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## Development of silicon nitride ceramic for CAD/CAM restoration

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White Silicon nitride ( $\text{Si}_3\text{N}_4$ ) ceramic has unique characteristics. Because of its high fracture toughness, strength, and biocompatibility, it can therefore be used to fabricate dental restorations. The purpose of this study was to produce partially-sintered block of  $\text{Si}_3\text{N}_4$  for fabrication of CAD/CAM dental restorations. The related properties of this novel  $\text{Si}_3\text{N}_4$  were evaluated including sintered shrinkage, flexural strength and fracture toughness. Partially sintered  $\text{Si}_3\text{N}_4$  ceramic blocks were prepared by heating at  $1,400^\circ\text{C}$  for 2 h under  $\text{N}_2$  gas. After full sintering at  $1,650^\circ\text{C}$  for 2 h, the linear shrinkage value was recorded at  $19.88 \pm 0.56\%$ . The flexural strength and fracture toughness were measured, the results were  $891.21 \pm 37.25$  MPa and  $6.33 \pm 0.30$   $\text{MPa} \cdot \text{m}^{1/2}$ , respectively. These results showed that flexural strength and fracture toughness of  $\text{Si}_3\text{N}_4$  were more than 800 MPa and 5  $\text{MPa} \cdot \text{m}^{1/2}$ , the white  $\text{Si}_3\text{N}_4$  developed in this study can be used to fabricate multi-unit dental restorations According to ISO 6872.

**Keywords:** Silicon nitride, CAD/CAM, Linear Shrinkage, Flexural strength, Fracture toughness

### INTRODUCTION

In dentistry, ceramic is currently widely used as restorative material because of its outstanding properties of biocompatibility and esthetics<sup>1-4</sup>. One of the most common ceramic core materials used in all ceramic restoration is yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) because of its high fracture toughness and strength. However, there are reports showing that Y-TZP can exhibit a progressive aging degradation, which limits its long-term stability<sup>1-5</sup>. A number of reports indicated that aging occurs by a progressive tetragonal to monoclinic transformation at the surface triggered by water molecules penetrate the interior zirconia lattice, which can result in surface roughening and microcracking<sup>1-7</sup>. These underlying problems led us to investigate and develop other alternative materials to replace zirconia.

Silicon nitride ( $\text{Si}_3\text{N}_4$ ) ceramic is more reliable than zirconia in term of its structure and thus it is a preferred candidate for many applications in industrial engineering and orthopedic implants<sup>8-11</sup>. As an alternative to zirconia,  $\text{Si}_3\text{N}_4$  ceramic has a potential because of its high fracture toughness and strength, wear resistance, antimicrobial and non-cytotoxicity<sup>9-15</sup>. Therefore  $\text{Si}_3\text{N}_4$  ceramic is a suitable material that can be used to produce a core structure for dental crowns or bridges. However, only few studies in  $\text{Si}_3\text{N}_4$  ceramic for dental application were done in the past 10 years. The main reason was that the obtained products are rather dark or grayish in color. Currently, the white color and high density  $\text{Si}_3\text{N}_4$  ceramic can be prepared by pressureless sintering at  $1,650^\circ\text{C}$  in

nitrogen atmosphere<sup>14</sup>. Moreover, the result of the study by Wananurksawong *et al.* had showed that  $\text{Si}_3\text{N}_4$  ceramic core material with borosilicate glass veneering ceramic can be used to produce dental restorations in term of strength and biocompatibility<sup>14,16</sup>.

The purpose of this study was to produce partially-sintered block of  $\text{Si}_3\text{N}_4$  for fabrication of dental restorations using commercial dental computer-aided design (CAD) and computer-aided manufacturing (CAM) software and milling system. This study focuses on the application of  $\text{Si}_3\text{N}_4$  ceramic as a core structure for dental crowns and bridges. The related material properties (sintering shrinkage, flexural strength and fracture toughness) were measured and compared to those of commercial dental zirconia as a control.

### MATERIALS AND METHODS

$\text{Si}_3\text{N}_4$  ceramic was developed and evaluated, comparing to a commercial zirconia (inCoris ZI 40/19, Lot no. 2011482375, Dentsply Sirona, USA)

#### *Partially-sintered $\text{Si}_3\text{N}_4$ ceramic blocks preparation*

Partially-sintered  $\text{Si}_3\text{N}_4$  ceramic blocks (Fig. 1,  $39 \times 19 \times 15.5$  mm) were prepared according to Wananurksawong *et al.*, 2011<sup>14</sup>. The raw materials consist of high purity  $\alpha$ - $\text{Si}_3\text{N}_4$  powders (particle size  $0.8 \mu\text{m}$ , SN E-10 grade, Ube Industries, Tokyo, Japan) with  $\text{Y}_2\text{O}_3$  (RU, Shin-Etsu Chemical, Tokyo, Japan),  $\text{SiO}_2$  (KE-P30, Nippon Shokubai, Osaka, Japan), and  $\text{MgO}$  (MJ-30, surface area  $31.7 \text{ m}^2/\text{g}$ , Iwatani, Tokyo, Japan) as sintering aids with the weight ratio of silica:magnesia:yttria at 3:3:5,

respectively. The powders were mixed by ball milling in a polyethylene bottle with  $\text{Si}_3\text{N}_4$  balls in ethanol for 24 h. Subsequently, the mixture was added in Polyvinyl butyral (PVB 630, mineral 75% and viscosity 50–70 cP) by 1 wt%. The homogenized mixture was dried at 60°C in rotary evaporator and screening through a 100-mesh sieve. The mixed powder was formed by hydraulic pressing at 50 MPa and binder burnout at 600°C for 1 h in air. Specimens were pre-sintered in a crucible by varying temperatures (1,400, 1,450 and 1,500°C) for 2 h under 1 atm of  $\text{N}_2$  gas flow.

#### Sintering shrinkage evaluation

This study was designed in order to investigate the effect of pre-sintering temperature (1,400, 1,450 and 1,500°C) to linear sintering shrinkage of fully crystallized  $\text{Si}_3\text{N}_4$  ceramic comparing that of a commercial zirconia ceramic (inCoris ZI 40/19, Dentsply Sirona). To determine the linear sintering shrinkage, pre-sintered  $\text{Si}_3\text{N}_4$  ceramic and commercial zirconia rectangular bar specimens ( $n=10$  for each sintering temperature level) were produced by using a high-speed cutting machine with 5×7×39 mm in dimension. Then, fully crystallizing  $\text{Si}_3\text{N}_4$  bar specimens using sintering temperature at 1,650°C for 2 h in  $\text{N}_2$  atmosphere. Fully crystallized commercial zirconias (inCoris ZI) were prepared by sintering process according to manufacturer's instruction by choosing



Fig. 1 Partially-sintered  $\text{Si}_3\text{N}_4$  ceramic block (39×19×15.5 mm).

program inCoris ZI (Dentsply Sirona inFire HTC furnace) (Table 1).

The linear sintering shrinkage (%) was calculated from the difference in length, width and height between before and after sintering (measured with Mitutoyo calipers with a resolution of 0.05 mm) as following equation;

$$\Delta L = [L_0 - L] / L_0 \times 100$$

$$\Delta W = [W_0 - W] / W_0 \times 100$$

$$\Delta H = [H_0 - H] / H_0 \times 100$$

where  $\Delta L$ ,  $\Delta W$ ,  $\Delta H$ : linear sintering shrinkage (%)

$L_0$ ,  $W_0$ ,  $H_0$ : the length, width and height of the specimen before sintering

$L$ ,  $W$ ,  $H$ : the length, width and height of the specimen after sintering

#### Flexural strength and fracture toughness specimens preparation

To determine the flexural strength and fracture toughness, pre-sinter  $\text{Si}_3\text{N}_4$  ceramic (partially sintered at 1,400°C) and commercial zirconia rectangular bar-shaped specimens ( $n=10$  for each group) were produced using high-speed cutting machine (5×7×39 mm in dimension). All specimens were ground using 1 micron diamond slurry. The edges were chamfered according to ISO 6872(2015)<sup>17</sup>. Then, fully crystallizing  $\text{Si}_3\text{N}_4$  ceramic using sintering temperature of 1,650°C for 2 h in  $\text{N}_2$  atmosphere. Fully crystallized commercial zirconias (inCoris ZI) were prepared by sintering process according to manufacturer's instruction.

#### Flexural strength evaluation

The three-point bending flexural strength tests were done under displacement control (crosshead speed 0.5 mm/min), using an Instron universal testing machine with a 5 kN load cell. The flexural strength values were calculated using an equation described by the ISO 6872(2015)<sup>17</sup>.

$$\sigma = 3 FL / 2 wh^2$$

where  $\sigma$  is modulus of rupture or flexural strength (MPa)

$F$  is force (N)

$L$  is length of the span (mm)

$w$  is width of the specimen (mm)

$h$  is height (thickness) of the specimen (mm)

#### Fracture toughness evaluation

The specimens were tested for fracture toughness using

Table 1 Sintering process of inCoris ZI according to manufacturer's instruction (inCoris ZI, Dentsply Sirona, Technical Documentation)

Heating rate (°C /min)	Holding temperature (°C)	Holding time (min)
25	800	0
15	1,510	120
30	200	0

single edge “V” notch beam method. The V-notch of the specimens ( $n=10$ ) were created using diamond saw disc. Specimens were positioned with the V-notched centered on the supporting rollers of the four-point bending apparatus following the recommendations of the ISO 6872(2015)<sup>17)</sup> consisted of four 5 mm diameter stainless steel rollers. The supported roller were placed 24 mm apart and the loading rollers were positioned 12 mm apart on the top of the specimen. (Fig. 2) The specimen was loaded to failure with a crosshead speed of 0.5 mm/min. The fracture load was recorded using an Instron universal testing machine. The specimens were evaluated under 10x magnification using a stereomicroscope to assure that the fracture started at the bottom of the notch and continued over its entire length (if this was not the case the specimen had to be discarded). The depth of notch was measured under SEM at 50x magnification (3 times per specimen). A fracture surface were randomly observed under SEM at 30000x magnification. The fracture toughness were calculated according to the

equation described by the ISO 6872(2015).<sup>17)</sup>

$$K_{IC} = \sigma \sqrt{a} Y = \frac{P}{b\sqrt{w}} \times \frac{S1-S2}{w} \times \frac{3\sqrt{a}}{2(1-\alpha^{1.5})} Y$$

$$\alpha = a/w$$

Where  $K_{IC}$  is the fracture toughness in megapascals by square for meter  
 $\sigma$  is the fracture toughness in megapascals  
 $P$  is the fracture load in meganewton  
 $b, w$  is the specimen's thickness and width in meters  
 $S1, S2$  are the support span ( $S1>S2$ ) in meter  
 $Y$  is the stress intensity shape factor  
 $a$  is the average notch depth in meters  
 $\alpha$  is the relative notch depth

#### Statistical analysis

For shrinkage statistical analysis, Shapiro-Wilk normality test showed that shrinkage data of at least one group was not normally distributed. Therefore, non-parametric one-way ANOVA (Kruskal-Wallis) test was used for statistically analysis of the linear shrinkage between experimental groups. All tests were conducted at a 95% level of confidence.

For flexural strength and fracture toughness statistical analysis, Shapiro-Wilk normality test showed that flexural strength data and fracture toughness of all groups are normally distributed. An independent *T*-test was used to compare the flexural strengths and fracture toughness between  $Si_3N_4$  and inCoris ZI. All tests were conducted at a 95% level of confidence.

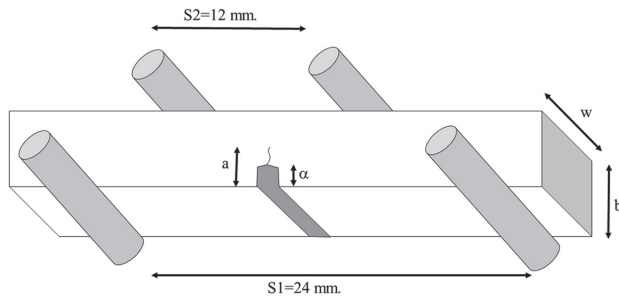


Fig. 2 Fracture toughness specimen.

Table 2 Linear sintering shrinkage (%) values of  $Si_3N_4$  at three difference partially sintered temperature (1,400, 1,450 and 1,500°C) comparing to commercial zirconia (inCoris ZI; as control group) by Kruskal-Wallis test

No	Linear shrinkage (%)											
	$Si_3N_4$									inCoris ZI		
	Presinter at 1,400°C			Presinter at 1,450°C			Presinter at 1,500°C					
	$\Delta L$	$\Delta W$	$\Delta H$	$\Delta L$	$\Delta W$	$\Delta H$	$\Delta L$	$\Delta W$	$\Delta H$	$\Delta L$	$\Delta W$	$\Delta H$
1	20.13	19.27	20.51	16.96	17.29	16.83	6.62	5.65	6.48	20.34	20.50	20.41
2	19.79	18.27	20.49	16.98	15.24	17.92	6.37	5.46	7.24	20.32	20.40	20.33
3	19.82	19.57	20.22	16.95	15.56	17.78	6.82	5.84	7.40	20.30	20.03	19.96
4	20.04	19.00	21.21	16.88	14.95	18.53	6.71	6.72	7.54	20.32	20.17	21.43
5	19.75	20.56	19.67	16.95	17.97	17.18	6.91	6.82	7.23	20.27	19.90	21.59
6	19.71	19.57	20.24	16.96	16.92	16.93	6.82	7.44	6.62	20.32	20.66	20.61
7	20.03	19.60	19.35	16.69	15.75	15.95	7.09	7.65	7.84	20.28	20.17	20.49
8	19.73	19.17	19.86	16.97	16.67	15.79	6.93	6.91	7.51	20.32	19.90	20.33
9	20.20	20.26	19.78	16.91	17.49	16.14	6.95	7.78	7.85	20.28	20.63	20.21
10	19.83	20.39	20.30	16.72	17.97	16.69	6.65	8.33	6.80	20.32	20.33	21.22
Mean (SD)	19.88 (0.56) <sup>A</sup>			16.82 (0.86) <sup>B</sup>			6.97 (0.65) <sup>C</sup>			20.41 (0.39) <sup>A</sup>		

SD=Standard deviation.

Different superscript uppercase letters show linear shrinkage with statistically significant difference ( $p>0.05$ ) according to Kruskal-Wallis.



## RESULTS

### Sintering shrinkage evaluation

The results of linear sintering shrinkage percentage of  $\text{Si}_3\text{N}_4$  at three difference partially sintered temperatures (1,400, 1,450 and 1,500°C) comparing to commercial zirconia (inCoris ZI; as control group) were listed in Table 2. The pre-sintered temperature of  $\text{Si}_3\text{N}_4$  increasing from 1,400, 1,450 and 1,500°C, led to decreasing linear shrinkage (%) from  $19.88 \pm 0.56$ ,  $16.82 \pm 0.86$  and  $6.97 \pm 0.65$  respectively. According the result of this study was supported that statistically not different of linear sintering shrinkage (%) between  $\text{Si}_3\text{N}_4$  (Presinter at 1,400°C) and InCoris ZI.

### Flexural strength and fracture toughness

The results of flexural strength values (MPa) and fracture toughness  $K_{IC}$  ( $\text{MPa} \cdot \text{m}^{1/2}$ ) for each group were presented in Table 3. The study found that fully sintered  $\text{Si}_3\text{N}_4$  has average flexural strength of  $891.21 \pm 37.52$  MPa and average fracture toughness of  $6.33 \pm 0.30$   $\text{MPa} \cdot \text{m}^{1/2}$ , while fully sintered zirconia has average flexural strength of  $960.34 \pm 25.97$  MPa and average fracture toughness of  $5.94 \pm 0.32$   $\text{MPa} \cdot \text{m}^{1/2}$ . The SEM image of

fully sintered  $\text{Si}_3\text{N}_4$  and zirconia ceramic structure are shown in Figs. 3 and 4, respectively. Figure 3 shows dark gray of  $\text{Si}_3\text{N}_4$  rod-like interlocked grains glued to each other by partially crystalline grains and a lighter gray of grain-boundary glassy phase. Figure 4 shows gray of fine tetragonal zirconia polycrystalline grains and the grain size was not uniform. Pores appear black from grains-pull out were observed.

## DISCUSSION

### Sintering shrinkage evaluation

Fully-sintered  $\text{Si}_3\text{N}_4$  is too hard to mill and may easily damage the milling burs. Instead, the fabrication of  $\text{Si}_3\text{N}_4$  should be machined by CAD/CAM system in a partially-sintered (presinter) block. The pre-sinter temperature of partially crystallized ceramic may have an effect to linear shrinkage and fit of the final restorations. Thus, the suitable pre-sinter temperature that was used to prepare CAD/CAM  $\text{Si}_3\text{N}_4$  block should also be considered. The results from this study reveal that the most suitable pre-sintering temperature for preparing  $\text{Si}_3\text{N}_4$  blocks is 1,400°C where the linear shrinkage ( $19.88 \pm 0.56\%$ ) is most comparable to that of commercial CAD/CAM

Table 3 Mean (SD) of flexural strength and fracture toughness of  $\text{Si}_3\text{N}_4$  comparing to commercial zirconia (inCoris ZI; as control group) by independent *T*-test

Mean (SD)			
Flexural strength (MPa)		Fracture toughness, $K_{IC}$ ( $\text{MPa} \cdot \text{m}^{1/2}$ )	
$\text{Si}_3\text{N}_4$ ( <i>n</i> =10)	inCoris ZI ( <i>n</i> =10)	$\text{Si}_3\text{N}_4$ ( <i>n</i> =10)	inCoris ZI ( <i>n</i> =10)
891.21 (37.25) <sup>A</sup>	960.34 (25.97) <sup>B</sup>	6.33 (0.30) <sup>a</sup>	5.94 (0.32) <sup>b</sup>

SD=Standard deviation.

Different superscript capital letters show mean values of flexural strength with statistically significant difference ( $p > 0.05$ ) according to independent *T*-test.

Different superscript lowercase letters show mean values of fracture toughness with statistically significant difference ( $p > 0.05$ ) according to independent *T*-test

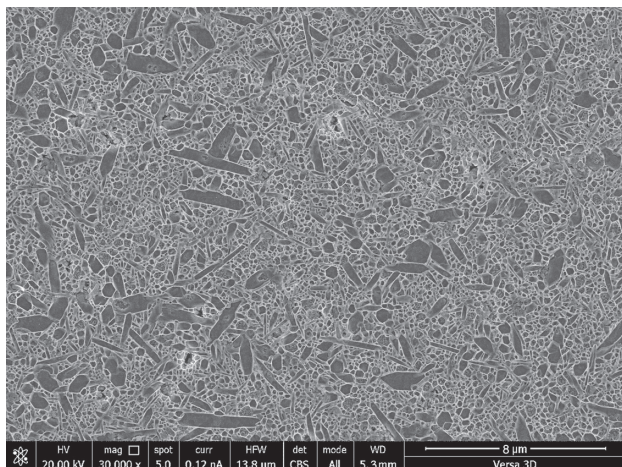


Fig. 3 SEM image of fully sinter  $\text{Si}_3\text{N}_4$  specimen.

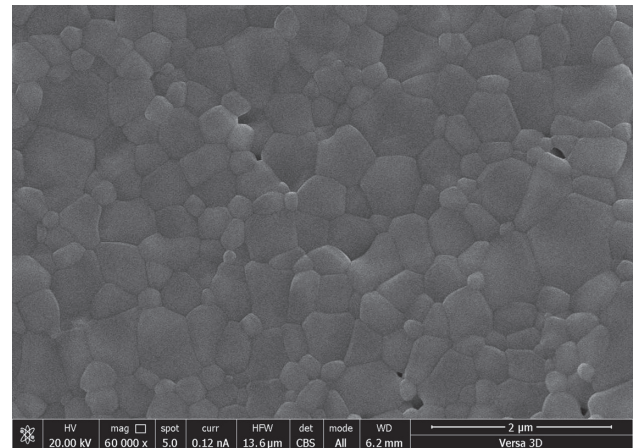


Fig. 4 SEM image of fully sinter commercial zirconia (inCoris ZI) specimen.

zirconia blocks ( $20.41 \pm 0.39\%$ ).

#### Flexural strength and fracture toughness

The strength of  $\text{Si}_3\text{N}_4$  ceramic was evaluated in this study, comparing to that of commercial zirconia.  $\text{Si}_3\text{N}_4$  has significantly lower flexural strength but higher fracture toughness than that of commercial zirconia. However, flexural strength and fracture toughness of both  $\text{Si}_3\text{N}_4$  and commercial zirconia were more than 800 MPa and  $5 \text{ MPa} \cdot \text{m}^{1/2}$  that means both materials can be used to fabricate four or more units dental bridges (according to ISO 6872, 2015)<sup>17</sup>. As a results,  $\text{Si}_3\text{N}_4$  ceramic can be used for fabrication of dental core for posterior molar crown or bridges.

In the present study, flexural strength and fracture toughness from different materials differed significantly, this may due to the difference in microstructures<sup>18</sup>.  $\text{Si}_3\text{N}_4$  derives its strength and toughness from its microstructure, which is composed of asymmetric  $\beta\text{-Si}_3\text{N}_4$  rod-like interlocked grains (crystals) glued to each other by partially crystalline (crystals) grain boundary phase or grain-boundary glassy (amorphous) phase (Fig. 3)<sup>9,13,19</sup>. Unlike zirconia, no phase transformation is involved,  $\text{Si}_3\text{N}_4$  exists as an irreversibly stable phase at room temperature, but an advancing crack must navigate a high energy path through the ceramic, and bridging grains within the crack wake restrict its continued propagation<sup>13</sup>. While, the toughness of Y-TZP is dependent on the phase transformation-toughening ability. The tetragonal grains (Fig. 4) can expand at the crack tip and transform into a monoclinic, stable phase and prevent cracks from developing<sup>2,3</sup>.

Flexural strength and fracture toughness test with bar-shape specimens were basic methods for predicting performance of dental ceramic. However, clinically crown-shape restoration may show different behavior than bar-shape specimen due to their complex geometry. The fracture toughness also depends on test method that make some inconsistency of fracture toughness of the same material from difference studies<sup>20</sup>. In this study, the determination of fracture toughness by single edge notched beam method with high sensitivity in sharpening notch for tough zirconia<sup>20</sup>. Therefore, the data obtained from this study must be limited in comparison only in this study. Further studies should therefore be investigated to confirm the strength and minimal thickness requirement for the CAD/CAM  $\text{Si}_3\text{N}_4$  crown restoration. In addition, machinability of new CAD/CAM material is a very important. The mechanical tests related to machinability, such as brittleness index and hardness, necessary to considered of partially-sintered CAD/CAM  $\text{Si}_3\text{N}_4$  block<sup>21</sup>.

#### CONCLUSION

This *in vitro* study revealed that white pre-sintered  $\text{Si}_3\text{N}_4$  ceramic at  $1,400^\circ\text{C}$  can be used as an alternative material for fabrication of a core structure for dental restorations when using CAD/CAM system. Nevertheless, there are many factors that could affect the quality of dental

restoration such as marginal and internal gap, surface wear, tooth antagonist wear, esthetic, adhesive bonding material and procedures. Therefore, further pre-clinical and clinical studies are needed in order to finalize the properties of this new material before this new material could be launched into the market for clinical use.

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#### CONFLICT OF INTEREST

On behalf of all authors, the corresponding author declare no conflicts of interest.

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