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Subsolidus phase relations in the systems K_2O–ZnO–AO_3 (A = Mo, W)

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Abstract

The subsolidus phase relations of the systems K_2O–ZnO–AO_3 (A = Mo, W) have been investigated by X-ray diffraction (XRD) analyses. The phase diagrams have been constructed. There are six binary compounds and two ternary compounds in the K_2O–ZnO–MoO_3 system, it can be divided into 11 three-phase regions. The K_2O–ZnO–WO_3 system consists of six binary compounds and one ternary compound. This system can be divided into 9 three-phase regions. DTA results indicated the compounds K_2MoO_4 and K_2WO_4 are not suitable to be fluxes for ZnO crystal growth.

Keywords: ZnO; X-ray diffraction; Phase diagram; DTA; Crystal growth

1. Introduction

Single crystal zinc oxide is a wide band gap semiconductor with great potential for a variety of commercial applications including substrates, UV detectors, acoustic wave devices, light emitting diodes, laser diodes and high frequency electronic devices because of its wide band gap of 3.37 eV and large exciton binding energy of 60 meV [1]. With the increasing need for high quality and large size ZnO crystals, several methods have so far applied to the bulk ZnO crystal growth, such as the flux method [2,3], the melt method [4,5], the hydrothermal method [6–8] and the vapor growth technique [9,10]. High-quality ZnO crystals have been grown by the vapor phase and hydrothermal methods. Rather recently, high-quality crystals have been also grown by the melt-growth technique [4,5]. This method can also produce large size diameter single-crystals, which makes them also useful for substrate applications. The ZnO melts congruently at 1975 °C and is rather volatile at higher temperatures, so the Czochralski method is not suitable for growing ZnO single crystal. In order to suppress the ZnO evaporation during crystal growth, the crystal must be grown from a solvent with a growth temperature as low as possible. The high temperature equilibrium between K_2O–MoO_3 [11,12] and K_2O–WO_3 [13–15] have been investigated. According to the reported phase diagram for the system K_2MoO_4–$ZnMoO_4$ [16], there are two ternary compounds, $K_4Zn(MoO_4)_3$ and $K_2Zn_2(MoO_4)_3$, melting incongruently at 615 °C and 610 °C, respectively. The eutectic relationship was found between $K_4Zn(MoO_4)_3$ and $K_2Zn_2(MoO_4)_3$ at 530 °C. There is one ternary compound $K_4Zn(WO_4)_3$ in the K_2WO_4–$ZnWO_4$ phase diagram [16], which melts incongruently at 635 °C. If eutectic exists between ZnO and these compounds, the ZnO single crystal could be grown at very low temperature. So we have studied the phase relations of K_2O–ZnO–AO_3 (A = Mo, W) systems in order to find suitable fluxes and growth regions for growing large and high quality ZnO single crystal at relative low temperature.

2. Experimental

A series of samples with different compositions were all prepared by solid-state chemistry reaction in air. The purity of the starting materials (ZnO, K_2CO_3, MoO_3, WO_3) is higher than 99.9%. The K_2CO_3 was dried at 300 °C for 2 h to remove moisture. The raw powders with certain chemical compositions were mixed thoroughly, ground in an agate mortar, and pressed into pellets with diameter of 10 mm and thickness of 1–2 mm at a pressure around 10^8 Pa. Then the pellets were sintered at the proper temperature in air for about 72–96 h

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Table 1
List of phase identification for samples with different composition in the system K_2O–ZnO–MoO_3

Samples	MoO_3 (mol%)	ZnO (mol%)	K_2O (mol%)	Phase identification
1	33.3	0	66.7	$K_2MoO_4 + K_2CO_3$
2	10	65	25	$ZnO + K_2MoO_4 + K_2CO_3$
3	10	20	70	$ZnO + K_2MoO_4 + K_2CO_3$
4	20	40	40	$ZnO + K_2MoO_4 + K_2CO_3$
5	35	15	50	$ZnO + K_2MoO_4 + K_2CO_3$
6	40	20	40	$ZnO + K_2MoO_4$
7	25	50	25	$ZnO + K_2MoO_4$
8	10	80	10	$ZnO + K_2MoO_4$
9	25	55	20	$ZnO + K_2MoO_4 + K_4Zn(MoO_4)_3$
10	40	25	35	$ZnO + K_2MoO_4 + K_4Zn(MoO_4)_3$
11	30	50	20	$ZnO + K_4Zn(MoO_4)_3$
12	40	40	20	$ZnO + K_4Zn(MoO_4)_3 + K_2Zn_2(MoO_4)_3$
13	20	70	10	$ZnO + K_4Zn(MoO_4)_3 + K_2Zn_2(MoO_4)_3$
14	30	60	10	$ZnO + K_2Zn_2(MoO_4)_3$
15	30	65	5	$ZnO + K_2Zn_2(MoO_4)_3 + Zn_3Mo_2O_9$
16	47.5	50	2.5	$K_2Zn_2(MoO_4)_3 + Zn_3Mo_2O_9 + ZnMoO_4$
17	43.75	50	6.25	$K_2Zn_2(MoO_4)_3 + Zn_3Mo_2O_9$
18	65	30	5	$K_2Mo_4O_{13} + ZnMoO_4 + MoO_3$
19	80	10	10	$K_2Mo_4O_{13} + + ZnMoO_4 + MoO_3$
20	66.7	22.2	11.1	$K_2Mo_4O_{13} + ZnMoO_4$
21	60	30	10	$K_2Mo_3O_{10} + ZnMoO_4$
22	70	15	15	$K_2Mo_4O_{13} + K_2Mo_3O_{10} + ZnMoO_4$
23	55	35	10	$K_2Zn_2(MoO_4)_3 + K_2Mo_3O_{10} + ZnMoO_4$
24	60	25	15	$K_2Zn_2(MoO_4)_3 + K_2Mo_3O_{10} + ZnMoO_4$
25	60	20	20	$K_2Zn_2(MoO_4)_3 + K_2Mo_3O_{10}$
26	65	10	25	$K_2Zn_2(MoO_4)_3 + K_2Mo_2O_7 + K_2Mo_3O_{10}$
27	55	20	25	$K_2Mo_2O_7 + K_2Zn_2(MoO_4)_3 + K_4Zn(MoO_4)_3$
28	55	15	30	$K_2Mo_2O_7 + K_2Zn_2(MoO_4)_3 + K_4Zn(MoO_4)_3$
29	58.3	16.7	25	$K_2Mo_2O_7 + K_2Zn_2(MoO_4)_3$
30	55.6	11.1	33.3	$K_2Mo_2O_7 + K_4Zn(MoO_4)_3$
31	55	5	40	$K_2MoO_4 + K_2Mo_2O_7 + K_4Zn(MoO_4)_3$

and slowly cooled in the furnace to room temperature. The heating temperature is 450–650 °C for the K_2O–ZnO–MoO_3 system and 600–850 °C for the K_2O–ZnO–WO_3 system. The temperature of the furnace was measured with a Pt–PtRh thermocouple and was precisely controlled to within ±2 °C up to 1200 °C with an intelligent controller. The above process should repeat several times until the X-ray pattern of the specimen showed no change upon successive heat treatment, which represented the equilibrium was achieved .The compositions of the samples prepared in the system K_2O–ZnO–MoO_3 are shown in Table 1 and the system K_2O–ZnO–WO_3 shown in Table 2.

Phase identification of the samples was carried out on a PANalytical X'Pert Pro diffractometer with Cu Ka radiation (45 kV × 40 mA) using continuous mode at a rate of $2\theta = 4°$/min.

The DTA investigation was conducted by NETZSCH-STA449C (Germany) in platinum crucible. The measurements were performed in the atmosphere of nitrogen in the temperature range 30–1200 °C. The heating rate was 10 K/min and the reference substance was α-Al_2O_3.

3. Result and discussion

3.1. Pseudo-binary system

3.1.1. K_2O–MoO_3 system

The K_2O–MoO_3 pseudo-binary system was reported by Spitsyn and Kuleshov [11] in 1951 and Caillet [12] in 1967. Six compounds were observed in their results, K_2MoO_4, $K_2Mo_2O_7$, $K_2Mo_3O_{10}$, $K_2Mo_4O_{13}$, $K_2Mo_6O_{19}$, $K_2Mo_8O_{25}$. The former four compounds melt incongruently in both reports [11,12]. The compounds $K_2Mo_6O_{19}$, $K_2Mo_8O_{25}$ shown in Spitsyn's reports seem more plausible, but in Caillet's [12] reports compound $K_2Mo_6O_{19}$ exists only between 542 °C and 562 °C. Under our experimental conditions, four compounds, K_2MoO_4, $K_2Mo_2O_7$, $K_2Mo_3O_{10}$, $K_2Mo_4O_{13}$ were found. Compound K_2MoO_4 belongs to the monoclinic system with space group $C2/m$. Its lattice parameters are $a = 12.340$ Å, $b = 6.081$ Å, $c = 7.538$ Å and $\beta = 115.74°$ [17]. The compound $K_2Mo_2O_7$ crystallizes in triclinic system with space group $P\bar{1}$. Its lattice parameters are $a = 7.510$ Å, $b = 7.240$ Å, $c = 6.950$ Å and $\alpha = 92.00°$, $\beta = 120.00°$, $\gamma = 82.50°$ [18]. Compound $K_2Mo_3O_{10}$ belongs to the monoclinic system with space group $C2/c$. Its lattice parameters are $a = 13.990$ Å, $b = 7.877$ Å, $c = 8.874$ Å and $\beta = 99.234°$ [19]. Compound $K_2Mo_4O_{13}$ belongs to a triclinic system with space group $P\bar{1}$. Its lattice parameters are $a = 7.972$ Å, $b = 8.352$ Å, $c = 10.990$ Å and $\alpha = 119.40°$, $\beta = 62.70°$, $\gamma = 109.80°$ [20].

3.1.2. K_2O–WO_3 system

The phase diagram of K_2O–WO_3 system has been reported in the literature for many times. Hoermann [13] reported it for the first time in 1928. In his reports, only three compounds were found, K_2WO_4, $K_2W_3O_{10}$, $K_2W_4O_{13}$. Except the three compounds, Guerin et al. [14] also showed three intermediate phases, $K_2W_2O_7$, $K_2W_6O_{19}$, $K_2W_8O_{25}$. They indicated that pure $K_2W_2O_7$ was difficult to obtain from K_2CO_3 and WO_3 but was prepared in pure form by heating K_2WO_4 and $K_2W_3O_{10}$

Table 2
List of phase identification for samples with different composition in the system K_2O–ZnO–WO_3

Samples	WO_3 (mol%)	ZnO (mol%)	K_2O (mol%)	Phase identification
1	33.3	0	66.7	$K_2WO_4 + K_2CO_3$
2	10	65	25	$ZnO + K_2WO_4 + K_2CO_3$
3	15	45	40	$ZnO + K_2WO_4 + K_2CO_3$
4	35	15	50	$ZnO + K_2WO_4 + K_2CO_3$
5	10	15	75	$ZnO + K_2WO_4 + K_2CO_3$
6	10	80	10	$ZnO + K_2WO_4$
7	25	50	25	$ZnO + K_2WO_4$
8	40	20	40	$ZnO + K_2WO_4$
9	20	65	15	$ZnO + K_2WO_4 + K_4Zn(WO_4)_3$
10	35	35	30	$ZnO + K_2WO_4 + K_4Zn(WO_4)_3$
11	30	50	20	$ZnO + K_4Zn(WO_4)_3$
12	20	75	5	$ZnO + K_4Zn(WO_4)_3 + ZnWO_4$
13	40	45	15	$ZnO + K_4Zn(WO_4)_3 + ZnWO_4$
14	50	30	20	$K_4Zn(WO_4)_3 + ZnWO_4$
15	70	25	5	$K_2W_6O_{19} + ZnWO_4 + WO_3$
16	85	10	5	$K_2W_6O_{19} + ZnWO_4 + WO_3$
17	75	15	10	$K_2W_6O_{19} + ZnWO_4$
18	60	35	5	$K_2W_4O_{13} + K_2W_6O_{19} + ZnWO_4$
19	70	20	10	$K_2W_4O_{13} + K_2W_6O_{19} + ZnWO_4$
20	65	25	10	$K_2W_4O_{13} + ZnWO_4$
21	62.5	27.5	10	$K_2W_3O_{10} + K_2W_4O_{13} + ZnWO_4$
22	75	5	20	$K_2W_3O_{10} + K_2W_4O_{13} + ZnWO_4$
23	65	20	15	$K_2W_3O_{10} + ZnWO_4$
24	60	25	15	$K_2W_2O_7 + K_2W_3O_{10} + ZnWO_4$
25	67.5	10	22.5	$K_2W_2O_7 + K_2W_3O_{10} + ZnWO_4$
26	60	20	20	$K_2W_2O_7 + ZnWO_4$
27	55	25	20	$K_2W_2O_7 + ZnWO_4 + K_4Zn(WO_4)_3$
28	60	10	30	$K_2W_2O_7 + ZnWO_4 + K_4Zn(WO_4)_3$
29	55.56	11.11	33.33	$K_2W_2O_7 + K_4Zn(WO_4)_3$
30	55	5	40	$K_2W_2O_7 + K_4Zn(WO_4)_3 + K_2WO_4$

at 580 °C for 300 h. The phase $K_2W_8O_{25}$ has some range of solid solution and melts incongruently in their reports [14]. Under our experimental conditions, five compounds, K_2WO_4, $K_2W_2O_7$, $K_2W_3O_{10}$, $K_2W_4O_{13}$ and $K_2W_6O_{19}$ were identified. This result is in good agreement with Chang's report [15]. Compound K_2WO_4 belongs to the monoclinic system with space group $C2/m$. Its lattice parameters are $a = 12.380$ Å, $b = 6.119$ Å, $c = 7.552$ Å and $\beta = 115.95°$ [21]. Compound $K_2W_2O_7$ also belongs to a monoclinic system with space group $P2_1/c$. Its lattice parameters are $a = 3.883$ Å, $b = 13.650$ Å, $c = 5.960$ Å and $\beta = 90.40°$ [22]. The compound $K_2W_3O_{10}$ also crystallizes in monoclinic system with space group $P2/c$. Its lattice parameters are $a = 10.940$ Å, $b = 3.864$ Å, $c = 31.950$ Å and $\beta = 108.44°$ [23]. Compound $K_2W_4O_{13}$ belongs to a hexagonal system with space group $P\bar{3}$. Its lattice parameters are $a = 15.560$ Å, $c = 3.746$ Å [24]. The compound $K_2W_6O_{19}$ crystallizes in orthorhombic system with space group $Pmmm$. Its lattice parameters are $a = 7.305$ Å, $b = 25.410$ Å, $c = 7.631$ Å [25].

3.1.3. ZnO–MoO_3 system

In ZnO–MoO_3 binary system, we found two compounds, $ZnMoO_4$ and $Zn_3Mo_2O_9$. This result is in good agreement with the phase diagram reported by Kohlmuller and Faurie [26]. The compound $ZnMoO_4$ belongs to a triclinic system with space group $P\bar{1}$. Its lattice parameters are $a = 8.367$ Å, $b = 9.691$ Å, $c = 6.964$ Å and $\alpha = 106.87°$, $\beta = 101.72°$, $\gamma = 96.73°$ [27]. Compound $Zn_3Mo_2O_9$ belongs to a monoclinic system with space group $P2_1/m$. Its lattice parameters are $a = 7.757$ Å, $b = 7.131$ Å, $c = 8.370$ Å and $\beta = 117.39°$ [28].

3.1.4. ZnO–WO_3 system

The phase diagram of ZnO–WO_3 has already been reported by Yanushkevich et al. [29] and Shchenev et al. [30]. In their reports, only one intermediate phase $ZnWO_4$ exists. Our result is in good agreement with the result reported by Yanushkevich and Shchenev. The compound $ZnWO_4$ belongs to monoclinic system with space group $P2/c$. Its lattice parameters are $a = 4.692$ Å, $b = 5.721$ Å, $c = 4.928$ Å and $\beta = 90.632°$ [31].

3.1.5. ZnO–K_2O system

In ZnO–K_2O binary system, no binary compound has been reported, and under our experimental conditions, no binary compound has been found.

The pseudo-binary system ZnO–K_2MoO_4 and ZnO–K_2WO_4 have been investigated by means of DTA and XRD methods. The XRD results indicate there is no intermediate compound in ZnO–K_2MoO_4 system and ZnO–K_2WO_4 system. DTA curves of different components are presented in Fig. 1 and Fig. 2. In our result, K_2MoO_4 melts congruently at 927 °C, which is in accordance with reports [11,12]. K_2WO_4 melts congruently at 921 °C, which is in accordance with reports [13–15]. Only endothermal peak of eutectic exists in the measurement temper-

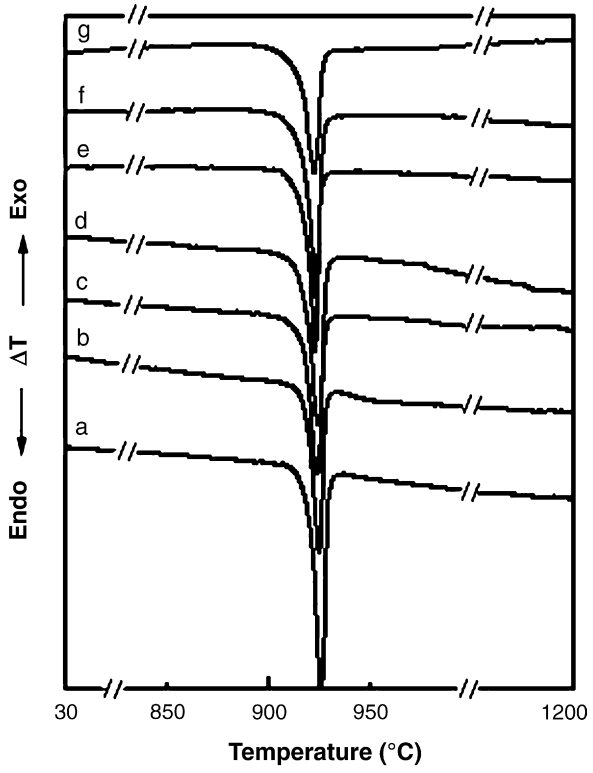

Fig. 1. DTA for different component K_2MoO_4 and ZnO (a) pure K_2MoO_4; (b) 95 mol% K_2MoO_4 + 5 mol% ZnO; (c) 90 mol% K_2MoO_4 + 10 mol% ZnO; (d) 85 mol% K_2MoO_4 + 15 mol% ZnO; (e) 70 mol% K_2MoO_4 + 30 mol% ZnO; (f) 50 mol% K_2MoO_4 + 50 mol% ZnO; (g) 20 mol% K_2MoO_4 + 80 mol% ZnO.

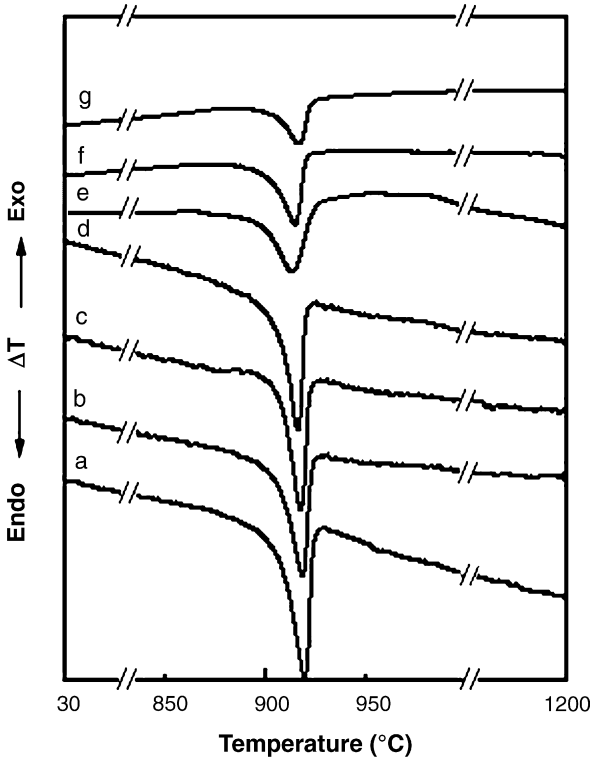

Fig. 2. DTA for different component K_2WO_4 and ZnO (a) pure K_2WO_4; (b) 95 mol% K_2WO_4 + 5 mol% ZnO; (c) 90 mol% K_2WO_4 + 10 mol% ZnO; (d) 85 mol% K_2WO_4 + 15 mol% ZnO (e) 70 mol% K_2WO_4 + 30 mol% ZnO; (f) 50 mol% K_2WO_4 + 50 mol% ZnO; (g) 20 mol% K_2WO_4 + 80 mol% ZnO.

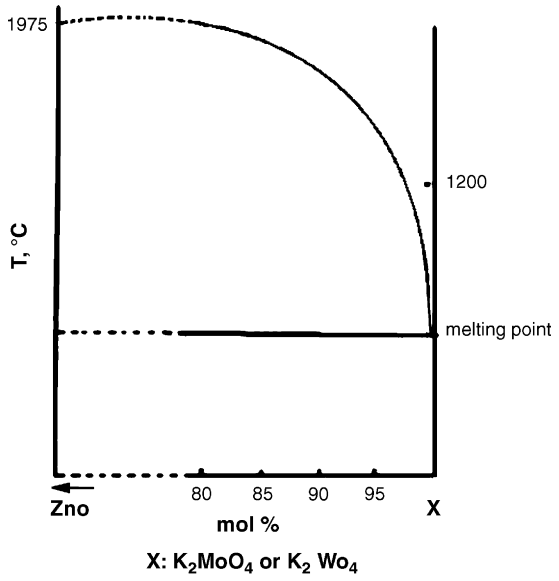

Fig. 3. The simulating phase diagram of systems ZnO–K_2MoO_4 and ZnO–K_2WO_4.

ature range 30–1200 °C. It shows that these are eutectic systems and the eutectic temperature is very close to the melt point of K_2MoO_4 and K_2WO_4, respectively. It shows the solid (K_2MoO_4 or K_2WO_4)-liquid equilibrium regions of the eutectic systems are small or degenerate and disappear in the low composition of K_2MoO_4 and K_2WO_4. When the temperature arrived at 1200 °C, we did not observe the endothermal peak of liquidus. That is to say, ZnO is not fusible in the melted state of the systems below 1200 °C. So the compounds K_2MoO_4 and K_2WO_4 are not suitable to be fluxes for ZnO crystal growth below 1200 °C. The simulating phase diagram of the pseudo-binary system ZnO–K_2MoO_4 and ZnO–K_2WO_4 is shown in Fig. 3.

3.2. Subsolidus phase relations of ternary system

3.2.1. Subsolidus phase relations of the K_2O–ZnO–MoO_3 system

The subsolidus phase relations of the K_2O–ZnO–MoO_3 system are shown in Fig. 3. Which consist of 11 three-phase regions, two ternary compounds and six binary compounds (K_2MoO_4, $K_2Mo_2O_7$, $K_2Mo_3O_{10}$, $K_2Mo_4O_{13}$, $ZnMoO_4$ and $Zn_3Mo_2O_9$). In K_2O–ZnO–MoO_3 pseudo-ternary system there exist two ternary compounds, $K_4Zn(MoO_4)_3$ and $K_2Zn_2(MoO_4)_3$ under our sintered condition which is consistent with Efremov's report [16]. The compound $K_4Zn(MoO_4)_3$ belongs to orthorhombic system with space group $P2_12_12_1$. Its lattice parameters are $a = 10.630$ Å, $b = 21.970$ Å, $c = 6.065$ Å [32]. Compound $K_2Zn_2(MoO_4)_3$ crystallizes in monoclinic system with space group $P2_1/c$. Its lattice parameters are $a = 7.000$ Å, $b = 9.010$ Å, $c = 20.680$ Å and $\beta = 111.65°$ [33]. From the phase relations of the K_2O–ZnO–MoO_3 system, the possible component regions for ZnO single crystal flux growth are ZnO–K_2MoO_4–K_2O, ZnO–K_2MoO_4–$K_4Zn(MoO_4)_3$, ZnO–$K_4Zn(MoO_4)_3$–$K_2Zn_2(MoO_4)_3$ and ZnO–$K_2Zn_2(MoO_4)_3$–$Zn_3Mo_2O_9$.

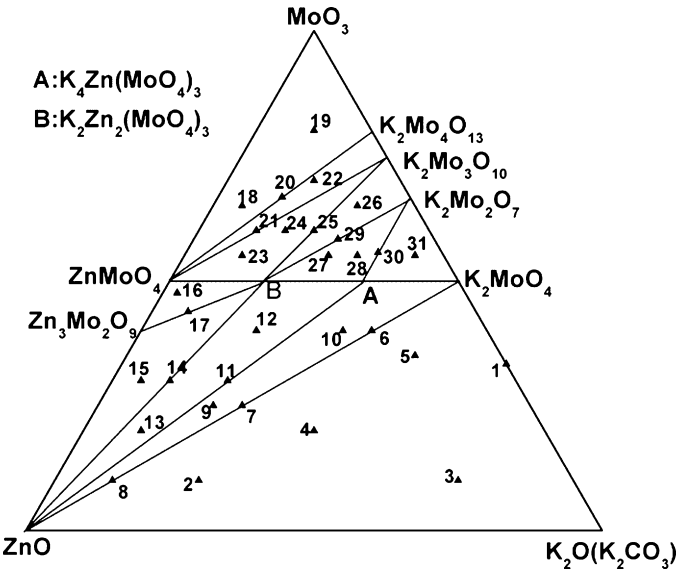

Fig. 4. Subsolidus phase relations of the system K_2O–ZnO–MoO_3.

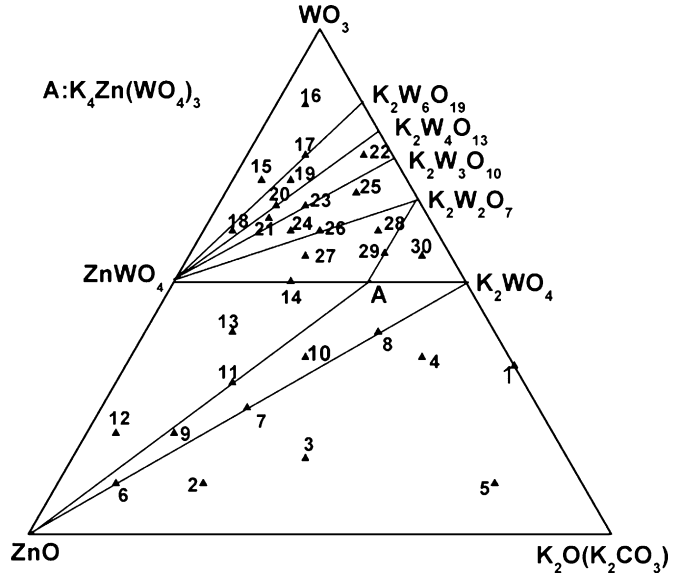

Fig. 5. Subsolidus phase relations of the system K_2O–ZnO–WO_3.

3.2.2. *Subsolidus phase relations of the K_2O–ZnO–WO_3 system*

In K_2O–ZnO–WO_3 pseudo-ternary system only one ternary compound $K_4Zn(WO_4)_3$ had been synthesized in our experiment. This is consistent with Efremov's report [16]. The subsolidus phase relations of the K_2O–ZnO–WO_3 system are shown in Fig. 4. The system consists of 9 three-phase regions, one ternary compound and six binary compounds (K_2WO_4, $K_2W_2O_7$, $K_2W_3O_{10}$, $K_2W_4O_{13}$, $K_2W_6O_{19}$ and $ZnWO_4$). The compound $K_4Zn(WO_4)_3$ belongs to triclinic system with space group $P\bar{1}$. Its lattice parameters are $a = 9.957$ Å, $b = 9.891$ Å, $c = 7.807$ Å and $\alpha = 105.55°$, $\beta = 106.16°$, $\gamma = 94.13°$ [34]. From the phase relations of the K_2O–ZnO–WO_3 system, the possible component regions for ZnO single crystal flux growth are ZnO–K_2WO_4–K_2O, ZnO–K_2WO_4–$K_4Zn(WO_4)_3$, ZnO–$K_4Zn(WO_4)_3$–$ZnWO_4$ (Fig. 5).

4. Conclusions

Phase relations in the system K_2O–ZnO–AO_3, where A is Mo or W, are investigated. The K_2O–ZnO–MoO_3 system consists of six binary compounds and two ternary compounds, $K_4Zn(MoO_4)_3$ and $K_2Zn_2(MoO_4)_3$. There are 11 three phase-regions in this system. The K_2O–ZnO–WO_3 system consists of six binary compounds and one ternary compound, $K_4Zn(WO_4)_3$. This system can be divided into 9 three-phase regions. DTA results indicated the compounds K_2MoO_4 and K_2WO_4 are not suitable to be fluxes for ZnO crystal growth.

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