

CHAPTER 3

EXPERIMENTAL PROCEDURE

3.1 Sample preparation

3.1.1 Process flow chart and experimental conditions

The process flow chart is shown in Fig 3.1. Bulk density, mass of specimen and α/β phase of Si_3N_4 in packing powder and specimen were measured for all specimens. Vickers hardness, fracture toughness, bending strength and microstructure were measured and observed for selected specimens. Raw powders were mixed with ethanol by ball milling using Si_3N_4 pot (500 ml) and Si_3N_4 ball (diameter 5 mm) for 192 h. The mixture slurry was dried at temperature around 80°C . The dried powder was passed through 100 mesh. The sieved powders were formed into disc-shaped specimen of 25 mm in diameter and 5 mm in thickness.

Initially, the mixed powders without binder are compacted by a uniaxial press at 20 MPa. Some specimens were subsequently cold isostatic pressed (CIP) to obtain higher density compacts with pressure of 200 MPa. The specimens were set in an Al_2O_3 crucible with Si_3N_4 and Al_2O_3 packing powder. Sintering was performed in the air atmosphere furnace at various temperatures in the range of 1550 to 1700°C . The heating rate was $5\text{-}10^\circ\text{C}/\text{min}$ and holding time was 1-2 h. All sintering conditions are shown in Table 3.1 and Table 3.2.

Bulk density and water absorption were measured by Archimedes' method.³²⁾ Crystalline phases were identified using X-ray diffraction (XRD, D8-Advance, Bruker, Ltd.).³³⁾ The cross-section morphology of fracture surface was observed by using scanning electron microscope, SEM. (JSM-5410LV, JEOL Co., Ltd.)

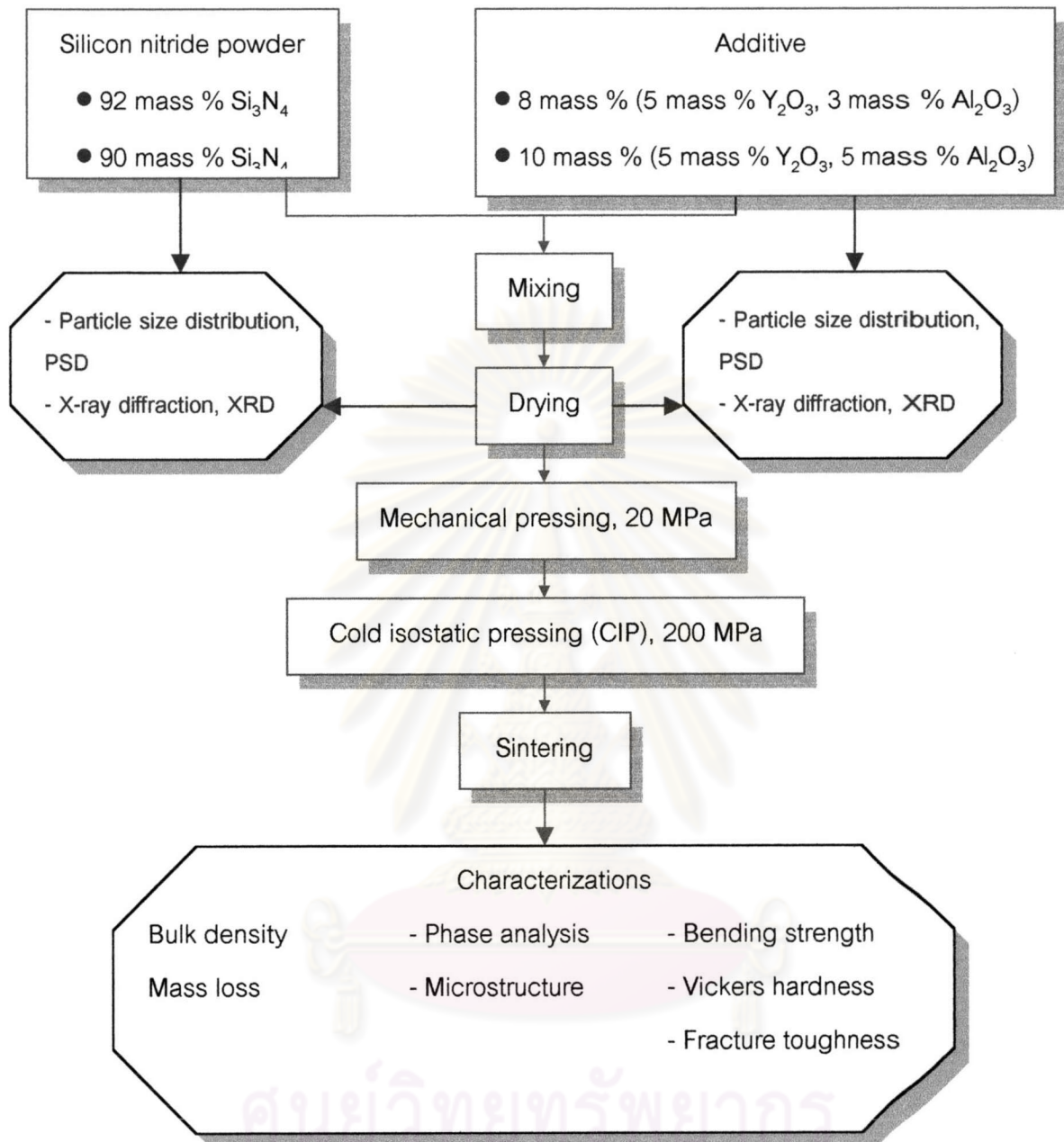


Fig. 3.1 Experimental flow chart of specimen preparation.

Table 3.1 Sintering conditions of mixed powder A

Experimental Conditions	Temperature (°C)	Packing powder Conditions	Heating rate (°C/min)	Cooling rate (°C/min)	Soaking time (h)
C1	1550	SN-7, AM-21	5	5	2
C2	1550	SN-E10,AM-21	5	5	2
C3	1550	[SN-7+BN], A-11	5	5	2
C4	1550	SN-E10, A-11	5	5	2
C5	1600	[SN-7+BN], A-11	5	5	2
C6	1600	SN-E10, A-11	5	5	2
C7	1600	[SN-7+BN], A-11	10	Natural cooling	2
C8	1600	SN-E10, A-11	10	Natural cooling	2
C9	1650	[SN-7+BN], A-11	10	Natural cooling	2
C10	1650	SN-E10, A-11	10	Natural cooling	2
C11	1700	[SN-7+BN], A-11	10	Natural cooling	1
C12	1700	SN-E10, A-11	10	Natural cooling	1
C13	1700	SN-E10, A-11	10	Natural cooling	2

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Table 3.2 Sintering conditions of mixed powder B

Experimental Conditions	Temperature (°C)	Mix powder Conditions	Packing Powder	Heating rate (°C/min)	Cooling rate (°C/min)	Soaking time (h)
E1	1650	Lot 1	SN-KO5 A-11	10	Natural cooling	2
E2	1650	Lot 1	SN-F2 A-11	10	Natural cooling	2
E3	1700	Lot 1	SN-KO5 A-11	10	Natural cooling	1
E4	1700	Lot 2	SN-KO5 A-11	10	Natural cooling	1
E5	1700	Lot 1	SN-F2 A-11	10	Natural cooling	1
E6	1700	Lot 2	SN-F2 A-11	10	Natural cooling	1
E7	1700	Lot 1	SN-KO5 A-11	10	Natural cooling	2
E8	1700	Lot 2	SN-KO5 A-11	10	Natural cooling	2
E9	1700	Lot 1	SN-F2 A-11	10	Natural cooling	2
E10	1700	Lot 2	SN-F2 A-11	10	Natural cooling	2

3.1.2 Crucible structure

Al_2O_3 is a candidate material for crucible as described in previous paper.²²⁾ The crucible structure is shown in Fig 3.2 (a) for Mixed powder A and (b) for Mixed powder B. The specimens are set in a small Al_2O_3 crucible (Nikkato SSA-S, 50 cm^3) with Si_3N_4 packing powder. Then the small crucible is set again in a larger crucible (280 cm^3) filled with Al_2O_3 packing powder.

Several kinds of Si_3N_4 powders were used as packing powder, which are shown in Table 3.3. Al_2O_3 packing powders are also shown in Table 3.4. More details for these powders are shown in Appendices Table A-1, A-2 and A-3.

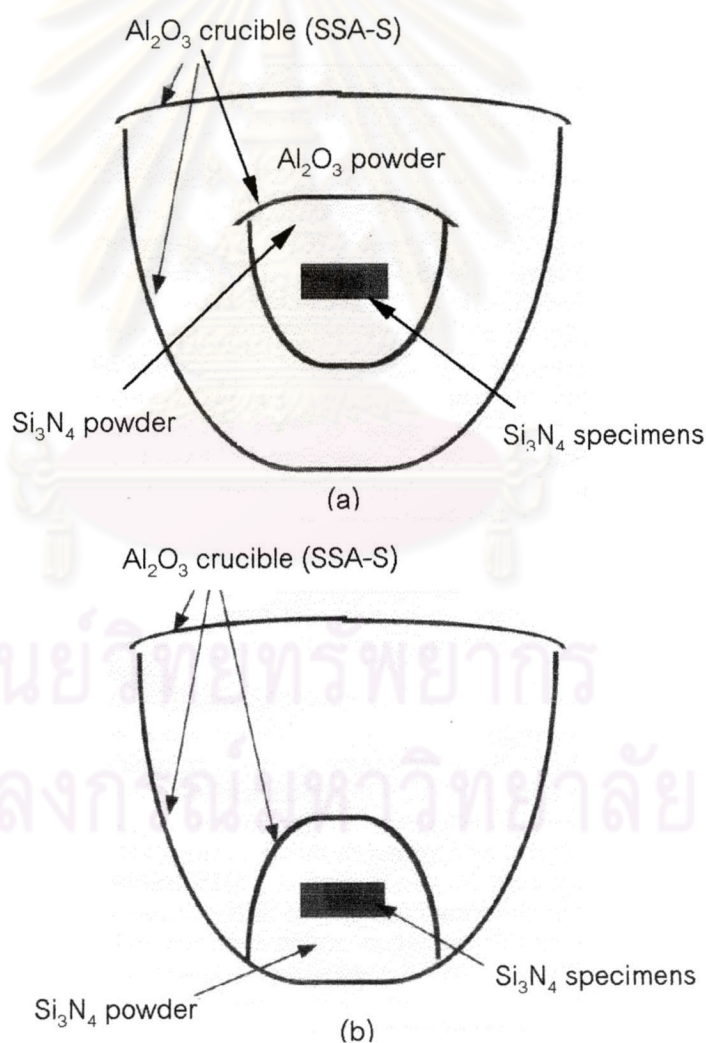


Fig 3.2 Schematic diagram of the crucible structure

Table 3.3 Name and properties of Si₃N₄ packing powder

Grade	Chemical Composition (%)	Specific surface area* (m ² / g)	Mean Particle sizes** (μm)	Tap density*** (g / cm ³)
E-10 ¹⁾	α % > 95	10.45	0.83	0.63
SN-7 ²⁾	α % = 74.0	3.79	3.40	1.16
SN-KO5 ¹⁾	α % = 81.6	5.03	1.68	0.68
SN-F2 ²⁾	α % < 1	0.89	2.22	1.61
- Fine			21.71	1.39
- Coarse				

- 1) = Ube Industries Ltd.
 2) = Denki Kagaku Kogyo Co., Ltd.
 * = BET analysis method
 ** = Sedimentation method
 *** = Tap-Pak Volumeter method

Table 3.4 Name and properties of Al₂O₃ and BN packing powder

Grade	Chemical Composition (%)	Specific surface area* (m ² / g)	Mean Particle sizes** (μm)	Tap density*** (g / cm ³)
AM-21 ¹⁾	Al ₂ O ₃ = 99.7	1.27	4.0	1.67
A-11 ²⁾	Al ₂ O ₃ = 99.7	0.79	9.0	0.96
h-BN ³⁾	BN = 99.9	-	-	-

- 1) = Sumitomo Chemicals
 2) = Fuji Kasei Co., Ltd.
 3) = Denki Kagaku Kogyo Co., Ltd.
 * = BET analysis method
 ** = Sedimentation method
 *** = Tap-Pak Volumeter method

3.1.3 Raw powder and preparation of powder mixing

The commercial Si_3N_4 powder (E-10, Ube industries, Ltd.) was used as the raw Si_3N_4 powder, because it was known as one of the best powders.¹⁸⁻²²⁾ The powder has high purity. The alpha-phase content is 95 %. Major impurities are aluminum, calcium and iron (< 100 ppm), and oxygen content is less than 2 Wt %. Two kinds of sintering aids: Y_2O_3 (RU, Shin-Etsu Chemical Co., Ltd.), and Al_2O_3 (AKP-30, Sumitomo Co., Ltd.) were mixed with Si_3N_4 powder. The properties of E-10, RU and AKP-30 are shown in Table A-1, A-4 and A-5 in appendix.

Table 3.5 shows the two compositions of Si_3N_4 specimen bodies with different amounts of sintering aids: (A) 5.0 mass % Y_2O_3 and 3.0 mass % Al_2O_3 , (B) 5.0 mass % Y_2O_3 and 5.0 mass % Al_2O_3 . Both compositions were mixed by ball milling using Si_3N_4 pot and Si_3N_4 ball with 150 cm³ of ethanol and 40 g of mixed powder for 192 h, and the rotation speed was 150 rpm. Then, the mixtures were dried in the oven at 80 °C and passed through a 100-mesh nylon sieve. The milling conditions of mixed powder are shown in Table 3.6. (Powder A was prepared in Japan with 24 h milling time by Prof. WADA²²⁾, powder B was prepared at Chulalongkorn University.)

Table 3.5 Experimental conditions of mixed powder A and B

Compositions (mass %)	Mixed powder A	Mixed powder B
Si ₃ N ₄	92	90
Y ₂ O ₃	5	5
Al ₂ O ₃	3	5

Table 3.6 Milling conditions of mixed powder B

Mill and ball	Si ₃ N ₄ pot, 500 cm ³ , Si ₃ N ₄ , ϕ 5 mm, 250 cm ³ (Half a volume of pot)
Medium	150 cm ³ of ethanol
Powder	Si ₃ N ₄ with Y ₂ O ₃ -Al ₂ O ₃ additive (40 g of the mixed powder) ³⁴⁾
Rotation speed	150 rpm ³⁴⁾
Time	192 h

3.2 Temperature inside crucible

Specimen was set in double crucibles and 2 kinds of packing powders. Generally the thermal conductivity of powder is very low. Therefore, it was thought that the temperature of specimen would lag behind that in the furnace. Then the temperature inside Si₃N₄ packing powder, which was equal to the temperature of specimen, was compared with that in the furnace. Commercially available R-Type thermocouples (87% Platinum, 13% Rhodium (Pt & Pt-Rh)), which is noble-metal thermocouples that can be used up to high temperature as 1750 °C, were used for temperature measurement.

Two thermocouples were used. One was inserted from top of the furnace to the inside the small Al₂O₃ crucible, which was covered with Aluminum packing powder A-11 (no specimens). Another thermocouple was inserted from backside of furnace to measure the temperature inside furnace.

3.3 Properties measurement and characterization

3.3.1 Particle size distribution of raw materials and mixed powders

For these experiments, two methods were used; sedimentation method and sieve analysis method.

Sedimentation method

Sedimentation method (Shimadzu SA-CP2) was used for investigating particle size distribution of raw materials; powder A, B, SN-E10, SN-7, SN-KO5, and SN-F2.

Approximately 0.5 gram of the powder was dispersed in 50 cm³ water, which was 0.2 mass % aqueous solution of sodium hexametaphosphate (71600 NaHMP, Fluka chemic ltd.). Sample particles were dispersed completely using magnetic stirrer, at least for 30 min. Furthermore, ultrasonic bath was used to enhance powder deagglomeration. Gravity or centrifugal mode was selected for powder diameter bigger than 10 μm or smaller than 8 μm , respectively. Sedimentation depth No.3 and 1000 rpm of revolution were used as the condition for analysis. The average particle size diameters for all samples were calculated at 50% cumulative.

Sieve analysis method

100 gram of SN-F2 was sieved in a series of sieves No. 50, 100, 140, 200, and 325 meshes, which was equivalent to particle diameters +300, +150, +106, +75, and +45 μm , respectively. Average particle size of SN-F2 was calculated from the oversize powder weight that remains on each sieve.

3.3.2 Oxygen analysis in mixed powder and estimation of mixed powder composition.

Oxygen contents of powder A, powder B and SN-F2 were measured by Nitrogen/Oxygen determinater at the SIAM Research and Development Co., LTD.

Oxygen exists as either SiO_2 or $\text{Si}_2\text{N}_2\text{O}$ in the surface layer of Si_3N_4 powders. The sintering additives Y_2O_3 and Al_2O_3 create a liquid phase at high temperatures by reacting with the SiO_2 , which presents in Si_3N_4 powder. Thus control of the oxygen content in the powder is crucial for controlling the densification. In addition, the amount of oxygen increases during the ball-milling.³⁴⁾

In this experiment, the contents of oxygen before and after milling of mixed powder are analyzed. The analyzed amount of oxygen in mixed powder includes oxygen from Y_2O_3 and Al_2O_3 . Therefore, the oxygen which belongs to Y_2O_3 and Al_2O_3 is subtracted from the analyzed amount. Then oxygen which belongs to SiO_2 is determined.

a) For the composition, 92 mass % Si_3N_4 , 5 mass % Y_2O_3 and 3 mass % Al_2O_3 and Si_3N_4 -E10 are including 1 % of oxygen.

$$\text{O}_2 (\text{mass } \%) = \text{Si}_3\text{N}_4 \text{ mass } \% * (1/100) = 92 * 0.01 = 0.92$$

From 0.92 mass % of O_2 , mass % of SiO_2 in Si_3N_4 powder is calculated.

$$\text{SiO}_2 (\text{mass } \%) = \frac{60.08}{(2 \times 15.99)} \times 0.92 = 1.73$$

b) Mass % of oxygen in oxide M_aO_b can be calculated as follows:

$$\text{Mass \% O (} M_aO_b \text{)} = \left(100 \times \frac{b \times MW_O}{MW_{M_aO_b}} \right) \times [\text{mass \% (Oxide)}] \quad \text{---(1)}$$

Where:

- a = Mole of metal
 b = Mole of oxygen
 MW_O = Molecular weight of oxygen
 $MW_{M_aO_b}$ = Molecular weight of oxide

From equation (1):

$$\text{Mass \% Oxygen of (5 mass \% } Y_2O_3 \text{)} = \left(100 \times \frac{3 \times 16}{225.81} \right) \times 5 = 1.06$$

$$\text{Mass \% Oxygen of (3 mass \% } Al_2O_3 \text{)} = \left(100 \times \frac{3 \times 16}{101.96} \right) \times 3 = 1.41$$

c) The theoretical density of glassy phase is calculated from SiO_2 , Y_2O_3 , and Al_2O_3 composition because the glassy phase is made from them.³⁵⁾ In this case, using theoretical density value 4.84 g/cm^3 , 4 g/cm^3 and 2.2 g/cm^3 for Y_2O_3 , Al_2O_3 and SiO_2 , respectively.^{2, 36, 37)}

The density of glass could be estimated as follows:

$$\text{Glass density} = \sum (\text{Weight fraction of oxide} \times \text{density of oxide})$$

$$\text{Glass density} = \left[\left(\frac{1.73}{9.73} \right) \times 2.2 \right] + \left[\left(\frac{5}{9.73} \right) \times 4.84 \right] + \left[\left(\frac{3}{9.73} \right) \times 4 \right]$$

$$\text{Glass density} = 0.39 + 2.49 + 1.23 = 4.11$$

d) The theoretical density of mixed powder is the sum of theoretical density of Si_3N_4 and glassy phase. The theoretical density of mixed powder A before milling (Si_3N_4 , Y_2O_3 , $\text{Al}_2\text{O}_3 = 92, 5, 3$ (mass %)) is calculated as 3.30 g/cm^3

Powder A Compositions	Real Composition (%)	Theoretical Density (g/cm^3)	Density (g/cm^3)
Si_3N_4	90.27	3.21	2.90
Glass	9.73	4.11	0.40
Total	100	-	3.30

All calculated results for mixed powder A and B, before and after mixing, are shown in the following Table:

Table 3.7 Calculated theoretical density of mixed powder A and B, before and after milling

Powder		Analyzed SiO_2 content (mass %)	Analyzed Oxygen content (mass %)	Calculated theoretical density (g/cm^3)
A	Before	1.73	3.39*	3.30
	After	3.66	4.42	3.28
B	Before	1.69	4.31*	3.31
	After	6.90	7.06	3.26

* Estimated values from calculations.

3.3.3 Outlook observation of specimens and packing powders

As shown in the previous works, Si_3N_4 ceramics with high density was obtained by sintering with packing powder.¹⁸⁻²²⁾ However, the kind of packing powders used and the easiness of taking off specimens from the packing powder were not discussed in detail.

In this experiment, it was found that the packing powder tended to agglomerate after sintering. The agglomeration made the releasing of specimens from packing powder difficult. Therefore, one of the major aims of this experiment is to find a good packing powder, which does not agglomerate. Seven types of packing powder, five types of Si_3N_4 and two types of Al_2O_3 were used. The five types of Si_3N_4 packing powder are SN-E10, SN-7, SN-7 plus 10 Wt % of BN, SN-KO5, and SN-F2. In case of SN-F2, two conditions, no sieved and sieved were used. Besides, two types of Al_2O_3 packing powders, AM-21 and A-11, were also investigated.

3.3.4 Mass change measurement

The mass of specimen changed when reactions (1) – (4) described in 2.4 occur during sintering. Thus, mass change of Si_3N_4 as a function of sintering temperature was measured. The mass loss of each specimen was calculated from the weight measurement of specimens before sintering W_B and after sintering W_A (g). The mass loss is calculated as follows:

$$\text{Mass change (\%)} = \left(\frac{W_B - W_A}{W_A} \right) \times 100$$

3.3.5 Bulk density and relative density

Archimedes' method

1. Dried test specimens to constant weight by heating to 105 to 110 °C. Determine the dry weight (D) in grams.
2. Place the test specimens in a suitable vacuum-pressure vessel. Close, secure, and pump down, hold this for 30 min. Pour water into the vessel, maintaining the vacuum for 5 min. Then close the vacuum. The saturated specimens are now ready for weighing.
3. Determination of the suspended weight (S) and the saturated weight (W). Used this value for calculations.

Calculation:³²⁾

1. Volume, V (cm³)

$$V = \frac{(W - S)}{\rho_w} \quad \text{-----}(1)$$

Where, ρ_w is the density of water.

2. Volume of open pore, O (cm³)

$$O = \frac{(W - D)}{\rho_w} \quad \text{-----}(2)$$

3. Water absorption, Aw (%)

$$Aw (\%) = \frac{(2)}{D} \times 100$$

4. Bulk density, B (g/cm³)

$$B = \frac{D}{(1)}$$

3.3.6 Identification of material and determination of phase content in Si_3N_4 by X-ray diffraction analysis

The $\alpha \rightarrow \beta$ Si_3N_4 phase transformation was important for the final microstructure.^{8, 29)} Starting raw materials (powder A, powder B) and packing powders (SN-E10, SN-7, SN-KO5, SN-F2) were measured by X-ray diffraction (D8-Advance, Bruker Co., Ltd.). The 2θ around 15-50 deg, and scanning speed 2 °C deg/min were used as conditions. The weight fraction of α/β content in the specimens were calculated from the XRD peaks of $\alpha\text{-Si}_3\text{N}_4$ and $\beta\text{-Si}_3\text{N}_4$ phases by the method proposed by Gazzara and Messier.³³⁾ In addition, identification of unknown phases formed upon heat treatment was also performed by XRD. The specimens analyzed by X-ray diffraction analysis are shown in Table 3.8 to Table 3.11.

Sample preparation

Sample powders approximately 1-2 gram, which passed through sieve number 230 mesh (70 μm) was used.

Calculation method

The calculation procedure of α/β content proposed by Gazzara and Messier is as follows:

1. Intensity of peaks, 7 peaks for $\alpha\text{-Si}_3\text{N}_4$ and 4 peaks for $\beta\text{-Si}_3\text{N}_4$, is measured.
2. These intensities divided by L value for standardization as shown in the following table.
3. Sum up Y_j , and get average.
4. The ratio of average Y_j for α and β is the weight ratio of α and β in the specimen.

For example, α/β ratio of SN-7 raw material can be calculated as follows:

Phase	Peak	2θ (CuK α)	Intensity (%)	L^*	Y_j
α -Si ₃ N ₄	(101)	20.68	100.00	7.50	13.33
	(110)	23.23	41.90	3.58	11.70
	(200)	26.64	36.90	2.44	15.12
	(201)	31.10	93.50	7.44	12.57
	(102)	34.69	70.60	6.69	10.55
	(210)	35.45	89.20	6.76	13.14
	(301)	43.60	36.90	3.13	11.79
Average					12.60
β -Si ₃ N ₄	(110)	23.49	14.30	4.21	3.40
	(200)	27.16	41.60	10.53	3.95
	(101)	33.78	42.70	10.90	3.92
	(201)	36.19	26.50	11.21	2.36
Average					3.41

* Reference (36)

$$I_{\alpha/(\alpha+\beta)} = \frac{12.60}{12.60 + 3.41} = 0.7870$$

$$I_{\beta/(\alpha+\beta)} = \frac{3.41}{12.60 + 3.41} = 0.2130$$

Thus:

$$\alpha \text{ (mass \%)} = 0.7870 \times 100 = 78.70$$

$$\beta \text{ (mass \%)} = 0.2130 \times 100 = 21.30$$

The calculation results of Y_j , α (Wt %), and β (mass %) are shown in Table C-1 and C-2 of appendix C.

Table 3.8 XRD conditions of SN-E10 and SN-7 packing powder materials

Conditions	SN-E10				SN-7				SN-7+BN			
	No Separate	Glassy Phase	Top	Bottom	No Separate	Glassy Phase	Top	Bottom	No Separate	Glassy Phase	Top	Bottom
1600 °C 5 °C / min	○				○					○	○	
1600 °C 10 °C / min										○	○	
1650 °C 10 °C / min			○	○						○	○	○
1700 °C 10 °C / min			○	○						○	○	
1700 °C 10 °C / min	○											

Table 3.9 XRD conditions of SN-F2 and SN-KO5 packing powder materials

Conditions	SN-F2		SN-KO5	
	Top [*]	Bottom ^{**}	Top [*]	Bottom ^{**}
1650 °C, 2 h	○	○	○	○
1700 °C, 1 h	○	○	○	○
1700 °C, 2 h	○	○	○	○

Note: All sample sintered with heating rate 10 °C / min.

^{*} = Top part of packing powders

^{**} = Bottom part of packing powders

Table 3.10 XRD conditions of mixed powder A Specimens

Conditions	Packing powders		Packing powders	
	SN-E10	SN-7	SN-E10	SN-7+BN
1550 °C, 2 h 5 °C / min	○	○	○	○
1600 °C, 2 h 5 °C / min			○	○
1600 °C, 2 h 10 °C / min			○	○
1650 °C, 2 h 10 °C / min			○	○
1700 °C, 1 h 10 °C / min			○	○
1700 °C, 2 h 10 °C / min			○	

Table 3.11 XRD conditions of mixed powder B specimens

Conditions	Sintering with SN-KO5		Sintering with SN-F2	
	Lot 1	Lot 2	Lot 1	Lot 2
1650 °C, 2 h	○		○	○
1700 °C, 1 h	○	○	○	○
1650 °C, 2 h	○	○	○	○

Note: All sample sintered with heating rate 10 °C / min.

3.3.7 Observation of microstructure by scanning electron microscope (SEM)

The microstructure of fractured surface and polished surface were characterized by scanning electron microscope (SEM), (JSM-5410L, JEOL Co., Ltd.). Sintered Si_3N_4 specimens of powder B at 1700°C for 2 h with two types of packing powder, SN-KO5 or SN-F2, were selected as samples. The procedure of sample preparation is shown in Fig 3.3.

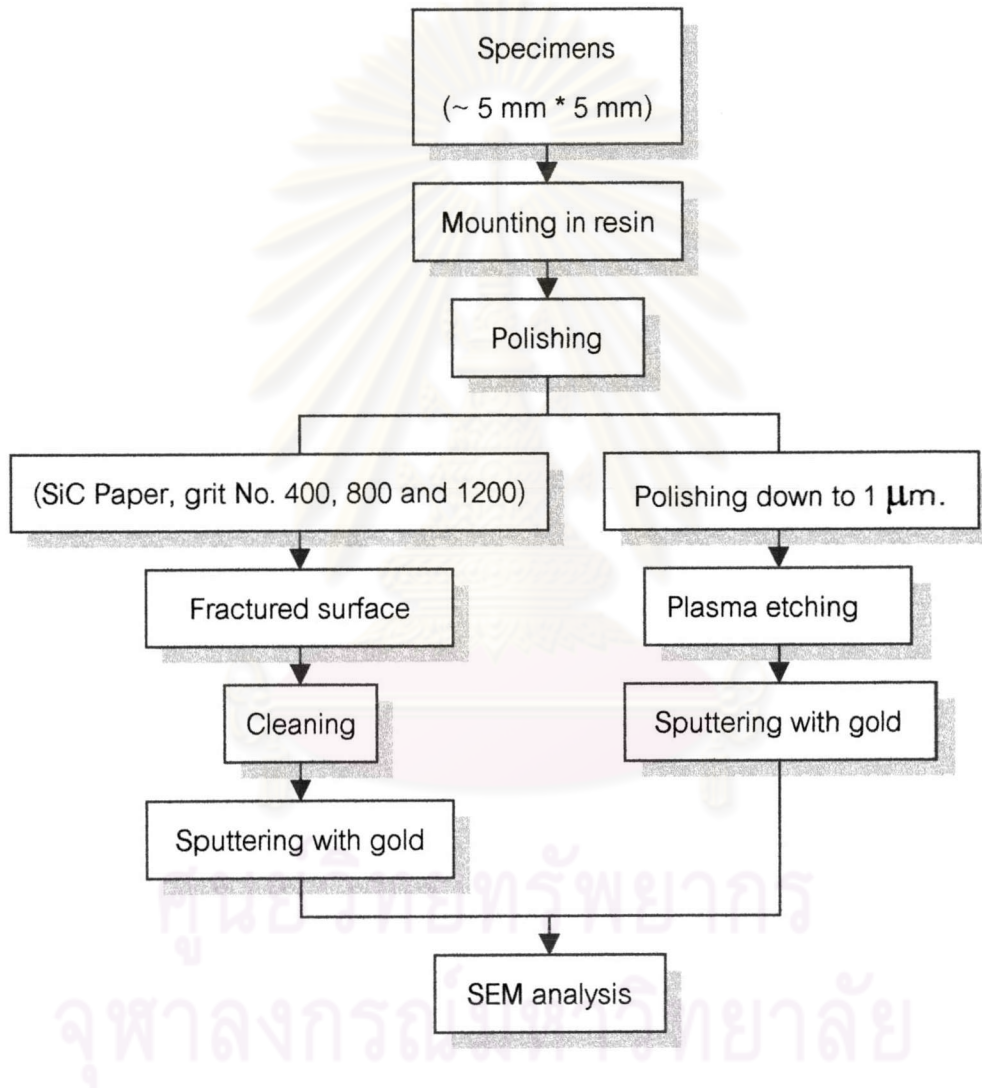


Fig 3.3 Sample preparation flow chart for SEM analysis

3.3.8 Vickers hardness and fracture toughness

Sample preparation:

The fracture toughness and the hardness of the sintered body were measured with a load of 98 N by Vickers indentation (Zwick 3212, Zwick GmbH & Co.) and calculated by JIS-R1607³⁸⁾ and JIS-R1610³⁹⁾, respectively. For this experiment, specimens sintered with SN-F2 packing powder at 1700 °C for 2 h and heating rate 10 °C/min were selected as examples.

Finish the surfaces of other test specimens by grinding with 170, 200 and 400 grit of diamond wheel, respectively. Then, polish with 800 and 1,200 grit SiC paper (Buehler grinding paper) down to 15.30 μm. The surface was examined at intervals using optical microscope (BX60MF5, OLYMPUS OPTICAL Co., Ltd., Japan). After polishing (Using PHOENIX 4000, Buehler Co., Ltd.), specimens were washed with alcohol under the ultrasonic to remove dirt and debris from the surfaces. Vickers indentation was applied at a load of 98.07 N (10 kg) JIS standard (Designation R 1610-1991) using microhardness tester.(Zwick 3212, Zwick GmbH & Co.)

Calculation:

Vickers hardness, (HV) is calculate by the following equation:

$$HV = 1.8554 \times \left(\frac{P}{(2a)^2} \right)$$

Where: P = Load (Kg)

a = Diameter (mm)

Fracture toughness, (K_{1C}) is calculate by the following equation in conformity with JIS R1607¹⁹⁹⁵

$$K_{1C} = 0.026 \left(\frac{E^{\frac{1}{2}} \times P^{\frac{1}{2}} \times a}{c^{\frac{3}{2}}} \right)$$

Where: c = Crack length (mm)

E = Young' modulus ~ 280 GPa

Table 3.12 Specimens for SEM, Vickers hardness (VH) and fracture toughness (K_{1C}) test.

Measurement's Method	Conditions			
	SN-KO5		SN-F2	
	1 h	2 h	1 h	2 h
SEM	○	○	○	○
VH			○	○
K_{1C}			○	○

Note: All specimens were sintered at 1700 °C, 10 °C / min.



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3.3.9 Flexural strength test by biaxial bending test at room temperature

Flexural strength was measured by a biaxial-bending test in conformity with ASTM-F394-78⁴⁰⁾

Sample preparation:

Specimens for strength tests were sintered at 1700 °C for 2 h with SN-F2 packing powder. The specimens were ground to flat surface by # 170 diamond wheel. Then, their tensile surfaces were ground with 200 and 400 grit diamond wheel and followed by polishing with 800 and 1200 grit SiC paper (Buehler grinding paper), respectively. All tests were performed using LLOYD 500, Intro enterprise Co., Ltd. The crosshead speed was constant at 0.5 mm/min.

Calculation:

Flexural strength is calculated by the following equations.

$$S = \frac{-0.2387P \times (X - Y)}{d^2} \quad \text{-----}(1)$$

$$X = \left\{ (1 - \nu) \times \ln \left(\frac{B}{C} \right)^2 \right\} + \left\{ \frac{(1 - \nu)}{2} \times \left(\frac{B}{C} \right)^2 \right\} \quad \text{-----}(2)$$

$$Y = \left\{ (1 - \nu) \times \left(1 + \ln \left(\frac{A}{C} \right)^2 \right) \right\} + \left\{ (1 - \nu) \times \left(\frac{A}{C} \right)^2 \right\} \quad \text{-----}(3)$$

Where:

S = Maximum center tensile stress (MPa)

P = Total load causing fracture (N)

μ = Poisson's ratio, 0.23

A = Radius of support circle (12.5 mm)

B = Radius of loaded area or ram tip (2.5 mm)

C = Radius of specimen (13.5 mm)

d = specimen thickness at fracture origin (1.5 mm)

The relation between X and B, when C = 13.5, is shown in Fig 3.4.

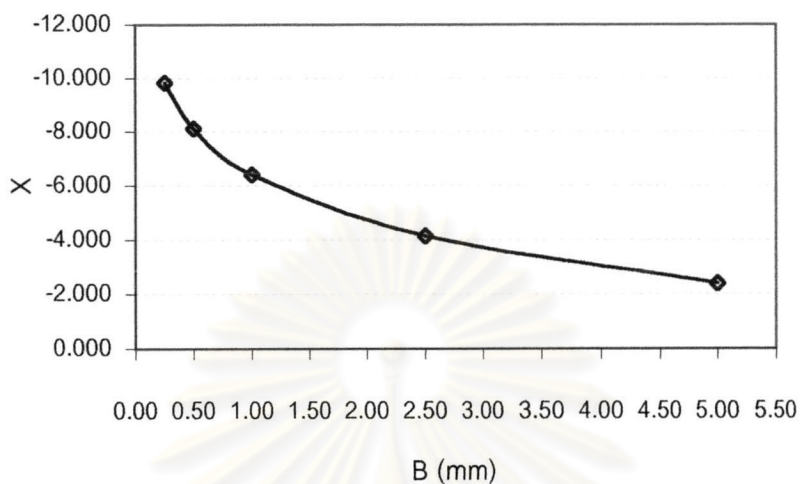


Fig 3.4 Relationship between X and B value for 3-point bending strength test

Miss. Bongoch⁴¹⁾ used the Si_3N_4 ball of 2.5 mm. radius as the ram tip for her master thesis. The contact radius of the ball was 0.125 mm. From Fig 3.4, it is understood that X value changes much when contact radius B changes a little at the range of $B < 0.5$ mm. The ram tip of 1.5 mm. radius is thought to be a little weak to the applied load. Considering these facts, the radius of ram tip of 2.5 mm was selected.

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