# Phase Equilibria and Crystal Chemistry in the System CaO-Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub>

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The phase relations in the system CaO-Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> at 1400 and 1500 °C have been determined. The phase relations presented are in agreement with the calculated phase diagram by Udalov et al. (*Russ. J. Inorg. Chem.*, 1979, 24(10), p 1549-1553). In addition ternary solid solution series of the binary compounds YAM (Y<sub>4</sub>Al<sub>2</sub>O<sub>9</sub>), YAP (YAlO<sub>3</sub>), and YAG (Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>) could be described.

Keywords crystal chemistry, phase equilibria, Y-gehlenite, YAG, YAM, YAP

### 1. Introduction

Luminescent materials are used in many technical applications, e.g., in solid state lasers. A commonly utilized material for solid state lasers is Nd: YAG. Due to its crystal structure doped gehlenite could be also a promising material for luminescent applications. Gehlenite CaAl<sub>2</sub>SiO<sub>7</sub>, a member of the melilite group is a mineral with wide variation in crystal chemistry. By joint substitution of Ca<sup>2+</sup> with Y<sup>3+</sup> and by Si<sup>4+</sup> with Al<sup>3+</sup> the isostructural, compound CaYAl<sub>3</sub>O<sub>7</sub> will be formed. This material is also called Y-gehlenite or Ca-Y-Al-melilite (CYAM). For technical applications and synthesis it is inevitable to know crystal chemical aspects concerning promising dopands and phase equilibria in the system CaO-Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub>.

# 2. Previous Work

Many publications describe the important system CaO-Al<sub>2</sub>O<sub>3</sub>.<sup>[1,2]</sup> A calculated phase diagram with good agreement to former experimental work was published 1990.<sup>[3]</sup> In this calculated system four stable phase C<sub>3</sub>A (Ca<sub>3</sub>Al<sub>2</sub>O<sub>6</sub>), CA (CaAl<sub>2</sub>O<sub>4</sub>), CA<sub>2</sub> (CaAl<sub>4</sub>O<sub>7</sub>), and CA<sub>6</sub> (CaAl<sub>12</sub>O<sub>19</sub>) are presented. In aquatic environments a further phase C<sub>12</sub>A<sub>7</sub> (Ca<sub>12</sub>Al<sub>14</sub>O<sub>33</sub>) exists.<sup>[4]</sup> The melting behavior whether congruent or incongruent of the phases C<sub>12</sub>A<sub>7</sub>, CA, and CA<sub>2</sub> is still under discussion. In the binary system a eutectic melt is formed with a composition near to C<sub>12</sub>A<sub>7</sub> at 1365 °C.

The system CaO-Y<sub>2</sub>O<sub>3</sub> was investigated by DTA and annealing and quenching method.<sup>[5]</sup> A later calculation of

the system differs in the existence of high temperature phases and the liquidus area.<sup>[6]</sup> In both diagrams exist in the subsolidus region up to 1800 °C only the solid solutions of the components. The same results are described in newer publications investigating the CaO-Y<sub>2</sub>O<sub>3</sub> subsystem at 900 °C<sup>[7]</sup> and 1400 °C.<sup>[8]</sup>

The Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> phase diagram consists of the two components Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub> and three intermediate binary phases, yttrium aluminum monocline (YAM, Y<sub>4</sub>Al<sub>2</sub>O<sub>9</sub>), yttrium aluminum perovskite (YAP, YAlO<sub>3</sub>), and yttrium aluminum garnet (YAG, Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>). About the melting behavior and the stability of YAP there is a dispute discussion in former experimental studies. The discussion is summarized in a review<sup>[9]</sup> and in studies modeling the phase diagram with thermodynamic calculations.<sup>[10-12]</sup> Whether YAP melts congruent or incongruent is not significant for studying subsolidus reactions. The stability of YAG from room temperature to the congruent melting point at 1942 °C is certain.<sup>[13,14]</sup> The stability of YAM and YAP is discussed controversial. In former studies they were found to be metastable.<sup>[13]</sup> Studies by high temperature neutron diffraction determined all intermediate phases to be stable from room temperature until melting.<sup>[14]</sup> In the literature deviations of stoichiometry in YAP and YAG are reported.<sup>[15,16]</sup> According to calculated defect formation energies an antisite disorder is liable for Al-excess in YAP and Y-excess in YAG.<sup>[16]</sup>

For the ternary system CaO-Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> one calculated phase diagram is published<sup>[17]</sup> (Fig. 1). The calculations are supported and verified by experimental data. CaYAlO<sub>4</sub> and CaYAl<sub>3</sub>O<sub>7</sub> (Y-gehlenite) are the stable ternary phases. In the phase diagram possible solid solutions are not considered. In the pseudo binary system  $C_{12}A_7$ -CaYAlO<sub>4</sub> the formation of bulk glass was studied.<sup>[17]</sup> The recrystallization of the glass material results at 1000 °C in a phase equilibrium of  $C_{12}A_7$  and CaYAlO<sub>4</sub>.

### 3. Experimental Methods

For the determination of the phase relations in the ternary system  $CaO-Al_2O_3-Y_2O_3$  several mixtures were prepared (Table 1). The mixtures were chosen appropriate to reach

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Fig. 1 Phase diagram CaO-Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> in mole percent (excerpt from Phase Equilibria Diagrams Database 3.1 according to Udalov et al.<sup>[17]</sup>)

Table 1	Mixtures in	the system	CaO-Al <sub>2</sub> O <sub>3</sub> -Y <sub>2</sub> O <sub>3</sub>
in mole	percent		

Sample	CaO	Y <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>
CAY-1	33.33	16.67	50.00
CAY-2	31.56	24.37	44.07
CAY-3	23.08	18.27	58.65
CAY-4	36.87	15.23	47.90
CAY-5	40.05	13.71	46.24
CAY-6	14.00	14.00	72.00
CAY-7	1.50	44.00	54.50
CAY-8	61.00	9.00	30.00
CAY-9	69.00	20.00	11.00
CAY-10	20.00	50.00	30.00
CAY-11	23.00	63.00	14.00
CAY-12	32.00	14.00	54.00
CAY-13	10.00	35.00	55.00
CAY-14	10.00	50.00	40.00
CAY-15	55.00	8.00	37.00
CAY-16	5.00	10.00	85.00

multiphase paragenesis. The high-purity starting materials (>99.9%) CaCO<sub>3</sub> (Wako), Y<sub>2</sub>O<sub>3</sub> (Alfa Aesar), and Al<sub>2</sub>O<sub>3</sub> (Shin Etsu) were mixed in a tungsten-carbide mortar and calcinated at 1000 °C for 24 h. The powders were hydrostatically pressed into rods. Suitable samples were

equilibrated at 1400 and 1500 °C for 32, respectively, 7 days in a chamber furnace in air. The furnace temperature controller was compared with a calibrated external thermocouple. The external thermocouple was made of Pt30Rh-Pt6Rh and calibrated against the melting-point of gold (1064.4 °C), diopside (1394 °C), and pseudowollastonite (1547 °C) according to IPTS-68. The measurement setup reaches an accuracy of temperature about  $\pm 10$  °C. The quenching was done in air.

The chemical composition of the phases was analyzed by electron probe microanalysis (EPMA) with a Jeol JXA-8200. As standards corundum (Al<sub>2</sub>O<sub>3</sub>), pseudowollastonite (Ca<sub>3</sub>Si<sub>3</sub>O<sub>9</sub>), and metallic yttrium (Y°) were used. All measured data were corrected by the ZAF correction procedure. In backscattered electron images the measurement points for wavelength dispersive spectrometer analysis were chosen and the homogeneity was assured (Fig. 2). Only analyses of a total within a range of 98-102% except YAG were taken for evaluation. The error of the EPMA measurement is assumed to be less than 1 mol.%.

Some Al-rich samples (CAY6, 7, 13, 16) in spite of sintering for 32 days were to small-grained for EPMA analysis (Fig. 3). The samples were prepared in a sample holder made of single crystal silicon and were investigated by x-ray powder diffraction using a Siemens D5000 diffractometer with CuK $\alpha$  radiation. The powder patterns were qualitatively analyzed using the software package EVA



Fig. 2 BSE-image of sample CAY-2 after sintering 32 days at 1400  $^{\circ}\mathrm{C}$  in air



**Fig. 3** BSE-image of sample CAY-7 after sintering 32 days at 1400 °C in air. The grains are too small for EPMA-analysis. The phase content was studied by XRD

13 from Bruker AXS. The structures for phase determination are given in Table 2.

## 4. Results

In the binary system CaO- $Y_2O_3$  only the components exist as phases below temperatures of 1500 °C. Each of the components forms a limited solid solution (Fig. 4). With increasing temperature the amount of Yttrium ions in CaO is raised. The microprobe analysis of  $Y_2O_3$  has a rather high error in the measurement (Table 3), which is caused by the



Fig. 4 Binary Phase diagram  $CaO-Y_2O_3$  in mole percent with limited solid solutions

Ta	ble 3	EPMA	analysis	of fore	ign ion	substitı	ıtion	
in	the bin	nary sys	tem CaC	$Y_2O_3$	at 1400	and 15	600 °	С
in	mixtur	e CAY-	9					

DI	Ca-oxide			Y-oxide		
Phase mol.%	CaO	Al <sub>2</sub> O <sub>3</sub>	Y <sub>2</sub> O <sub>3</sub>	CaO	Al <sub>2</sub> O <sub>3</sub>	Y <sub>2</sub> O <sub>3</sub>
1400 °C	$97.9\pm0.1$	0	$2.1\pm0.1$	$1.2\pm0.6$	$0.1\pm0.1$	$98.7\pm0.6$
1500 °C	$96.5\pm0.1$	0	$3.5\pm0.1$	$0.9\pm0.4$	0	$99.1\pm0.4$

**ICDD PDF-number** Structure Author 33-40 YAG National Bureau of Standards (U.S.), Monograph; 19(1982); 11 76-665  $CA_6$ Kato, K., Saalfeld, H.; Neues Jahrbuch für Mineralogie-Abhandlungen; 109 (1968); 192 23-1037 Baldock et al.; Journal of Applied Crystallography; 10 (1970); 188  $CA_2$ 70-1677 YAP Diehl, R., Brandt, G.; Materials Research Bulletin; 10 (1975); 85 Y-gehlenite Kuz'micheva, G.M., Mukhin, B.V., Rybakov, V.B., Denisov, A.L., Zharikov, 77-1120 E.V., Smirnov, V.A; Zhurnal Neorganicheskoi Khimii; 40 (1995); 569 Cox, D.E., Moodenbaugh, A.R., Sleight, A.W., Chen, H.Y.; National Bureau of 75-1864 Corundum Standards (U.S.), Special Publication; 567 (1980); 189

 Table 2
 Structures used for x-ray powder diffraction analysis

splitting up Y L $\beta$  peak with several maximums. The limited solid solution of the components CaO and Y<sub>2</sub>O<sub>3</sub> in the binary system is in agreement with literature data.<sup>[5,6]</sup> The EPMA analyses show a substitution of aluminum for yttrium in Y<sub>2</sub>O<sub>3</sub>.

The analyzed phases  $CaAl_2O_4$ ,  $CaAl_4O_7$ , and  $CaAl_{12}O_{19}$  within the binary system  $CaO-Al_2O_3$  have stoichiometric composition and form no solid solution. The remaining binary phases are coexisting with melt. This area in the phase diagram cannot be analyzed without quenching in water. The qualitative melt phase is showed by the dotted line in the phase diagram.

Within the binary system  $Y_2O_3$ - $Al_2O_3$  there are three stable intermediate phases, YAM ( $Y_4Al_2O_9$ ), YAP (YAIO<sub>3</sub>), and YAG ( $Y_3Al_5O_{12}$ ).

Table 4EPMA-analysis of YAM phase in samplesCAY-10, -11, -14 at 1400 and 1500 °C in comparisonto stoichiometric YAM composition

Sample	Al <sub>2</sub> O <sub>3</sub> , mol.%	CaO, mol.%	Y <sub>2</sub> O <sub>3</sub> , mol.%
CAY10-1400-32d	$34.6\pm0.2$	$1.5 \pm 0.1$	$63.9\pm0.1$
CAY11-1400-32d	$35.4\pm0.2$	$1.5\pm0.2$	$63.2\pm0.2$
CAY14-1400-32d	$34.6\pm0.2$	$1.4 \pm 0.1$	$64.1\pm0.2$
Midpoint 1400 °C	34.83	1.55	63.62
CAY10-1500-7d	$34.6\pm0.1$	$1.8\pm0.1$	$63.6\pm0.1$
CAY11-1500-7d	$34.6\pm0.2$	$1.9\pm0.1$	$63.5\pm0.2$
CAY14-1500-7d	$34.7\pm0.1$	$1.8\pm0.1$	$63.5\pm0.1$
Midpoint 1500 °C	34.62	1.83	63.54
Stoichiometric Y <sub>4</sub> Al <sub>2</sub> O <sub>9</sub>	33.33	0.00	66.67

The errors were calculated from the averages and standard derivations of a minimum of five measurements The EPMA analysis for mixtures CAY-10, -11, and -14 show deviations of the stoichiometric YAM composition (Table 4). YAM forms a solid solution, which can be described by the formula:

$$Y_{4-x}Ca_{1/3x}Al_{2+2/3x}O_{9-1/6x}$$
  
with  $0 < x < 0.14 (1400 \,^{\circ}C \text{ and } 1500 \,^{\circ}C).$ 

Yttrium positions in the YAM structure are substituted by calcium and aluminum ions. For charge balance oxygen vacancies have to be formed.

The substitution mechanism in principle can be described as

$$3Y^{3+} \leftrightarrow Ca^{2+} + 2Al^{3+} + \frac{1}{2}$$

In case of YAP (YAlO<sub>3</sub>) a similar substitution mechanism is valid in the ternary system  $CaO-Al_2O_3-Y_2O_3$ . The YAG EPMA analysis differs from the stoichiometric

Table 5EPMA-analysis of YAP phase in samplesCAY-2, -7, -13, -14 at 1400 and 1500 °C in comparisonto stoichiometric YAP composition

Sample	Al <sub>2</sub> O <sub>3</sub> , mol.%	CaO, mol.%	Y <sub>2</sub> O <sub>3</sub> , mol.%
CAY13-1400-32d	$51.0 \pm 0.1$	$3.5 \pm 0.3$	$45.6\pm0.3$
CAY14-1400-32d	$50.9\pm0.4$	$3.0 \pm 0.3$	$46.1\pm0.1$
CAY2-1500-7d	$50.6\pm0.2$	$4.1 \pm 0.4$	$45.3\pm0.3$
CAY13-1500-7d	$50.6\pm0.4$	$3.6 \pm 0.3$	$45.8\pm0.6$
CAY14-1500-7d	$50.9\pm0.1$	$3.4 \pm 0.3$	$45.7\pm0.3$
Stoichiometric YAlO <sub>3</sub>	50.00	0.00	50.00

The errors were calculated from the averages and standard derivations of a minimum of five measurements



Fig. 5 X-ray powder diffraction diagram with qualitative evaluation for sample CAY-7 sintered 32 days at 1400 °C

composition (Table 5) and results in the proposed following solid solution series:

$$\begin{array}{l} Y_{1-x}Ca_{1/2x}Al_{1+1/2x}O_{3-1/2x}\\ \text{with} \quad 0 \leq x \leq 0.07 \ (1400\ ^{\circ}\text{C} \ \text{and} \ 1500\ ^{\circ}\text{C}) \end{array}$$

The solid solution series was verified by XRD measurement. The mixture CAY-7 was placed in the proposed two phase area. The XRD-analysis showed only two phase equilibria (Fig. 5), where all peaks can be interpreted with YAM and YAG structures.

Table 6 Coexisting phases in the system  $CaO-Al_2O_3-Y_2O_3$  at 1400 and 1500 °C

Coexisting phases at 1400 °C	Coexisting phases at 1500 °C
$Ca-oxide_{ss} + Y-oxide_{ss} + CaYAlO_4$	$Ca-oxide_{ss} + Y-oxide_{ss} + CaYAlO_4$
$\text{Y-oxide}_{\text{ss}} + \text{CaYAlO}_4 + \text{YAM}_{\text{ss}}$	$\text{Y-oxide}_{\text{ss}} + \text{CaYAlO}_4 + \text{YAM}_{\text{ss}}$
$CaYAlO_4 + YAM_{ss} + YAP_{ss}$	$CaYAlO_4 + YAM_{ss} + YAP_{ss}$
CaYAlO <sub>4</sub> + YAP <sub>ss</sub> + Y-gehlenite	CaYAlO <sub>4</sub> + YAP <sub>ss</sub> + Y-gehlenite
YAP <sub>ss</sub> + Y-gehlenite + YAG <sub>ss</sub>	$YAP_{ss} + Y$ -gehlenite + $YAG_{ss}$
$Y$ -gehlenite + $YAG_{ss}$ + $CA$	$Y$ -gehlenite + $YAG_{ss}$ + $CA_2$
$YAG_{ss} + CA + CA_2$	$YAG_{ss} + CA_2 + CA_6$
$YAG_{ss} + CA_2 + CA_6$	$YAG_{ss} + CA_6 + corundum$
$YAG_{ss} + CA_6 + corundum$	
CaYAlO <sub>4</sub> + Y-gehlenite + CA	

The mixtures on the Al-rich and Y-rich side of YAG have the same YAG chemistry and show no exchange of Y and Al. The analyzed Al and Y content of YAG deviates by about 1.3 mol.% on the Al-rich side from the stoichiometric composition. Those difficulties for YAG analyses concerning EPMA quantification has already been reported.<sup>[18]</sup> The total of measurement of YAG is about 103% and the chemical analysis gives rise to an incorporation of 0.4% CaO into the garnet structure. This amount is very probably substituted, but within accuracy of measurement a substitution mechanism can only be interpreted as

$$Y^{3+} \leftrightarrow Ca^{2+} + {}^1\!/_2 \square^{2-}$$

The same mechanism is already described in the literature.<sup>[19]</sup>

The CaO-Al<sub>2</sub>O<sub>3</sub> system and ternary phases CaYAlO<sub>4</sub> and Y-gehlenite exhibit no solid solution series. The coexisting phases at 1400 and 1500 °C differ (Table 6). Between 1400 and 1500 °C a solid state reaction takes place and changes the coexisting phases:

$$3CaYAl_3O_7 + CaAl_4O_7 \leftrightarrow 4CaAl_2O_4 + Y_3Al_5O_{12}$$

The results can be summarized in the experimental phase diagrams for the system CaO-Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> at 1400 °C (Fig. 6) and 1500 °C (Fig. 7). For both temperatures a liquid phase appears as indicated.



Fig. 6 Phase relations in the system CaO-Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> at 1400 °C in air in mole percent



Fig. 7 Phase relations in the system CaO-Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> at 1500 °C in air in mole percent

# 5. Conclusions

The phase relations in the system CaO-Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> at 1400 and 1500 °C have been determined. The phase relations presented are in agreement with the calculated phase diagram by Udalov et al.<sup>[17]</sup> In addition ternary solid solution series of the binary compounds YAM (Y<sub>4</sub>Al<sub>2</sub>O<sub>9</sub>) and YAP (YAlO<sub>3</sub>) could be described. In the temperature range between 1400 and 1500 °C a solid state reaction takes place and some phase relations change.

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