal Laves-type (MgZn₂) binary compounds HfRe₂ and HfAl₂ were found.

The Hf–Re–Al system differs from previously studied $T^{IV}-T^{VII}-M^{III}$ systems, both in the nature of the phase fields and in the number and structures of the ternary compounds. Hexagonal Laves-type phases also form in the {Ti, Zr, Hf}–Mn–Al and {Zr, Hf}–Mn–Ga systems, however, no ternary compound with a Ti₅Ga₄-(or Hf₅CuSn₃-) type structure was found in these systems.

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Потрійна система Hf-Re-Al при 1000°C

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Взаємодію компонентів у системі Hf-Re-Al досліджено методами рентгенівської порошкової дифракції, скануючої електронної мікроскопії та енергодисперсійної рентгенівської спектроскопії. Побудовано ізотермічний переріз діаграми стану при 1000 °C в повному концентраційному інтервалі. Визначено існування нової тернарної сполуки ~Hf₅Re₂Al₂, ізоструктурної до типу Ti₅Ga₄ (*hP*18, *P*6₃/*mcm*), і двох протяжних твердих розчинів складу Hf(Re,Al)₂.

Ключові слова: гафній, реній, алюміній, рентгенівська порошкова дифракція, енергодисперсійна рентгенівська спектроскопія, діаграма стану, ізотермічний переріз, кристалічна структура.