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Subsolidus Phase Relationships in Part of the System Si,Al,Y/N,O: The System Si_3N_4–AlN–YN–Al_2O_3–Y_2O_3

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The subsolidus phase relationships in the system Si,Al,Y/N,O were determined. Thirty-nine compatibility tetrahedra were established in the region Si_3N_4–AlN–Al_2O_3–Y_2O_3. The subsolidus phase relationships in the region Si_3N_4–AlN–YN–Y_2O_3 have also been studied. Only one compound, 2YN:Si_3N_4, was confirmed in the binary system Si_3N_4–YN. The solubility limits of the α'-SiAlON on the Si_3N_4–YN:3AlN join were determined to range from m = 1.3 to m = 2.4 in the formula $Y_{m/3}Si_{12-m}Al_mN_{16}$. No quinary compound was found. Seven compatibility tetrahedra were established in the region Si_3N_4–AlN–YN–Y_2O_3. [Key words: phases, silicon, aluminum, yttrium, nitrogen.]

I. Introduction

It is known that metal oxide additives are needed to aid densification of silicon nitride ceramics. During sintering, the metal oxide additives and silicon nitride form a eutectic melt which aids densification. The liquid composition affects the microstructure development and, hence, the properties of silicon nitride ceramics. The additives also determine the nature of the grain-boundary phases which affect the properties of the silicon nitride ceramics.

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Yttrium oxide and aluminum oxide are two of the most commonly used additives for densifying silicon nitride by either hot-pressing or pressureless sintering. Phase relationships in the system Si_3N_4–SiO_2–AlN–Al_2O_3–YN–Y_2O_3 and their subsystems are of special interest because many of the commercial silicon nitride ceramics are found in these systems. The systems Si_3N_4–SiO_2–AlN–Al_2O_3[1] and Si_3N_4–SiO_2–Y_2O_3[2] have been studied in detail. The subsolidus phase relationships in part of the system Si_3N_4–SiO_2–AlN–Al_2O_3–YN–Y_2O_3 have also been studied in this laboratory.[3] We reported the subsolidus compatibility relationships in the space bounded by the components Si_3N_4–β-SiAlON–Al_2O_3–SiO_2 and Y_2O_3. Figure 1 reflects those data, as well as new information about the subsolidus relationships in the quasi-quaternary system containing the compounds Si_3N_4–AlN–Al_2O_3–Y_2O_3. This part of the subsystem includes the AlN polytypoids[4] and α'-SiAlON[5] solid solutions which may be of importance for developing useful materials for technical applications. Some of the literature data[6] published since our last paper are also presented.

Most of the early work in the system Si,Al,Y/N,O has been restricted to the region bounded by Si_3N_4–β-SiAlON–Al_2O_3–SiO_2 and Y_2O_3, which does not include the solid solution α'-SiAlON. Huang *et al.* reported the α'-SiAlON formation in the system Si_3N_4–AlN–Y_2O_3[7] and Si_3N_4–AlN–rare-earth oxide.[8] The systems Si_3N_4–AlN–YN and Si_3N_4–YN:3AlN–Al_2O_3:AlN have also been studied by these authors.[9] In recent years, with the emergence of α'-SiAlON, information in the nitrogen-rich part of the system Si,Al,Y/N,O became necessary. The present paper completes the phase studies in the entire system Si,Al,Y/N,O.

II. Experimental Procedure

The starting powders used were α-Si_3N_4 (LC12, Herman C. Starck, Goslar, FRG), AlN (Grade A, Herman C. Starck),

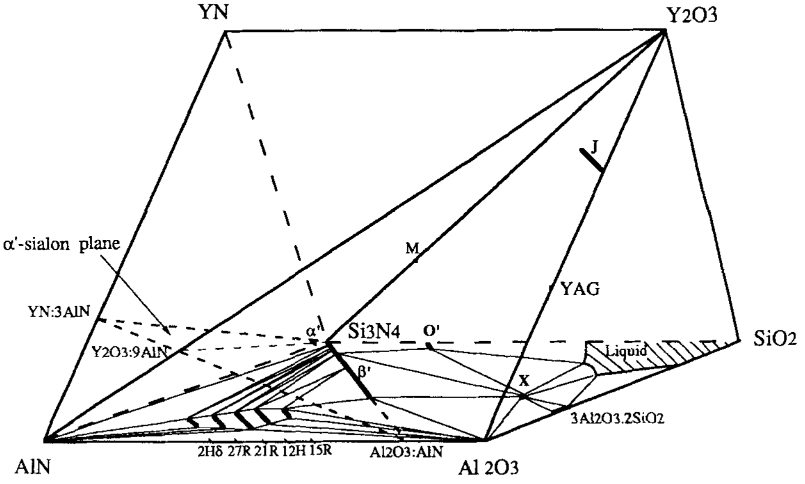

Fig. 1. Representation of Y-SiAlON system showing phases occurring in the region bound by Si_3N_4, Y_2O_3, Al_2O_3, and AlN, and Si-Al-O-N behavior diagram at 1700°C.

Table I. Compositions Studied in the System Si_3N_4–AlN–Y_2O_3

Composition (wt%)				Firing conditions		
Si_3N_4*	AlN†	Al_2O_3	Y_2O_3	T (C°)	Time (h)	Phases present
66.20	26.22	5.24	2.34	1850	1	α′, β′, 27R, 21R, 2H§
62.65	25.95	8.33	3.07	1800	1	β′, α′, 12H
56.46	29.96	9.49	4.09	1800	1	α′, β′, 12H
46.96	41.00	9.69	2.34	1850	1	α′, 21R, 27R, β′, 12H
46.94	43.99	6.73	2.34	1800	1	α′, β′, 27R, 2H§
46.15	38.70	12.09	3.06	1800	1	α′, 12H, β′, 21R
43.71	21.45	18.19	16.16	1575	1	12H, YAG, α′, β′
41.87	19.81	24.00	14.31	1600	1	YAG, β′, 12H
30.08	21.06	34.56	14.30	1600	1	β′, 15R, YAG
26.66	35.67	21.03	16.63	1575	1	YAG, 12H, α′, 21R
26.24	30.55	28.91	14.30	1600	1	YAG, 15R, β′
23.86	40.16	18.63	17.35	1575	1	YAG, 21R, 12H, M
22.53	43.48	16.65	17.35	1600	1	M, 21R, 27R
19.50	50.28	12.87	17.34	1600	1	M, 27R
18.55	34.41	11.05	35.99	1700	1	M, Jss, 27R, 21R
17.22	37.72	9.07	35.99	1700	1	M, Jss, 2H§
14.20	44.52	5.31	35.98	1700	1	M, Jss, AlN, 2H§
13.46	17.96	54.29	14.28	1600	1	YAG, 15R, Al_2O_3
13.37	16.61	18.48	51.54	1600	1	YAG, M, Jss, 21R
13.19	23.12	0	63.69	1700	2	Jss, AlN
9.68	37.41	33.87	19.04	1650	1	YAG, 12H, 15R
8.35	25.93	51.44	14.27	1650	1	15R, YAG, Al_2O_3
7.67	42.12	31.18	19.03	1650	1	YAG, 12H
6.37	46.63	27.97	19.02	1650	1	YAG, 21R, 27R
6.37	46.63	19.79	27.21	1650	1	YAG, AlN, 27R
5.47	50.80	24.71	19.03	1600	1	YAG, 27R, 2H§
5.47	50.80	16.53	27.20	1600	1	YAG, AlN, 27R
5.34	33.00	26.98	34.67	1750	1	YAG, 2H§
5.32	28.50	51.91	14.27	1650	1	YAG, 15R, Al_2O_3
3.72	31.99	50.00	14.27	1650	1	YAG, Al_2O_3, 15R, 12H
2.65	59.51	18.82	19.02	1650	1	YAG, 2H§, AlN
2.65	59.51	10.64	27.20	1650	1	AlN, YAG, Jss
1.53	41.75	42.46	14.27	1650	1	YAG, AlN, 21R, Al_2O_3

*Containing 2 wt% O. †Containing 1.3 wt% O.

α-Al_2O_3, and Y_2O_3. The oxygen content of the nitride powders was taken into account in computing the compositions. In the first part of the present paper, the compositions investigated were restricted to the region Si_3N_4, AlN, Al_2O_3, and Y_2O_3. The compositions studied are listed in Table I. In the later part of this paper, experimental results in the region including compound YN are reported. The compound YN used in this study was prepared in this laboratory.

Compositions without the compound YN were mixed in an alumina jar using 2-propanol in a planetary mill for 30 min. Mixtures were dried and pressed into disks 10 mm in diameter and were then isostatically pressed under a pressure of 300 MPa. All of the specimens were fired in a graphite-resistant furnace under static nitrogen of one atmospheric pressure for 1 h. The temperatures varied from 1550° to 1850°C. It was assumed that subsolidus equilibrium was attained when unreacted α-Si_3N_4 was no longer detected and no apparent liquid phase could be observed (i.e., no sintering occurred). Only specimens having less than 2% weight loss after firing were used for the data analysis. The phases present were identified by X-ray diffraction.

For compositions containing YN, batch mixtures without YN were first ground under 2-propanol in an agate mortar and pestle. The mixtures were dried and YN powder was

Table II. Compositions Studied in the System Si_3N_4–AlN–YN–Y_2O_3

Composition (wt%)					Firing conditions		
Si_3N_4*	AlN†	YN‡	Y_2O_3	Al_2O_3	T (C°)	Time (h)	Phases present§
82.00	12.68	5.31			1800	2	α′,¶ β-Si_3N_4
77.87	12.04	10.08			1800	2	α′**
67.15		32.85			1800	2	$Y_2Si_3N_6$, β-Si_3N_4, M
61.70	21.13	17.17			1800	2	α′,†† $Y_2Si_3N_6$, AlN
57.68		42.32			1800	2	$Y_2Si_3N_6$, M, β-Si_3N_4
47.61		52.39			1800	2	$Y_2Si_3N_6$, M
40.53		59.47			1800	2	$Y_2Si_3N_6$, M, J
25.42		74.58			1800	2	$Y_2Si_3N_6$, Y_2O_3, YN
16.51		52.86	29.00	1.64	1700	2	Y_2O_3, $Y_2Si_3N_6$, YN
14.42		46.16	37.99	1.43	1700	2	Y_2O_3, $Y_2Si_3N_6$, YN
13.61	5.89	44.38	32.46	3.66	1800	2	Y_2O_3, $Y_2Si_3N_6$, YN, AlN
11.71	5.07	38.18	41.89	3.15	1700	2	Y_2O_3, $Y_2Si_3N_6$, YN
8.05	15.28	44.28	32.39		1850	2	Y_2O_3, YN, Y_3O_3N AlN, $Y_2Si_3N_6$
7.51		44.10	48.38		1800	2	Y_2O_3, Y_3O_3N, $Y_2Si_3N_6$, YN
5.94		65.37	28.69		1800	2	Y_2O_3, YN, Y_3O_3N, $Y_2Si_3N_6$
5.79	20.31	55.25	18.65		1800	2	YN, Y_2O_3, AlN, Y_3O_3N, $Y_2Si_3N_6$
	27.52	34.56	37.92		1900	1	YN, Y_2O_3, AlN
	21.29	20.05	58.66		1900	1	Y_2O_3, YN, Y_3O_3N, AlN

*Containing 2.0 wt% O. †Containing 1.4 wt% O. ‡Containing 9 wt% C. §Residual carbon was not listed; M is melilite; J is J phase; a′ is a′-SiAlON. ¶a = 7.810 Å, c = 5.681 Å. **a = 7.821 Å, c = 5.693 Å; ††a = 7.863 Å, c = 5.731 Å.

Table III. Subsolidus Compatibility Tetrahedra in Si_3N_4–AlN–Al_2O_3–Y_2O_3*

Al_2O_3—β_{60}—15R—YAG	Al_2O_3—15R—15R′—YAG
Al_2O_3—15R′—12H′—YAG	Al_2O_3—12H′—21R′—YAG
Al_2O_3—21R′—AlN—YAG	15R—15R′—12H—12H′—YAG
12H—12H′—21R—21R′—YAG	21R—21R′—27R—27R′—YAG
27R—27R′—$2H^{\delta}$—$2H^{\delta\prime}$—YAG	2H—$2H^{\delta\prime}$—AlN—YAG
21R′—27R′—AlN—YAG	27R′—$2H^{\delta\prime}$—AlN—YAG
21R—27R—YAG—*Jss*	27R—$2H^{\delta}$—YAG—*Jss*
$2H^{\delta}$—AlN—YAG—*Jss*	AlN—YAG—*Jss*—YAM
AlN—YAM—J—Y_2O_3	β_{60}—β_{25}—15R—YAG
β_{25}—15R—12H—YAG	β_{25}—β_{10}—12H—YAG
β_{10}—α'—12H—YAG	α'—12H—21R—β_{10}
α'—21R—β_{10}—β_8	α'—21R—β_8—27R
α'—β_8—27R—β_2	α'—27R—β_5—$2H^{\delta}$
α'—β_5—$2H^{\delta}$—β_2	α'—$2H^{\delta}$—β_2—AlN
α'—β_2—AlN—Si_3N_4	α'—12H—21R—YAG
α'—21R—YAG—M	α'—21R—27R—M
α'—27R—$2H^{\delta}$—M	α'—2H—AlN—M
M—21R—YAG—*Jss*M—21R—27R—*Jss*	M—21R—27R—*Jss*
M—27R—$2H^{\delta}$—*Jss*	M—$2H^{8}$—AlN—*Jss*
M—AlN—*Jss*—J	

*YAM is $2Y_2O_3 \cdot Al_2O_3$; J is $2Y_2O_3 \cdot Si_2N_2O$; *Jss* is $2Y_2O_3 \cdot Al_2O_3$–$Y_2O_3 \cdot Si_2N_2O$; M is $Si_3N_4 \cdot Y_2O_3$; 15R, 12H, 21R, 27R, $2H^{\delta}$ are Si-rich terminals of AlN polytypoids; 15R′, 12H′, 21R′, 27R′, $2H^{\delta\prime}$ are Al-rich terminals of AlN polytypoids.

then added, using a dry box under flowing nitrogen with an agate mortar and pestle. The samples were compacted and fired at 1700° to 1900°C for 2 h under a static nitrogen atmosphere in a graphite-resistant furnace, which was vacuum-pumped to 30 mtorr (1 torr ~ 1.33 × 10^2 Pa) before heating. Each experimental run from sample preparation to X-ray analysis was made on the same day in order to prevent hydrolysis. Table II lists the starting compositions, firing conditions, and the resulting phases. The solubility limits of the single-phase α'-SiAlON solid solution were determined by the unit cell dimensions, based on the revised equations $a = 7.752 + 0.045m + 0.009n$ and $c = 5.620 + 0.048m + 0.009n$ ($Y_{m/3}Si_{12-(m+n)}Al_{m+n}O_nN_{16-n}$).[9]

The YN powder was prepared by using Y_2O_3 and carbon black as starting materials in a thermoreduction reaction, as indicated by the equation

$$Y_2O_3 + 3C + N_2(g) \rightarrow 2YN + 3CO \quad (1)$$

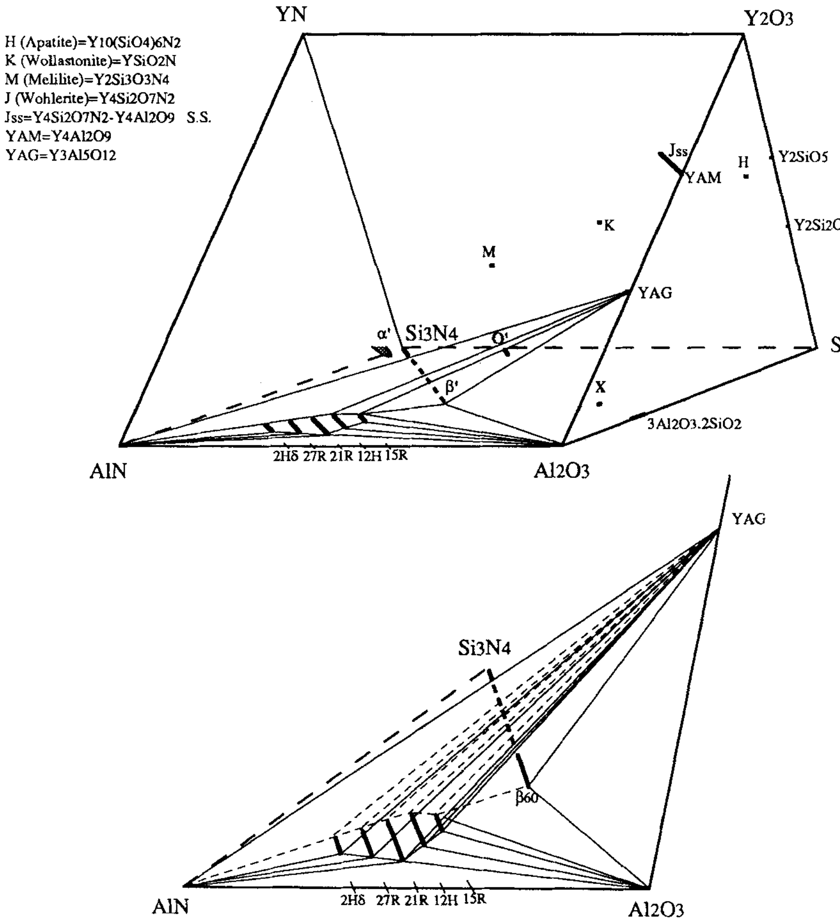

Fig. 2. YAG is compatible with all polytypoid phases, AlN and Al_2O_3 forming twelve compatibility tetrahedra: YAG–β_{60}–15R–Al_2O_3; YAG–15R–Al_2O_3; YAG–15R–12H; YAG–12H–21R; YAG–21R–27R; YAG–27R–2Hδ; YAG–2Hδ–AlN; YAG–2Hδ–AlN–27R; YAG–27R–AlN–21R; YAG–21R–AlN–Al_2O_3; YAG–21R–Al_2O_3–12H, and YAG–12H–Al_2O_3–15R.

The mixtures of Y_2O_3 with excess carbon black (Y_2O_3:C = 4:1 in weight ratio) were reacted in a graphite-resistant furnace under flowing nitrogen at 1900° to 1920°C for 4 h. The furnace was evacuated to 30×10^{-3} torr before heating to the reaction temperature (1000° to 1200°C). The control of oxygen partial pressure is a critical condition for the success of the preparation of YN. YN prepared under the above conditions contained residual carbon (about 9 wt%), a small amount of Y_2O_3, and/or YC_2. High temperatures favored the formation of YC_2. If less carbon (lower than 15 wt%) was used, YC_2 was produced. The reaction could not be completed at temperatures below 1850°C. Freshly prepared YN powder was kept in a desiccator under vacuum where the YN is stable for 2 to 3 weeks with respect to hydrolysis.

III. Results and Discussion

(1) The System Si_3N_4–AlN–Al_2O_3–Y_2O_3

Thirty-three compositions were studied in the region bounded by Si_3N_4–AlN–Al_2O_3–Y_2O_3 to establish the compatibility tetrahedra. The binary tie lines established were based on the results listed in Table I. No new phase was found in the composition region explored. Based on these results and some of the literature data,[6] 39 compatible tetrahedra were established in this part of the system (Table III).

As indicated in this table, tie lines exist between the compound $3Y_2O_3 \cdot 5Al_2O_3$ (YAG) and all of the AlN polytypoids. α′-SiAlON was found to be compatible with β′-SiAlON. Compatible triangles were formed between the Si-rich terminal compositions of AlN polytypoids and α′ and β′-SiAlONs; i.e., all of the compatibility triangles on the Si_3N_4–AlN–Al_2O_3 plane coexisted with either YAG or α′-SiAlON, forming 23 tetrahedra. These results demonstrated that β′-SiAlON coexists with all of the AlN polytypoids (from 15R to 2H[δ]), as seen in Fig. 1. The figure shows the region studied in this part of our work and also gives a recently revised Si-Al-O-N behavior diagram by Slasor.[10] Our work did not determine the exact β′-SiAlON compositions which are in equilibrium with different AlN polytypoids. A very small increase in the cell dimensions of β′-SiAlONs occurs with the increase of the SiO_2 content and was found in all of the β′-SiAlONs which are in equilibrium with the AlN polytypoids. Between the two regions (YAG-containing and α′-containing), there exist 16 compatibility tetrahedra. In this region, melilite ($Si_3N_4 \cdot Y_2O_3$) and *Jss* ($2Y_2O_3 \cdot Al_2O_3$–$2Y_2O_3 \cdot Si_2N_2O$ solid solutions) appeared. Both melilite and *Jss* (close to the intermediate composition) were in equilibrium with the Si-rich points of 21R, 27R, 2H[δ], and AlN. AlN coexisted with the entire single-phase region of *Jss*. YAG–AlN polytypoids and α′-β′ two-phase regions are graphically represented in Figs. 2 to 4. In the present work, α′-SiAlON is considered as a point composition. Detailed phase relationships involving α′-SiAlON are being determined and will be published separately.

(2) The System Si_3N_4–AlN–YN

Thompson reported that three compounds exist in the binary system Si_3N_4–YN.[6] The compositions of these compounds are 6YN:Si_3N_4, 2YN:Si_3N_4, and YN:Si_3N_4. The single-phase nitride containing α′-SiAlON was reported to exist at $m = 1.8$ to $m = 3.4$ ($Y_{m/3}Si_{12-m}Al_mN_{16}$).[6] However, Slasor reported that the single-phase nitride α′-SiAlON occurred at $m = 1.0$.[5] In the present work, only one binary compound, 2YN:Si_3N_4, was confirmed. The X-ray diffraction pattern of this compound is given in Table IV[§] (not published

[§]For Table IV, order ACSD-209 from Data Depository Service, The American Ceramic Society, 757 Brooksedge Plaza Drive, Westerville, OH 43081–6136.

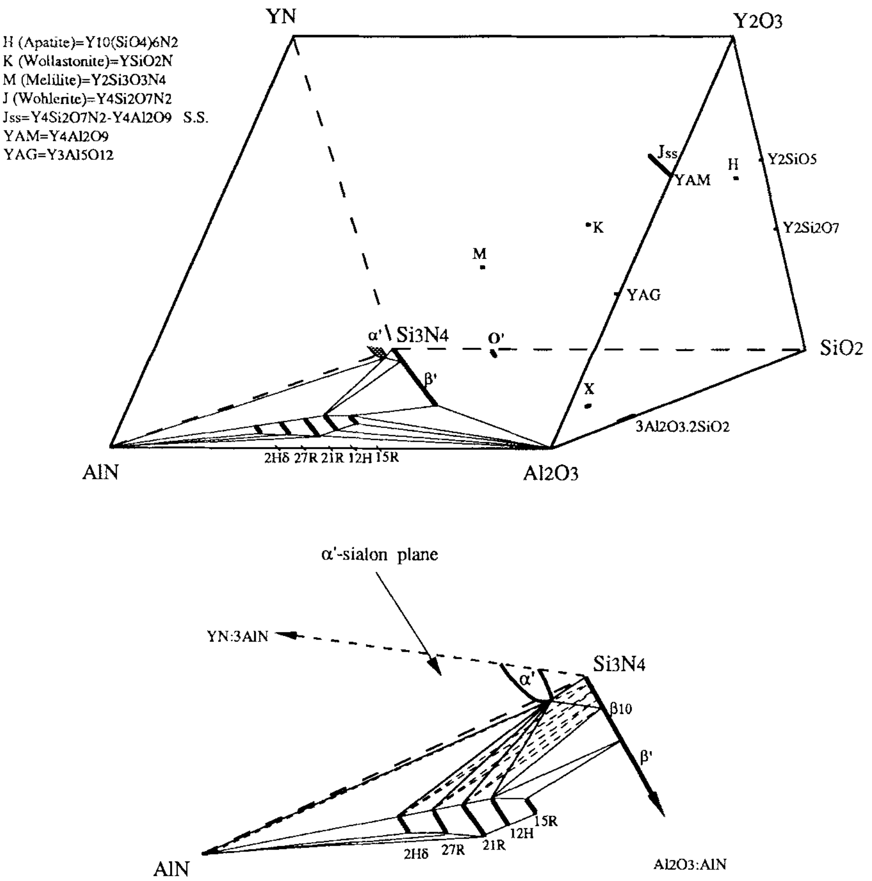

Fig. 3. α′-SiAlON is compatible with polytypoids (from 2Hδ to 12H), AlN and β′ forming eight compatibility tetrahedra: α′–12H–21R–β10; α′–21R–β10–β8; α′–21R–β8–27R; α′–β8–27R–β5; α′–27R–β5–2Hδ; α′–β5–2Hδ–β2; α′–2Hδ–β2–AlN and α′–β2–AlN–Si_3N_4.

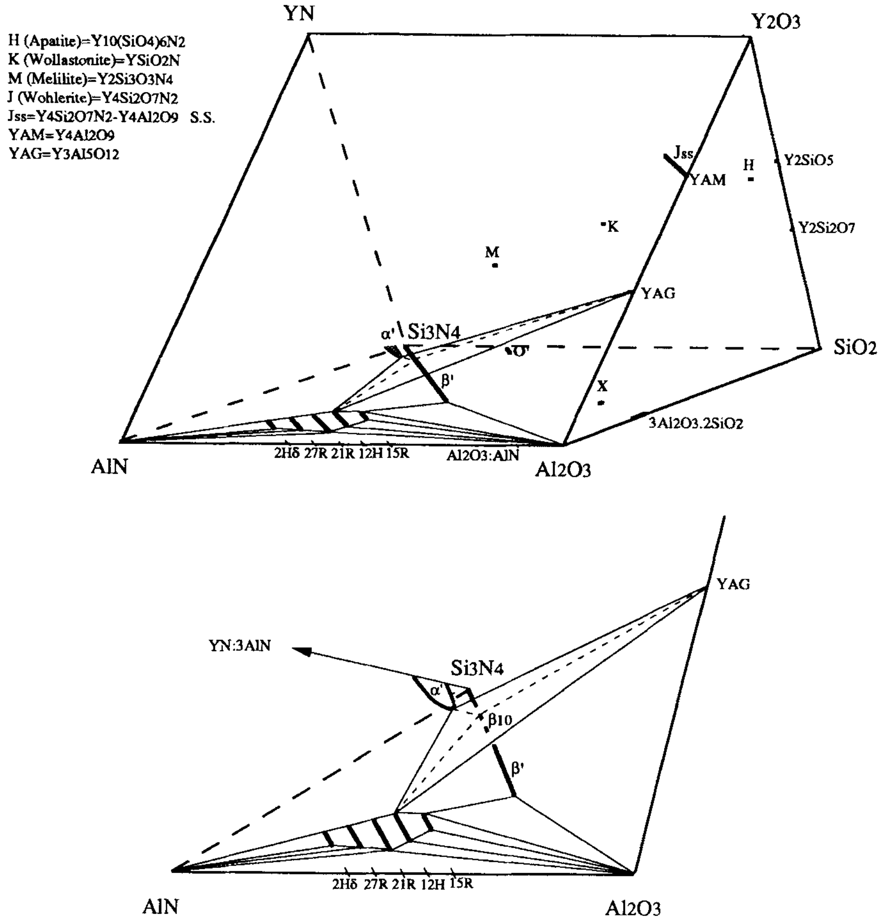

Fig. 4. Compatibility tetrahedron α'–β10–12H–YAG.

with this paper) and agrees with the X-ray pattern reported by Thompson for compound $6YN:Si_3N_4$.[6] The homogeneous range of the α'-SiAlON was determined to extend from $m = 1.3$ to 2.4, with unit cell dimensions of $a = 7.810$ Å, $c = 5.681$ Å, and $a = 7.863$ Å, $c = 5.731$ Å, respectively. The differences in compositions in our work and in Thompson's report probably can be attributed to the purity of the YN powder. YN is very sensitive to moisture in the atmosphere, and extreme precautions should be taken during the experiment. The X-ray diffraction lines of $2YN:Si_3N_4$ and $YN:Si_3N_4$ reported by Thompson were probably a mixture of melilite ($Y_2Si_3O_3N_4$), J phase ($2Y_2O_3:Si_2N_2O$), and other oxygen-containing phases. The oxidation of mixtures YN and Si_3N_4 will give melilite, J phase, and even Y_2O_3, as indicated in Table II.

(3) *The System Si_3N_4–AlN–YN–Y_2O_3*

Y_3O_3N has been reported to be a single-phase composition existing in the binary system Y_2O_3–YN.[6] Compound Y_3O_3N has been obtained in our laboratory, but it was found to be difficult to complete the reaction forming Y_3O_3N. All of the compositions in the compatibility tetrahedra YN–Y_3O_3N–

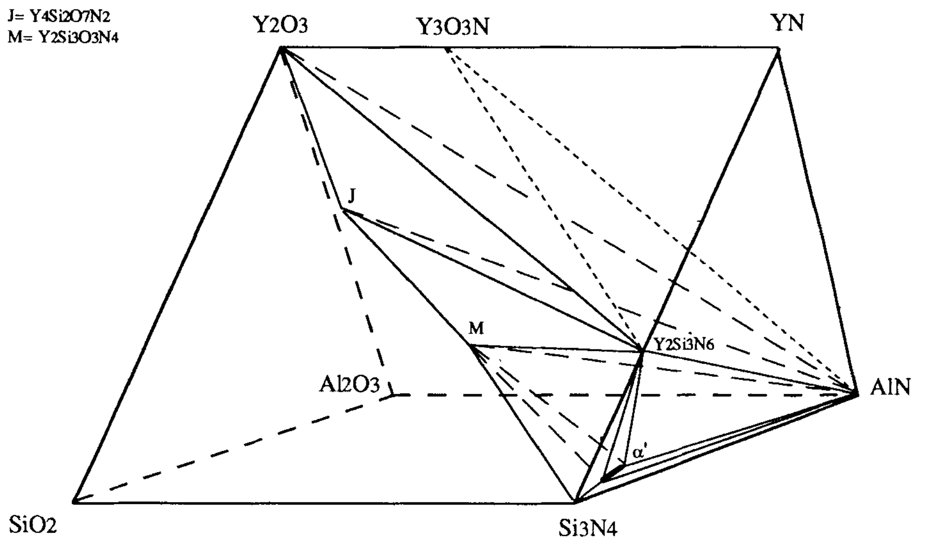

Fig. 5. Subsolidus phase relationships in the region bounded by Si_3N_4, AlN, YN, and Y_2O_3.

$Y_2Si_3N_6$–AlN and Y_3O_3N–$Y_2Si_3N_6$–AlN–Y_2O_3 contained small amounts of Y_2O_3, YN, and Y_3O_3N. Therefore, the tie lines Y_3O_3N–$Y_2Si_3N_6$ and Y_3O_3N–AlN are represented by dashed lines in Fig. 5. It is also possible that compound Y_3O_3N has a lower temperature stability limit. No quinary compound was observed in this region. $Y_2Si_3N_6$ is an important compound which coexisted with all phases occurring in the system. Seven compatibility tetrahedra are formed in this region:

AlN—YN—Y_3O_3N—$Y_2Si_3N_6$
AlN—Y_3O_3N—$Y_2Si_3N_6$—Y_2O_3
AlN—Y_2O_3—J phase—$Y_2Si_3N_6$
AlN—J phase—melilite—$Y_2Si_3N_6$
AlN—melilite—$Y_2Si_3N_6$—$\alpha'(m = 2.4)$
Melilite—$Y_2Si_3N_6$—$\alpha'(m = 1.3$ to $2.4)$
Melilite—$Y_2Si_3N_6$—$\alpha'(m = 1.3)$—β-Si_3N_4

$Y_2Si_3N_6$ was formed at relatively low temperatures (~1700°C). Like YN, it is also very sensitive to moisture. All bulk samples fired containing YN or $Y_2Si_3N_6$ became powder after aging in the air overnight or after a few days.

IV. Summary

The subsolidus phase relationships in the system Si,Al,Y/N,O were determined. Thirty-nine compatibility tetrahedra had been established in the region Si_3N_4–AlN–Al_2O_3–Y_2O_3. The subsolidus phase relationships in the region Si_3N_4–AlN–YN–Y_2O_3 were also studied. Freshly prepared YN powder was used as the starting material. Only one compound, 2YN:Si_3N_4, was confirmed in the binary system Si_3N_4–YN. The solubility limits of the α'-SiAlON on the Si_3N_4–YN:3AlN join were determined to range from $m = 1.3$ to $m = 2.4$ in the formula $Y_{m/3}Si_{12-m}Al_mN_{16}$. No quinary compound was found. Seven compatibility tetrahedra were established in the region Si_3N_4–AlN–YN–Y_2O_3.

Sixty-eight compatibility tetrahedra were established in the system Si,Al,Y/N,O: thirty-nine in the region bounded by Si_3N_4–SiO_2–AlN–Al_2O_3–Y_2O_3, seven in the region bounded by Si_3N_4–AlN–YN–Y_2O_3, and twenty-two (previously reported[3]) in region Si_3N_4–β_{60}–Al_2O_3–SiO_2 and Y_2O_3.

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