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Phase equilibria in Co−Al−Re ternary system at 1100 and 1300 °C

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Abstract: Isothermal sections of the Co−Al−Re ternary system at 1100 and 1300 °C were determined experimentally by electron probe microanalysis and X-ray diffraction. The results show that there are seven three-phase regions in the 1100 °C isothermal section and five three-phase regions in the 1300 °C isothermal section. The solid solubilities of αCo, εRe and CoAl increase a little with temperature increasing from 1100 to 1300 °C. The solubility of Co in compounds $AlRe_2$, $Al_{11}Re_4$ and Al_4Re is negligible, <1.5 at.%. And no ternary compounds are found.

Key words: Co−Al−Re; phase equilibria; isothermal section

1 Introduction

The phase equilibria of the Co−Al−Re ternary system are fundamentally important to understand and develop Co- and Ni-based superalloys.

Recently, $L1_2$-ordered phases have been observed in some Co−Al−X (X=W, V, etc) systems and have exhibited good performances [1−3]. Both Al and Co are important constituent elements of Ni-based superalloys. Thus, the Co−Al system plays an important role in Co- and Ni-based superalloys. Rhenium (Re) is an element commonly used in fourth- and fifth-generation Ni-based superalloys [4−8]. Elemental Re not only improves the strength and creep resistance of Co-based superalloys but also refines its morphology and decreases γ/γ' lattice parameter misfits [9,10]. Therefore, in order to develop Ni- and Co-based superalloys and understand the relationships between their microstructures and properties, knowledge of the phase equilibria occurring in the Co−Al−Re system is required. However, experimental information and phase diagrams of this ternary system have not been established.

In this study, experimental investigation of the phase equilibria occurring in the Co−Al−Re ternary system at 1100 and 1300 °C was conducted. The experimental data will help to establish thermodynamic databases and inform the design and development of Co- and Ni-based superalloys.

Phase equilibria in three constituent sub-binary systems of Co−Al [11−14], Co−Re [15−18], and Al−Re [19−24], have been comprehensively investigated and critically assessed in the literatures. The crystal information existing for the stable phases in the Co−Al−Re ternary system is listed in Table 1.

The Co−Al system was first mentioned in Ref. [11], and then STEIN et al [12] investigated the melting behaviour and homogeneity range of the *B*2 phase. Later, PRIPUTEN et al [13] studied the Co_4Al_{13} family of phases and reported that it has six phases: $CoAl_3$, $Y1$-Co_4Al_{13}, $Y2$-Co_4Al_{13},

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Table 1 Crystal structures of stable solid phases in Co−Al−Re ternary systems

System	Phase	Pearson's symbol	Prototype	Space group	Struktur−bericht	Lattice constant/Å	Ref.
Co−Al	Al	$cF4$	Cu	$Fm\overline{3}m$	$A3$	a=4.0496	[11]
	Co_2Al_9	$mP22$	Co_2Al_9	$P2_1/c$		a=6.2163, b=6.2883, c=8.5587, β=94.772°	[13]
	Co_4Al_{13}*	$oP102$	Co_4Al_{13}	$Pmn2_1$		a=8.158, b=12.342, c=14.452	[13]
	Co_2Al_5	$hP28$	Co_2Al_5	$P6_3/mmc$		a=7.671, c=7.608	[13]
	CoAl	$cP2$	CsCl	$Pm\overline{3}m$	$B2$	a=2.8611	[13]
	εCo	$hP2$	Mg	$P6_3/mmc$	$A3$	a=2.5071, c=4.0695	[11]
	αCo	$cF4$	Cu	$Fm\overline{3}m$	$A1$	a=3.5446	[11]
Co−Re	ε(Co,Re)	$hP2$	Mg	$P6_3/mmc$		a=2.64, c=4.28	[18]
	αCo	$cF4$	Cu	$Fm\overline{3}m$	$A1$	a=3.57	[18]
Al−Re	εRe	$hP2$	Mg	$P6_3/mmc$		a=2.760, c=4.458	[23]
	Al	$cF4$	Cu	$Fm\overline{3}m$		a=4.0496	[23]
	$Al_{12}Re$	$cI26$	$Al_{12}W$	$Im3$		a=7.5261(5)	[24]
	Al_6Re	$oC28$	Al_6Mn	$Cmcm$		a=7.608, b=6.617, c=9.046	[24]
	Al_4Re	$mC30$	Al_4W	Cm		a=5.132(2), b=17.470(1), c=5.167(2), β=100.43(3)°	[24]
	$Al_{33-x}Re_4$	$aP41$		$P1$		a=5.1535(6), b=9.0782(8), c=13.727(1), α=96.852(7)°, β=95.521(9)°, γ=92.392(9)°	[24]
	$Al_{11}Re_4$	$aP15$	$Al_{11}Mn_4$	$P1$		a=5.199(6), b=8.963(1), c=5.1693(6), α=90.44(1)°, β=99.76(1)°, γ=105.17(1)°	[24]
	AlRe	$tP4$	CuTi	$P4/nmm$		a=3.084(1), c=5.957(1)	[24]
	$AlRe_2$	$tI6$	$MoSi_2$	$I4/mmm$		a=2.981(1), c=9.584(4)	[24]

*: Co_4Al_{13} family of phases. Here, a typical crystal structure of the Co_4Al_{13} family is presented

M-Co_4Al_{13}, O-Co_4Al_{13} and O'-Co_4Al_{13}. They considered that $Y2$-Co_4Al_{13} phase is metastable and that a transition reaction might exist between O-Co_4Al_{13} and O'-Co_4Al_{13}. To simplify the phase diagram, some researchers [14] treat the Co_4Al_{13} family as a single stoichiometric phase named Co_4Al_{13}. This simplification is also adopted in the present study.

The Co−Re binary system was first reported by ELLIOT [15] in 1965. Then, PREDEL [16] investigated the martensitic character of the phase transformation. Experiments determined that the Co−Re system is simple without an intermetallic phase. Recently, LIU et al [17] and GUO et al [18] thermodynamically evaluated the Co−Re system and their findings were consistent with the experimental data. The newly-assessed Co−Re phase diagram of GUO et al [18] is applied in the present study, as shown in Fig. 1.

For Al−Re system, HUANG and CHANG [19] were the first to publish a thermodynamic diagram based on the experimental data of SAVITSKII et al [20], ELLIOTT [15], SHRINK [21] and SCHUSTER [22]. Then, CORNISH and WITCOMB [23], using metallographic methods and X-ray diffraction, confirmed the existence of the intermediate phases of $Al_{12}Re$, Al_6Re, Al_4Re and $Al_{11}Re_4$. SCHUSTER et al [24] investigated the Al−Re binary system and confirmed that there are seven intermetallic phases: $Al_{12}Re$, Al_6Re, $Al_{33-x}Re_8$, Al_4Re, $Al_{11}Re_4$, AlRe and $AlRe_2$. They also described the crystal structure and lattice parameters for all these phases. Here, the results of SCHUSTER et al [24] are adopted.

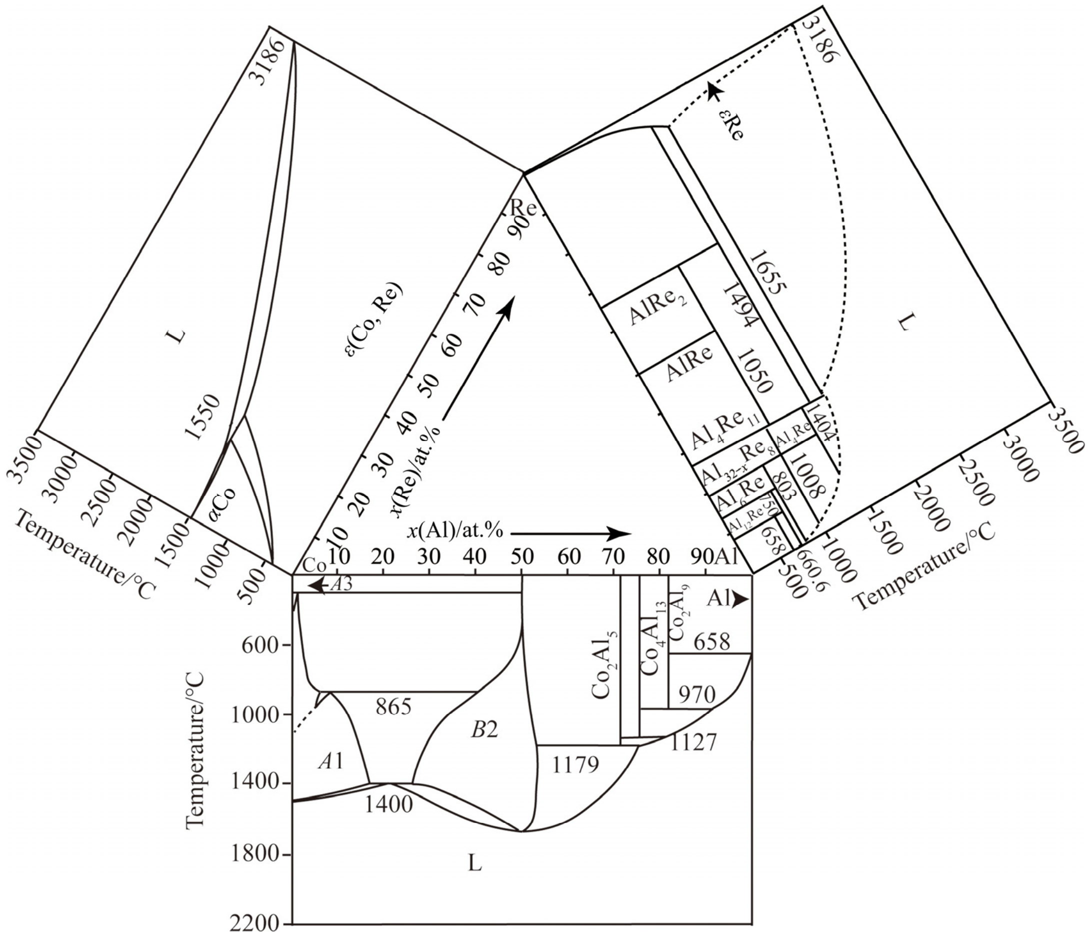

Fig. 1 Binary phase diagrams constituting Co−Al−Re ternary system [14,18,24]

2 Experimental

Usually, phase relationships are studied by the equilibrated alloy [25,26] and diffusion couple methods [27]. Here, the phase relationships of the Co−Al−Re ternary system at 1100 and 1300 °C are studied using equilibrated alloy. Pure metals of cobalt (99.9 wt.%), aluminium (99.9 wt.%) and rhenium (99.9 wt.%) were used as raw materials to prepare ingots by arc melting under a high-purity argon atmosphere. The mass of each sample was about 15 g. To achieve homogeneity, the ingots were remelted at least four times and their mass losses were <0.5 wt.%.

Then, the specimens were cut into plate shapes and put into quartz capsules that were filled with argon gas. In addition, pure Ti filings were added to the quartz capsules to prevent oxidation, and samples containing a liquid phase were wrapped in a pure Ta slice to prevent contact reaction with quartz. Specimens were annealed at 1100 and 1300 °C for various durations ranging from 1 h to 60 d and then quenched in ice water.

Backscatter electron (BSE) imaging and the equilibrium composition of each phase were determined by an electron probe micro analyser (EPMA; JXA−8100R, JEOL, Japan) with an accelerating voltage of 20.0 kV and a probe current of 1.0×10^{-8} A. X-ray diffraction (XRD) was performed for phase identification using Cu K_α radiation at 40 kV and 40 mA. To ensure the accuracy of the data, pure elements were used as standards to calibrate the measurements. The final values of the phase compositions are the averages of seven measurements.

3 Results and discussion

About 14 samples were used to determine the phase boundaries for isothermal sections. Figures 2 and 3 show typical BSE images of Co−Al−Re ternary alloys annealed at 1100 and 1300 °C, respectively. The corresponding XRD patterns are presented in Fig. 4. Tables 2 and 3 present the equilibrium compositions of the Co−Al−Re alloys quenched from 1100 and 1300 °C, respectively, as determined by EPMA.

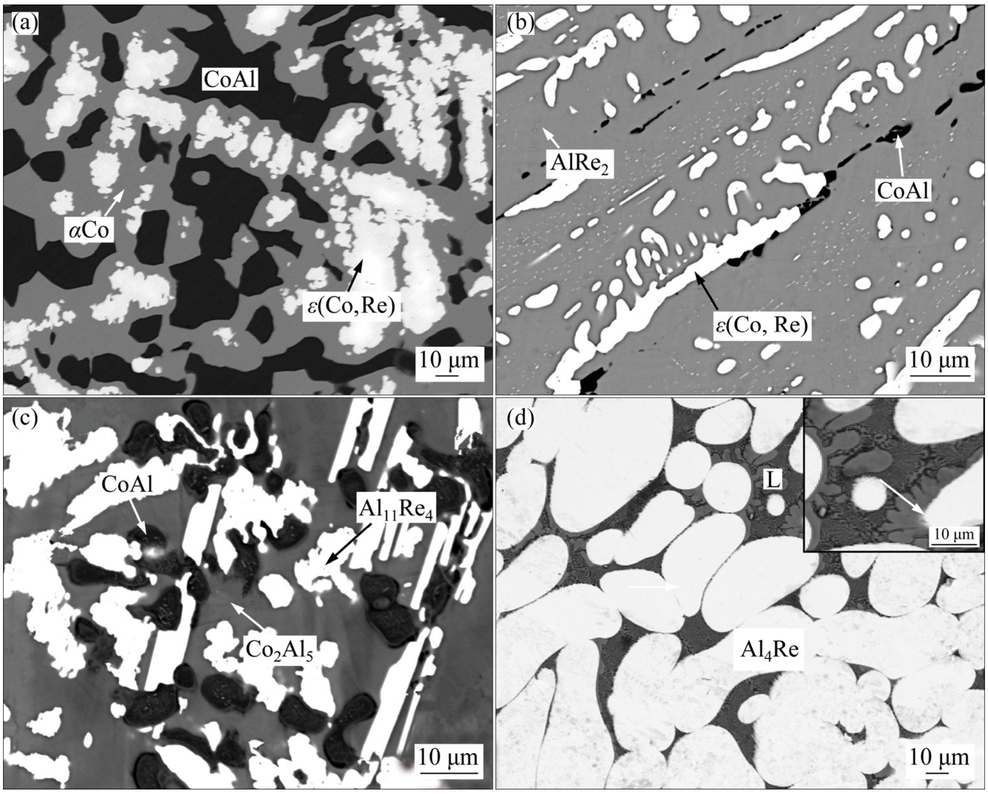

Fig. 2 Typical BSE images obtained from Co−Al−Re ternary alloys annealed at 1100 °C: (a) $Co_{61}Al_{19}Re_{20}$ (at.%); (b) $Co_{30}Al_{40}Re_{30}$ (at.%); (c) $Co_{22}Al_{70}Re_8$ (at.%); (d) $Co_1Al_{82}Re_{17}$ (at.%)

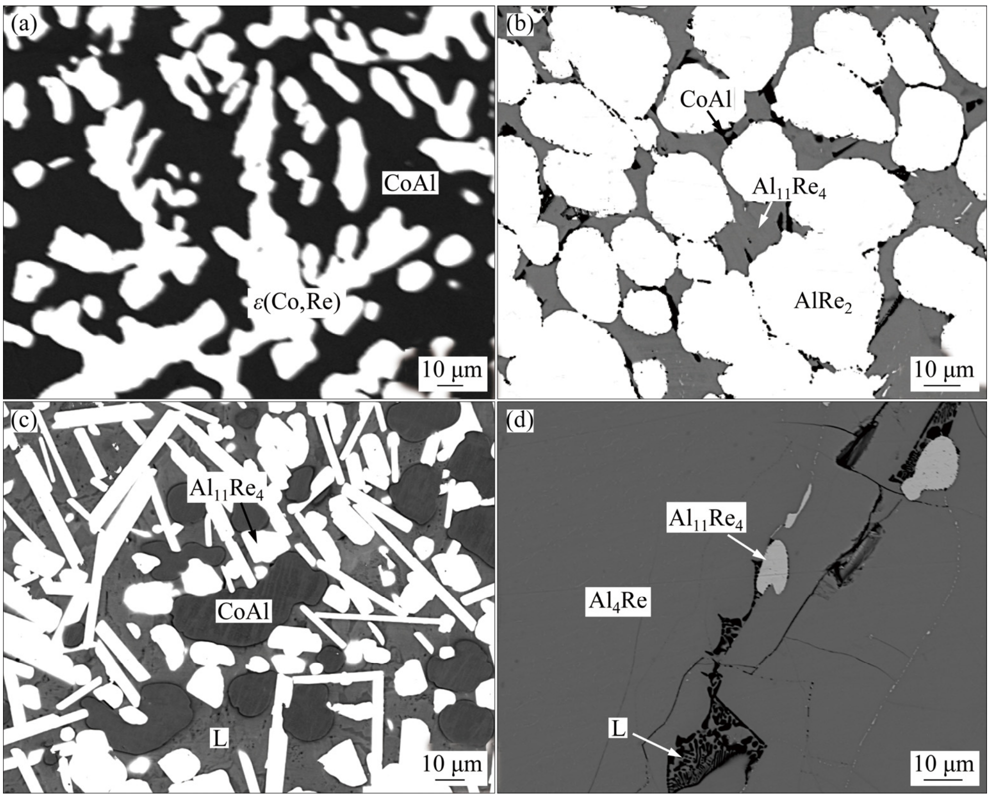

Fig. 3 Typical BSE images obtained from Co−Al−Re ternary alloys annealed at 1300 °C: (a) $Co_{38}Al_{32}Re_{30}$ (at.%); (b) $Co_{30}Al_{50}Re_{20}$ (at.%); (c) $Co_{22}Al_{70}Re_8$ (at.%); (d) $Co_2Al_{76}Re_{22}$ (at.%)

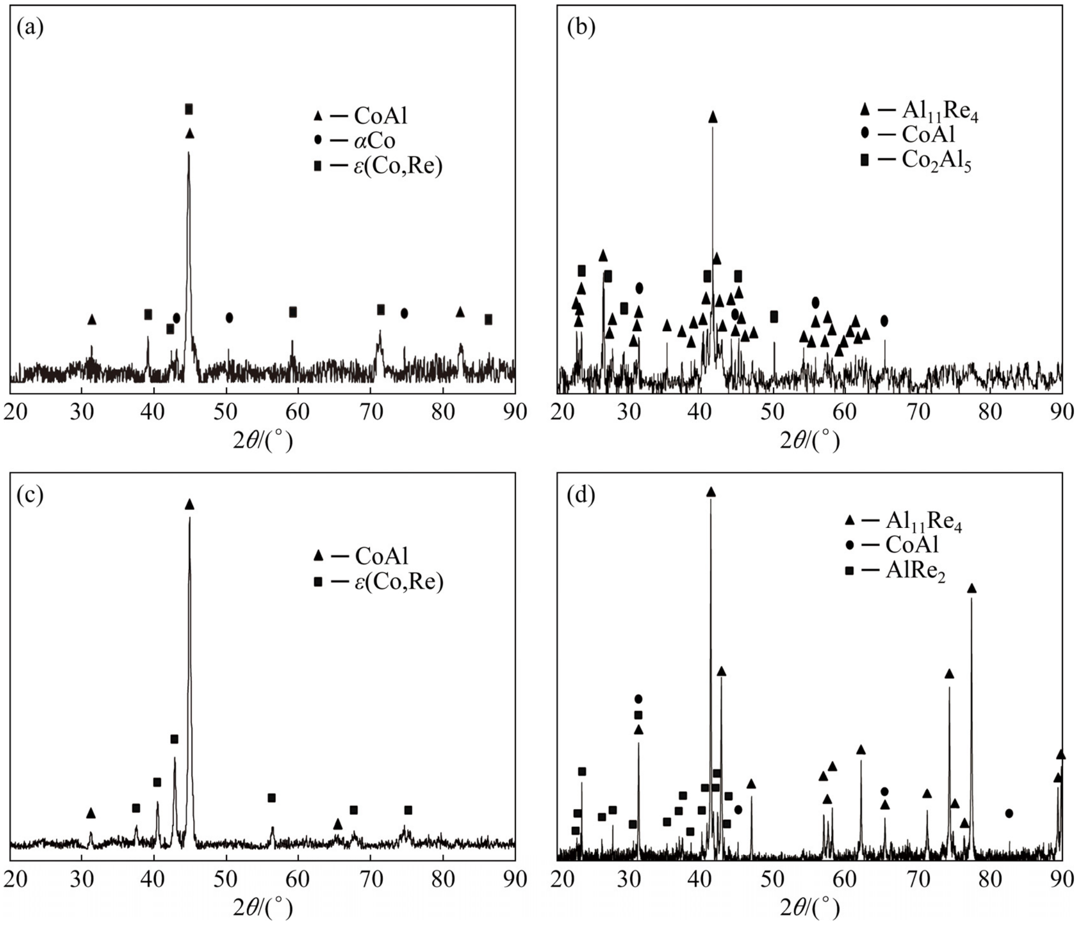

Fig. 4 X-ray diffraction patterns obtained from alloys: (a) $Co_{61}Al_{19}Re_{20}$ annealed at 1100 °C for 60 d; (b) $Co_{22}Al_{70}Re_8$ annealed at 1100 °C for 30 d; (c) $Co_{38}Al_{32}Re_{30}$ annealed at 1300 °C for 20 d; (d) $Co_{30}Al_{50}Re_{20}$ annealed at 1300 °C for 5 d

Table 2 Equilibrium compositions of Co−Al−Re ternary alloys determined at 1100 °C

Alloys	Annealing time/d	Equilibrium phase			Composition (at. %)					
		1	2	3	Phase 1		Phase 2		Phase 3	
					Re	Al	Re	Al	Re	Al
$Co_{61}Al_{19}Re_{20}$	60	αCo	CoAl	ε(Co,Re)	11.8	11.8	2.2	35.5	51.6	2.1
$Co_{77}Al_{20}Re_3$	60	αCo	CoAl	−	4.9	12.2	1.2	28.9	−	−
$Co_{74}Al_{20}Re_6$	60	αCo	CoAl	−	8.5	12.1	1.6	32.1	−	−
$Co_{78}Al_5Re_{17}$	60	αCo	ε(Co,Re)	−	12.2	6.1	19.8	4.1	−	−
$Co_{75}Al_8Re_{17}$	60	αCo	ε(Co,Re)	−	10.9	6.5	38.1	2.1	−	−
$Co_{55}Al_{25}Re_{20}$	60	CoAl	ε(Co,Re)		2.3	37.4	53.6	2.5	−	−
$Co_{45}Al_{25}Re_{30}$	60	CoAl	ε(Co,Re)	−	2.5	41.3	67.9	2.8	−	−
$Co_{37}Al_{33}Re_{30}$	60	CoAl	ε(Co,Re)	−	2.2	47.9	83.2	6.1	−	−
$Co_{30}Al_{40}Re_{30}$	60	CoAl	ε(Co,Re)	$AlRe_2$	2.1	49.2	87.5	7.3	66.7	32.9
$Co_{30}Al_{50}Re_{20}$	30	CoAl	$AlRe_2$	Al_4Re_{11}	1.6	52.1	65.5	34.1	26.3	73.1
$Co_{22}Al_{70}Re_8$	30	CoAl	Co_2Al_5	Al_4Re_{11}	0.7	53.5	3.2	72.5	25.8	73.7
$Co_2Al_{76}Re_{22}$	1	Al_4Re_{11}	Al_4Re	L	25.2	74.6	19.8	79.8	7.4	79.3
$Co_{34}Al_{64}Re_2$	1	CoAl	Co_2Al_5	−	0.3	54.3	2.2	72.2	−	−
$Co_1Al_{82}Re_{17}$	1	Al_4Re	L	−	18.9	80.5	9.2	89.2	−	−

Table 3 Equilibrium compositions of Co−Al−Re ternary alloys at 1300 °C

Alloy	Annealing time	Equilibrium phase			Composition/at.%					
					Phase 1		Phase 2		Phase 3	
		1	2	3	Re	Al	Re	Al	Re	Al
$Co_{61}Al_{19}Re_{20}$	20 d	αCo	CoAl	ε(Co,Re)	14.3	13.6	3.5	31.3	55.5	2.8
$Co_{77}Al_{20}Re_3$	20 d	αCo	CoAl	−	5.5	14.8	2.0	25.5	−	−
$Co_{73}Al_{20}Re_7$	20 d	αCo	CoAl	−	9.5	14.5	3.1	28.5	−	−
$Co_{74}Al_6Re_{20}$	20 d	αCo	ε(Co,Re)	−	15.2	6.5	27.5	4.2	−	−
$Co_{71}Al_9Re_{20}$	20 d	αCo	ε(Co,Re)	−	15.0	10.1	50.1	2.5	−	−
$Co_{56}Al_{24}Re_{20}$	20 d	CoAl	ε(Co,Re)	−	3.5	32.6	58.1	2.9	−	−
$Co_{46}Al_{24}Re_{30}$	20 d	CoAl/	ε(Co,Re)	−	3.8	37.8	69.5	3.9	−	−
$Co_{38}Al_{32}Re_{30}$	20 d	CoAl	ε(Co,Re)	−	3.0	44.5	84.1	6.1	−	−
$Co_{30}Al_{40}Re_{30}$	20 d	CoAl	ε(Co,Re)	$AlRe_2$	2.6	50.1	87.5	8.1	70.1	29.3
$Co_{30}Al_{50}Re_{20}$	5 d	CoAl	$AlRe_2$	$Al_{11}Re_4$	2.3	52.5	66.6	32.9	26.6	72.5
$Co_{22}Al_{70}Re_8$	1 h	CoAl	$Al_{11}Re_4$	L	1.5	53.5	25.5	73.3	5.5	74.5
$Co_2Al_{76}Re_{22}$	1 h	$Al_{11}Re_4$	Al_4Re	L	24.8	74.6	20.5	78.1	8.2	77.2
$Co_{34}Al_{64}Re_2$	1 h	CoAl	L	−	0.5	54.7	2.9	73.1	−	−
$Co_1Al_{82}Re_{17}$	1 h	Al_4Re	L	−	19.6	79.8	13.5	84.5	−	−

3.1 Isothermal section at 1100 °C

As shown in Fig. 2(a), a three-phase microstructure of αCo (grey), ε(Co,Re) (white), and CoAl (black) is found in $Co_{61}Al_{19}Re_{20}$ alloy annealed at 1100 °C for 60 d. The corresponding XRD pattern is presented in Fig. 4(a). When the $Co_{30}Al_{40}Re_{30}$ alloy is annealed at 1100 °C for 60 d, there are three different phases, including CoAl (black), $AlRe_2$ (grey), and ε(Co,Re) (white), as shown in Fig. 2(b). Similarly, a three-phase microstructure is found in $Co_{22}Al_{70}Re_8$ alloy (Fig. 2(c)). Figure 4(b) shows the corresponding XRD pattern of the $Co_{22}Al_{70}Re_8$ alloy. Three phases of $Al_{11}Re_4$ (white), Co_2Al_5 (grey), and CoAl (black) are clearly distinguishable in Fig. 4(b). In Fig. 2(d), a two-phase microstructure can be observed in the $Co_1Al_{82}Re_{17}$ alloy annealed at 1100 °C for 1 d, where the liquid phase (L) is dispersed in the matrix of solid-phase Al_4Re (white). From magnified image of the liquid phase inserted in Fig. 2(d), both the grey and dark regions are solidified from the liquid phase. According to the morphology, the grey region may be a dendritic structure of the solid-phase Al (FCC). Thus, the chemical composition of the liquid phase is determined from the average value of the dark region.

Based on the experimental data, an isothermal section of the Co−Al−Re ternary system at 1100 °C is constructed (Fig. 5(a)). There are five experimentally-determined three-phase regions: [ε(Co,Re)+αCo+CoAl], [ε(Co,Re)+CoAl+$AlRe_2$], [CoAl+$AlRe_2$+$Al_{11}Re_4$], [CoAl+$Al_{11}Re_4$+Co_2Al_5] and [$Al_{11}Re_4$+Al_4Re+L]. In the Al-rich corner, due to a Co_4Al_{13}-family phase existing in the temperature range of 1093−1153 °C [13], the phase relationships are very complicated. Here, to simplify the phase relationships, the Co_4Al_{13}-family phase is treated as a single phase and other two three-phase regions of [Co_2Al_5+Co_4Al_{13}+L] and [Co_2Al_5+$Al_{11}Re_4$+L] are deduced.

On the Al−Re side, the solubilities of Co in the compounds $AlRe_2$, $Al_{11}Re_4$ and Al_4Re are negligibly small, all <1.5 at.%, as shown in Fig. 4(a).

3.2 Isothermal section at 1300 °C

As shown in Fig. 3(a), two different phases are found in the $Co_{38}Al_{32}Re_{30}$ alloy annealed at 1300 °C. Combined with the XRD analysis in Fig. 4(c), the white phase is identified as ε(Co,Re), while the black phase is CoAl. When the $Co_{30}Al_{50}Re_{20}$ alloy is annealed at 1300 °C (Fig. 3(b)), three different phases, $Co_{11}Re_4$ (grey), $AlRe_2$ (white), and CoAl (black), are identified in combination with the XRD analysis shown in Fig. 4(d). In Fig. 3(c), two solid

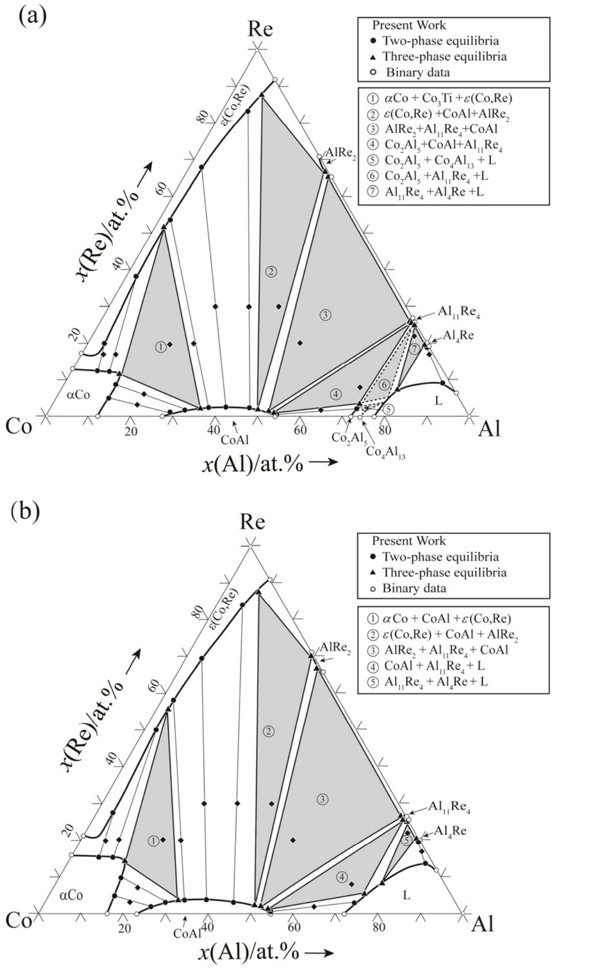

Fig. 5 Experimentally-determined isothermal sections of Co−Al−Re system: (a) 1100 °C; (b) 1300 °C

phases of $Al_{11}Re_4$ (white) and CoAl (dark grey), and a liquid phase (L, light grey) occur in the $Co_{22}Al_{70}Re_8$ alloy annealed at 1300 °C for 1 h. Likewise, a three-phase microstructure consisting of L (black), Al_4Re (dark grey) and $Al_{11}Re_4$ (light grey) can be seen in the $Co_2Al_{76}Re_{22}$ alloy, as shown in Fig. 3(d).

Then, the isothermal section of Co−Al−Re ternary system at 1300 °C is established (Fig. 5(b)). There are five three-phase regions: [ε(Co,Re) + αCo+CoAl], [ε(Co,Re)+CoAl+$AlRe_2$], [CoAl+ $AlRe_2$+$Al_{11}Re_4$], [$Co_{11}Re_4$+CoAl+L] and [$Co_{11}Re_4$+ Al_4Re+L]. Compared with the isothermal section obtained at 1100 °C, there is an obvious difference in the Al-rich corner at 1300 °C: the Co_4Al_{13}-family phases decompose and the liquid phase appears. Other solid solubilities of αCo, ε(Co,Re) and CoAl increase a little from 1100 to 1300 °C. The solubilities of Co in the compounds $AlRe_2$, $Al_{11}Re_4$ and Al_4Re are still very small and negligible.

4 Conclusions

(1) Five three-phase regions are determined and two three-phase regions are deduced at 1100 °C, while five three-phase regions are determined in the 1300 °C isothermal section.

(2) The solid solubilities of αCo, ε(Co,Re) and CoAl increase a little from 1100 to 1300 °C.

(3) The solubility of Co in compounds $AlRe_2$, $Al_{11}Re_4$ and Al_4Re is very low and negligible at both 1100 and 1300 °C.

(4) No ternary compounds are found.

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Co−Al−Re 三元系在 1100 °C 和 1300 °C 等温截面的相平衡

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摘 要：利用电子探针成分分析(EPMA)和 X 射线衍射分析(XRD)建立 Co−Al−Re 三元系的 1100 °C 和 1300 °C 等温截面相图。实验结果表明：在 1100 °C 等温截面相图中存在 7 个三相区，而 1300 °C 等温截面相图中存在 5 个三相区；随着温度从 1100 °C 上升到 1300 °C，*α*Co、*ε*Re 和 CoAl 的固溶度略微增加；Co 在 $AlRe_2$、$Al_{11}Re_4$ 和 Al_4Re 相中的固溶度极小，<1.5%(摩尔分数)；等温截面相图中均未发现三元化合物。

关键词：Co−Al−Re；相平衡；等温截面

(Edited by Bing YANG)