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APPENDICES

APPENDIX A

Publications Resulting from This Research Work

PUBLICATION

International Research Paper

1. C. Promdej, S. Areeraksakul, V. Pavarajarn, S. Wada, T. Wasanapiarnpong and T. Charinpanikul, "Preparation of translucent alumina ceramic specimen using slip casting method," *Journal of the Ceramic Society of Japan,* Vol. 116 [3], **2008**, Pages 409-413.

International Proceeding

1. C. Promdej, T. Wasanapiarnpong, S. Wada, and T. Charinpanikul, "A Challenge in Preparation of Transparent Alumina Ceramic Specimen Using Slip Casting Method" *Proceedings of International Conference on 7th Pacific Rim Conference on Ceramic and Glass Technology(PAC RIM 7)*, pp. 108, November 11-14, **2007**, Shanghai, China.

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PacRim 7 Abstract Book

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Reaction-Bonded Al_2O_3 ceramics using high Al alloy powder content was successfully fabricated. Al_2O_3/Al alloy powder mixture was milled, reaction-bonded, post-sintered, and characterized. During reaction bonding of Al_2O_3/Al alloy compacts, oxidation of Al alloy took place by solid and liquid state oxidation. The solid state oxidation exhibited little dependence on Al alloy particle size after milling, but showed strong dependence on surface defects of Al alloy particle on milling. Liquid state oxidation of Al alloy continuously proceeded and completed at a given temperature with a help of alloying elements. Al_2O_3/Al alloy powder compacts with high Al alloy content of ~80 vol.% could be successfully reaction-boned without cracks.

Session Chair: Jianfeng Yang, Xi'an Jiaotong University, China

S5-23-I, Nov. 13, 15:30-16:00
Influence of Catalyst Content on the Microstructure of Carbon/Carbon Composites by Chemical Liquid-Vapor Infiltration Process

H. J. Li,* X. T. Li, K. Z. Li, Q. G. Fu and C. Wang
Northwestern Polytechnical University, China

The catalyst content has very great influence on the microstructure of carbon/carbon composites densified by chemical liquid-vapor infiltration process. For investigating the influence, ferrocene, as the catalyst, had been added. The content was respectively designed as 0, 1, 3, 5 and 10 weight percentage corresponding to carbon felt preforms. The textures of C/C composites were analyzed by a polarized light microscope and a field emission scanning electron microscope. Results show that the texture of C/C composites without catalyst mainly consists of rough laminar (RL) pyrocarbon. After adding the catalyst, regeneration laminar pyrocarbon have been obtained when ferrocene content is 1 wt. %. When ferrocene content increases from 3 wt. % to 10 wt. %, the microstructures transfer from RL to SL pyrocarbon. RL pyrocarbon firstly forms around carbon fiber, following by SL pyrocarbon. The layer width of SL pyrocarbon increases and which of RL pyrocarbon decreases with the increase of ferrocene contents in the preforms.

S5-24-I, Nov. 13, 16:00-16:30
Processing of Advanced MMC's and CMC's by Reactive Infiltration

R. Janssen,*[1] N. Claussen,[1] N. A. Travitzky[2] and P.Greil[2]
[1]Hamburg University of Technology, Germany;
[2]Friedrich-Alexander University of Erlangen-Nuremberg, Germany

Liquid-reaction processing of composites represents a low-cost, fast, and simple manufacturing route to high-performance components. In this lecture, the processing of metal-ceramic composites/systems based on infiltration techniques are discussed with respect to their special features and advantages. Special emphasis is given to aluminium/alumina containing composites fabricated by reacting aluminium and oxides, i.e. Fe_2O_3, TiO_2, or even ores like ilmenite or chromite, at temperatures above T_{mAl}~660°C. The reactive infiltration is performed either by pressure assisted infiltration of molten aluminium into porous preforms or by in situ infiltration via heating powder mixtures containing safe (5 to 50 um) Al particles in a pressurized hot cell. Whereas the later one is especially suited for highly dense but geometrically simple (i.e. disc-like) parts, the infiltration by pressure casting routes allows the fabrication of complex shaped parts which contain typically some residual porosity. Both routes offer refractory metal-ceramic composites, i.e. even metallic alloys corresponding to aluminides or superalloys can be formed during low temperature processing. In the paper, the different reaction techniques will be outlined by examples of recent research and technology.

Session Chairs: Akira Kishimoto, Okayama University, Japan; Hyun-Kwuon Lee, Kumoh National Institute of Technology, Korea

S5-25-O, Nov. 13, 16:30-16:50
A Challenge in Preparation of Transparent Alumina Ceramic Specimen Using Slip Casting Method

C. Promdej,* T. Wasanapiarnpong, W. Shigetaka, T. Charinpanitkul
Chulalongkorn University, Thailand

A commercial alumina nanopowder was applied in a challenge to prepare transparent polycrystalline alumina ceramic specimens using slip casting method. Green body of alumina ceramic with the relative density higher than 55%, could be prepared from homogenous slurry which is a suspension of alumina powder mixed with added some deflocculant and binder. In this work, the deflocculant (polymethacrylic acid (PMAA)) and binder (polyvinyl alcohol (PVA)) have been employed to prevent the agglomeration of alumina particles in suspension, and improvement of green body strength, due to diminishing of abnormal grain growth.

S5-26-O, Nov. 13, 16:50-17:10
Preparation and Microstructure Characterization of Bulk Ti_2SnC

J. Zhang* and Y. C. Zhou
Institute of Metal Research, Chinese Academy of Sciences, China

The microstructures of bulk Ti_2SnC ceramic have been investigated using X-ray diffraction and transmission electron microscopy. The low-magnification TEM image shows that Ti_2SnC grains are not equiaxed, they

Journal of the Ceramic Society of Japan 116 [3] 409-413 2008 Paper

Preparation of translucent alumina ceramic specimen using slip casting method

Chutinan PROMDEJ, Sakkapas AREERAKSAKUL, Varong PAVARAJARN,* Shigetaka WADA, Thanakorn WASANAPIARNPONG** and Tawatchai CHARINPANITKUL†**

Center of Excellence in Particle Technology, Faculty of Engineering, Chulalongkorn University, Payathai Rd., Patumwan, Bangkok 10330, Thailamd
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A commercial alumina nanopowder has been applied to prepare high density alumina ceramic specimens using a simple slip casting method. Slurry preparation process is one of important steps to enhance density and optical property of alumina specimens to be fabricated. In this work, the effect of the amount of ammonium salt of poly(methacrylic) acid dispersant and polyvinyl alcohol binder in alumina suspension on the slurry viscosity was discussed. Green body with relative density higher than 55% could exhibit homogenous appearance when a certain ratio of dispersant to binder is employed. The fully dense translucent alumina compact consisting of submicron grain could be fabricated by atmospheric sintering at 1350°C with soaking time of 3600 s and further HIP-sintering at 1300°C for 7200 s in argon atmosphere.

Key-words : Alumina ceramic, Slip casting, Dispersant, Binder, Densification, Sintering

[Received November 18, 2007: Accepted February 21, 2008]

1. Introduction

Alumina ceramic materials have been used in various technological applications because of their advanced physical and chemical properties. It is well known that sintered polycrystalline alumina (PCA) which contains small grain size with high density could exhibit the transparent property.[1)-4)] Referring to the Rayleigh scattering model, Rolf et al.[5)] demonstrated that sintered PCA consisting of mean grain sizes smaller than 2 μm could provide outstanding optical transmitting property. However, sintering of ceramic specimens at high temperature to increase their density could oppositely result in growing of crystal grain.[6)-9)] Therefore, a controlled sintering process at comparatively low temperature to avoid grain growth is a key to get transparent alumina sintered specimens.

In general there are various shaping methods to prepare ceramic specimens. Anyway slip casting is normally used for forming of traditional ceramics because it could fabricate green body specimen in complicated shape with relatively high density. An important issue to fabricate green body with high density is to control slurry viscosity while solid content with uniform dispersion must be controlled.[10),11)] Accordingly various dispersants have been investigated because they could suppress agglomeration of suspending particles by increasing the interparticle repulsive force.[12),13)] Meanwhile some binders are also added into ceramic slurry for improving strength of green body.[14),15)]

The aim of this work is to investigate the possibility to make use of simple slip casting method to prepare translucent alumina ceramic specimens from polycrystalline commercial alumina, which will be a step towards transparent ceramic fabrication. Then the effects of dispersant and binder on properties of alumina slurry is examined to find out the suitable condition to prepare alumina slurry with high solid content for slip casting. Characteristics of calcined and sintered alumina specimens are carried out and then discussed with other previous results.

2. Experimental

2.1 Material

A high-purity (99.99%) commercial alumina powder (TM-DAR, Taimei Chemicals, Co. Ltd., Japan) with 0.17 μm mean diameter and 13.5 m^2/g specific surface area has been used for preparing alumina slurry. Ammomium salt of poly(methacrylic) acid (NH_4^+ -PMAA; Aron A6114, Molecular Weight 6000, Toagousei Co. Ltd., Japan) is added into the slurry to prevent agglomeration of suspended alumina particles. Additionally, a commercial organic binder, polyvinyl alcohol (PVA, Molecular Weight 13000-23000, Sigma-Aldrich, Inc., USA) is also employed to enhance mechanical strength of green body prepared in each experiment.

2.2 Sample preparation

Alumina slurries for slip casting have been prepared by dispersing alumina powder in demineralized water with the addition of dispersant and binder of 0.75-3.0 and 0.0-0.5 mass% of solid content, respectively. The prepared suspension is then ball-milled by high-grade alumina balls in a Polyethylene (PE) container for 86400 s (24 h). Cylindrical pellets with 30 mm diameter and 4 mm thickness are prepared from the homogenous slurry by slip casting method

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using gypsum molds, which are intentionally prepared to avoid contamination. After slip casting, green body specimens are dried in air and removed from gypsum mold before being dried in an oven at 120°C for 86400 s (24 h) and then calcined at 800°C for 7200 s (2 h). Calcined specimens are subject to 1 kg/m^3 HCl treatment for 3600 s (1 h) in order to remove calcium ion contamination. In sintering process, the specimens are sintered at 1250–1400°C at a heating rate of 1/6°C/s for 1800–18000 s (0.5–5.0 h) in an atmosphere furnace (Vecstar furnace, England) and atmosphere controlled furnace (High muti-5000, Fujidempa, Kogyo Co., Ltd., Japan).

2.3 Analysis

The viscosity of alumina slurry is measured by a viscometer (Brookfield DV-E, Brookfield Engineering Laboratories Inc., Massachusetts, USA). The density of alumina specimens is determined by the Archimedes method. The microstructure of prepared specimen are characterized by scanning electron microscope (SEM) (JSM-6480LV, JEOL Ltd., Tokyo, Japan). The shrinkage of specimen is also determined from the dimensional change of the specimens before and after sintering.

3. Results and discussion

3.1 Effect of NH_4^+ salt of PMAA dispersant

The concentration of NH_4^+-PMAA in the range of 0.75–3.0 mass% alumina content is varied to investigate its effect on viscosity of alumina slurry prepared for slip casting. As could be observed in **Fig. 1**, an increase in the dispersant content could result in a drastic decrease in the apparent viscosity of the alumina slurry when the dispersant concentration is lower than a certain value. But with a further increase in the dispersant content would give rise to a gradual increase in the apparent viscosity of slurry. Therefore, there exists a minimal value of the slurry apparent viscosity, which is also affected by the solid content. With higher solid content (75 mass%) the minimal apparent viscosity shifts to a higher values due to the influence of solid interaction.[13] The minimal alumina slurry viscosity with solid loading of 70 and 75 mass% could be obtained with the dispersant concentration of 1.18 and 1.25 mass%, respectively. This phenomenon is reasonably attributed to the fact that when the amount of dispersant added to the alumina suspension is low, insignificant repulsive force acting among each suspended particle could not hinder the agglomeration of fine alumina particles as illustrated in **Fig. 2**(a). At the optimal addition of dispersant content, sufficient amount of dispersant molecules would attach to the surface of suspended alumina particles, resulting in a balance of repulsive and attractive forces acting on suspended particles (Fig. 2(b)). As a result, a homogeneous suspension of alumina particles prepared with a minimal viscosity could enable us to prepare repeatable slip-cast specimens. However, with a further increase in dispersant concentration, the excessive amount of dispersant molecules would oppositely hinder movement of particles, leading to agglomeration of dispersed particles due to surface tension effect of dispersant as depicted schematically in Fig. 2(c).

3.2 Effect of PVA binder on the viscosity of alumina slurry

PVA binder in the range of 0–0.5 mass% is added to the alumina slurries mixed with the optimal dispersant concentration of 1.18 and 1.25 mass% with respect to the alumina content of 70, and 75 mass%. The relationship between the apparent viscosity of the alumina slurry and PVA concentration is shown in **Fig. 3**. For 70 mass% alumina content slurry, the apparent viscosity gradually increases from 39 to 280 mPa·s with the increased binder content. Interestingly, with alumina content of 75 mass%, the slurry viscosity increases gradually with the binder content up to 0.3 mass% (490 mPa·s), and then drastically increases to 1900 mPa·s when the 0.4 mass% binder is added. This might be attributable that the binder molecules adsorbed on surface of alumina particles could form weak bridging forces among alumina particles, resulting in flocculation and more viscous slurry.

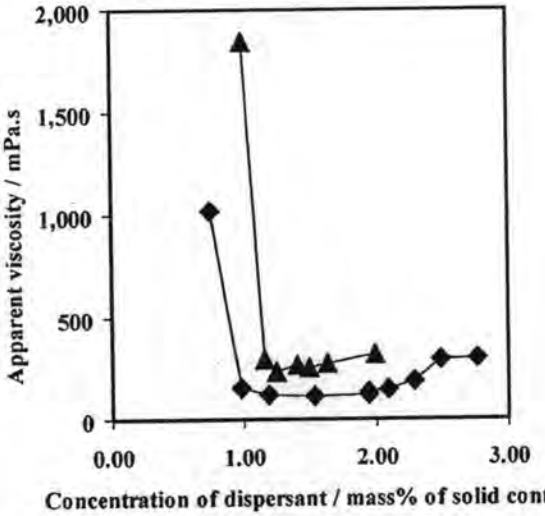

Fig. 1. Effect of NH_4^+ salt of PMAA dispersant concentration on viscosity of 70 mass% and 75 mass% solid loading (▲: 70 mass% alumina 1.18 mass% dispersant and ◆: 75 mass% alumina 1.18 mass% Dispersant).

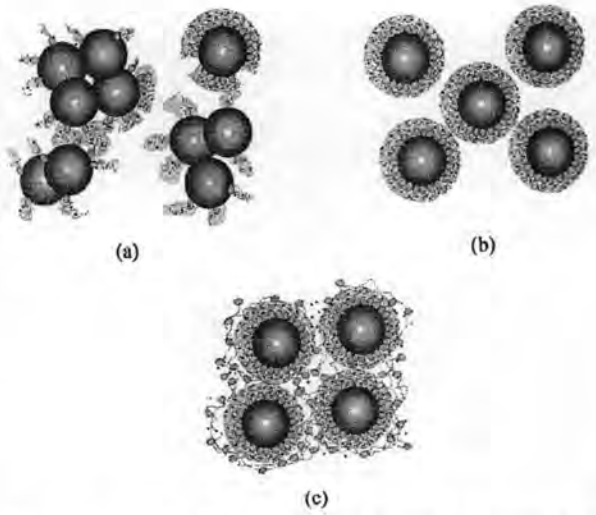

Fig. 2. A proposed model of alumina particle dispersion with different content of dispersant (a) insufficient dispersant (b) optimal dispersant (c) excessive dispersant.

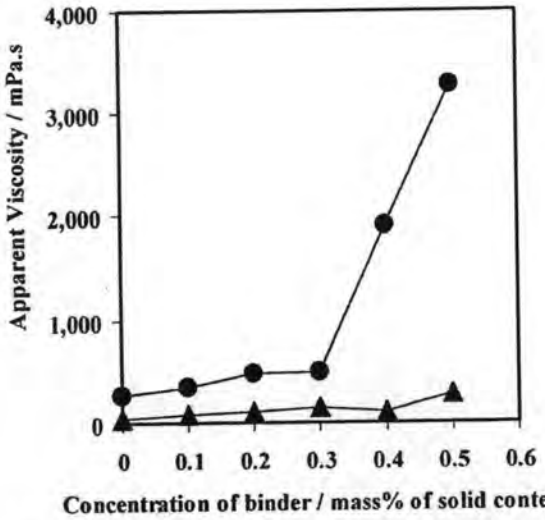

Fig. 3. Effect of PVA concentration and solid content on alumina slurry viscosity (●: 70 mass% alumina 1.18 mass% dispersant and ▲: 75 mass% alumina 1.18 mass% dispersant).

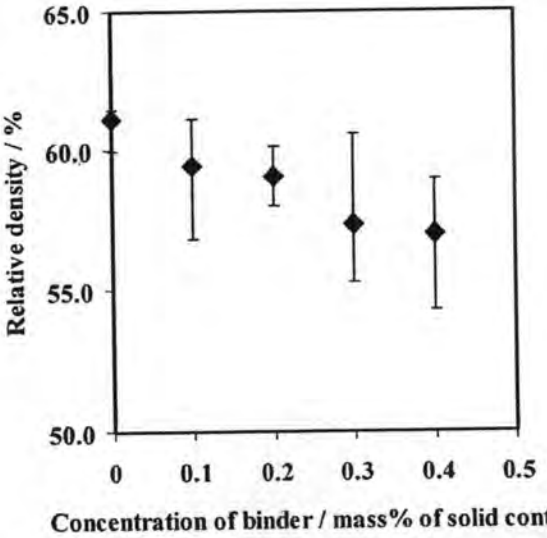

Fig. 4. Effect of PVA concentration on the relative density of calcined body with 75 mass% alumina content.

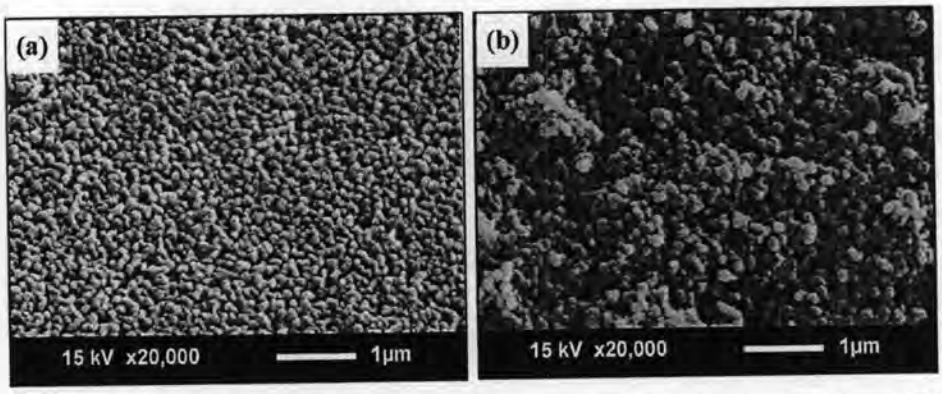

Fig. 5. SEM micrograph of (a) alumina green body and (b) calcined body with 75 mass% alumina content, 1.25 mass% NH_4^+ salt of PMAA dispersant and 0.2 mass% PVA.

It is well known that to obtain the green body with high density and high green strength, slurry with this highest alumina content is required. Higher alumina content would provide possibility to obtain green body with higher relative density due to formation of lower voidage.[14),16)] In this work the optimal composition of alumina suspension for slip casting in this experiment is 75 mass% alumina content and 1.25 mass% dispersant with the binder concentration not higher than 0.4 mass%. It is found that the strength of green body prepared with the binder content of 0.2 mass% is enough for handling.

3.3 Properties of calcined specimens

As shown in **Fig. 4**, the calcined alumina specimens with PVA content of 0.0–0.4 mass% have the relative density higher than 55%. It could experimentally be observed the relative density of prepared specimens decreases from 61% to 57% with the increasing PVA concentration from 0.0 to 0.4 mass%. The drop of relative density is attributed to an increase in porosity within the specimens due to the decomposition of PVA at the calcining temperature of 800°C. Baklouti et al.[15)] also reported a decrease in alumina specimen strength due to loss of organic binder after calcination because the calcined specimen strength is strongly dependent upon its porosity. **Figure 5.** is a typical microscopic image of alumina grains within the pre-calcined green body and calcined body specimens. It could be clearly observed that calcinations could result in a slice increase in the grain size but a small increase in the specimen porosity. For preparation of specimen with high relative density, sintering conditions would be taken into account in the next step of investigation.

3.4 Properties of sintered specimens

In the sintering step, calcined specimens will be heated to a certain temperature in a range of 1250–1400°C with a heating rate of 1/6°C/s. Then the specimens will be kept at the sintering temperature for a certain soaking time before cooled down with the cooling rate of 1/6°C/s. The relative density of sintered specimens is compared in **Fig. 6.** By pressureless sintering at 1300–1400°C with suddenly cooling (soaking time = 0 s), an increase in the relative density of sintered specimens (▲) from 77 to 97% could be achieved without

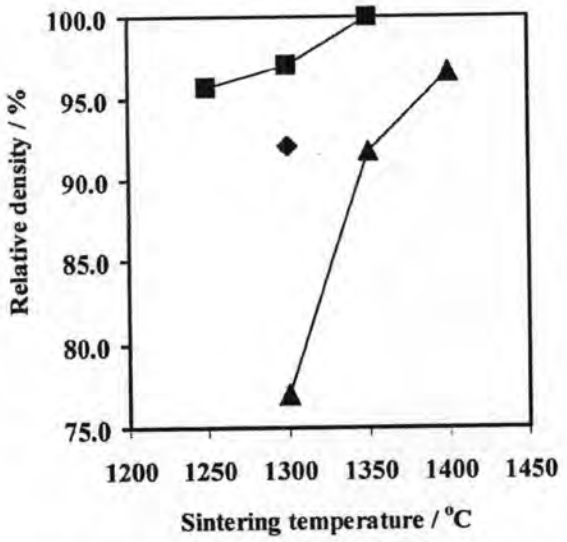

Fig. 6. Relative density of specimens sintered in atmosphere and pressureless furnaces at heating rate of 1/6°C/s and sintering temperature range of 1250 to 1400°C with different soaking time (▲: atmosphere sintering (soaking time 0 s), ■: atmosphere sintering (soaking time 7200 s) and ◆: pressureless sintering (soaking time 7200 s)).

Table 1. Relative Density, Grain Size and Linear Shrinkage of Alumina Specimen Sintered in Atmosphere Furnace at the Temperature of 1350°C and the Heating Rate of 1/6°C/s

Characteristic	Soaking Time				
	0 s	1,800 s	3,600 s	7,200 s	18,000 s
Relative Density (%)	91.6	99.1	99.9	100	100
Grain Size (μm)	-	0.23	0.24	0.60	0.62
Linear Shrinkage (%)	13.4	15.4	15.8	15.2	15.5

soaking period. On the other hand, the relative density of specimens sintered under atmospheric condition (■) increases from 96 to 100% when sintering at 1250–1350°C with soaking time of 7200 s (2 h). In general, it is supposed that alumina specimens sintered under vacuum condition would have relative density higher than that of atmospheric sintered specimens. However, the density of specimens sintered in a vacuum furnace (◆) is about 5% lower than that of atmospheric sintered specimens. This is attributable to the existence of graphite heating elements in the vacuum furnace, resulting in a reducing condition of oxygen with very low partial pressure. As a result, oxygen would react with the sintered alumina specimens and then lead to higher grain growth.[9]

Relative density, grain size and linear shrinkage of specimens sintered at 1350°C with different soaking time of 0 to 18000 s (5 h) are summarized in **Table 1**. The relative density increased from 91.6 to 99.9 with an increase in soaking time from 0 to 3600 s (1 h). Interestingly, it is found that the grain size increases insignificantly. On the other hand, with soaking time of 7200 s (2 h) or longer alumina grain size increases drastically while the specimen relative density

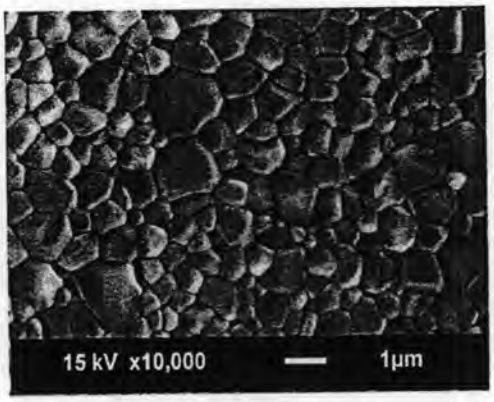

Fig. 7. SEM micrograph of alumina specimen sintered in atmosphere furnace at the temperature of 1350°C for 3600 s and the heating rate of 1/6°C/s.

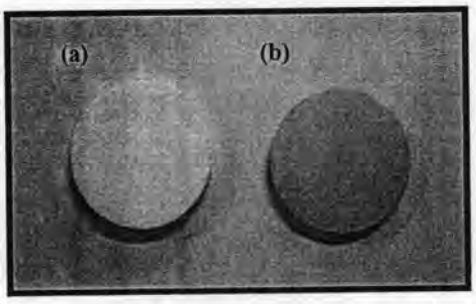

Fig. 8. Appearance of alumina specimens : (a) pre-sintered at 1350°C for 3600 s and (b) further HIP-sintered at 1300°C for 7200 s under 150 MPa in argon.

achieves 100%. This result suggests that suitable sintering time for preparing alumina specimen with fine grain size and high relative density should be selected. Meanwhile, the shrinkage ratio is not linearly affected by the soaking time. Comprehensive investigation on this issue should further be explored. A typical SEM micrograph of alumina specimen sintered at 1350°C for 3600 s (1 h) is shown in **Fig. 7**. Two pieces of 3.2-mm thick specimens are compared in **Fig. 8**. The left specimen was pre-sintered at a temperature of 1350°C for 3600 s (1 h) and the right specimen was further HIP-sintered at a temperature of 1300°C for 7200 s (2 h) under 150 MPa of argon. The later specimen occupying the relative density of 100% exhibits translucency as could be observed in Fig. 8. It contains fine grains with relatively uniform size smaller than 1 μm and some boundary motion as also reported by Wei.[17] Therefore, it should reasonably be noted that there is possibility to employ the simple slip casting method to prepare translucent and transparent alumina specimens. Anyway, it is also suggested that a post hot-isostatic pressing should be employed to improve the transparency of sintered specimens.

4. Conclusion

Dispersant and binder could provide significant effect on the apparent viscosity of alumina slurry which is attributable to the interaction among suspended particles. Alumina suspension suitable for slip casting could be prepared with a

condition of 75 mass% alumina content, 1.25 mass% dispersant and 0.2 mass% binder. The calcined alumina specimen could successfully be prepared with the relative density higher than 55%. Furthermore, fully densified translucent alumina specimen containing grains with uniform submicron size could be prepared by the atmospheric sintering at the temperature of 1350°C with the soaking time of 3600 s (1 h) and further HIP-sintered at 1300°C for 7200 s (2 h) in argon atmosphere.

Acknowledgement This work is supported by National Nanotechnology Center, National Science and Technology Development Agency. Partial support of Silver Jubilee Fund of Chulalongkorn University to Center of Excellence in Particle Technology is also gratefully acknowledged.

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APPENDIX B

EXPERIMENTAL RESULTS

Appendix B1 Calculation of green body strength

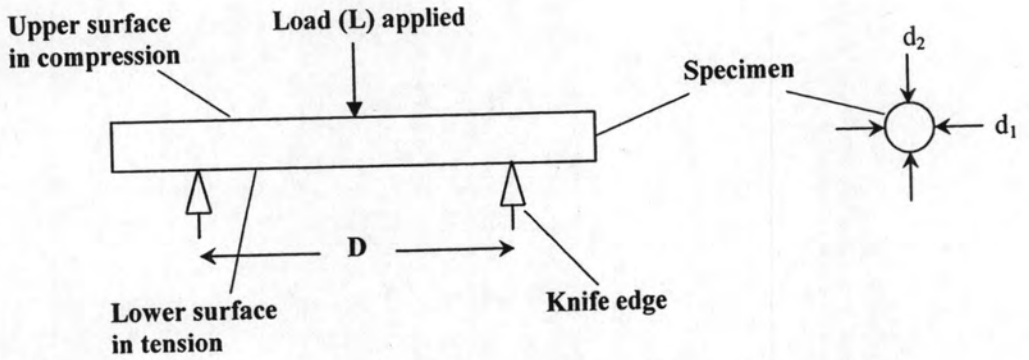


Figure B1 Schematic of strength test

$$MOR = \frac{8LD}{\pi(d_1)^2 d_2} \qquad (B1)$$

Where, L = Load applied

D = Distance between knife edges

d_1,d_2 = Diameter of specimen

For example, incase of the green body was the composition as binder 0.2 wt% with 75wt% alumina content and dispersant 1.25 wt%. The samples were cylindrical shape.

Code	Load applied (kg)	d1 (mm)	d2 (mm)	MOR (MPa)	Average MOR (MPa)
C3-1	1.755	12.4	12.35	11.67	10.36
C3-2	1.305	12.2	12.35	9.04	

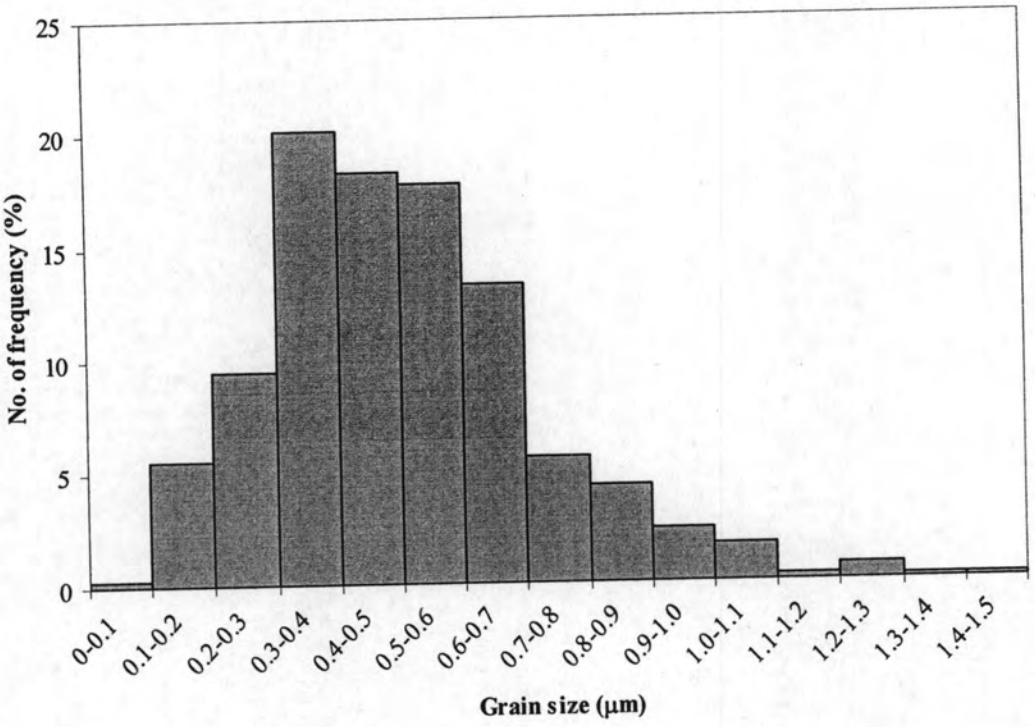
Appendix B2 Example calculation of relative density of calicined body with 75 wt% alumina content, dispersant 1.25 wt% and binder 0.2 wt%

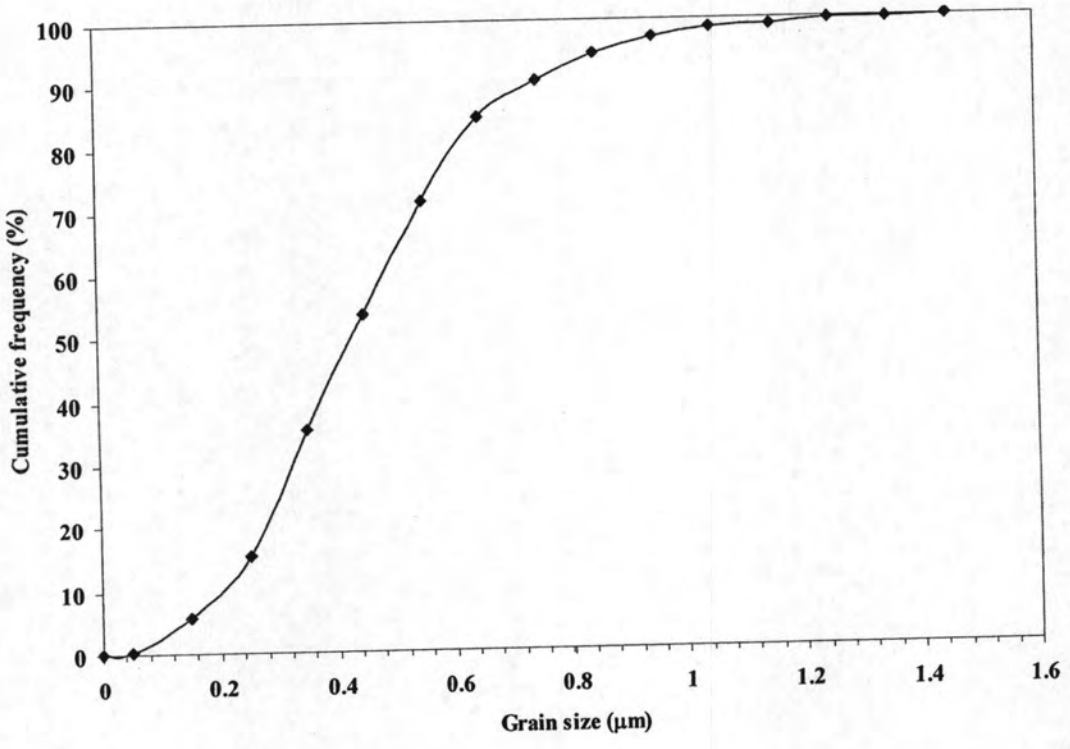
Code	Dry weight (g)	Weight in water (g)	Wet weight (g)	Density (g/cm^3)	Relative density (%)	AVG.	S.D.
C3-1	6.10	4.56	7.23	2.28	57.4	59.1	0.9
C3-2	6.76	5.02	7.91	2.34	58.8		
C3-3	6.66	4.93	7.79	2.33	58.6		
C3-4	6.07	4.51	7.10	2.34	59.0		
C3-5	5.95	4.43	6.96	2.35	59.2		
C3-6	6.97	5.20	8.13	2.38	59.9		
C3-7	6.00	4.47	6.99	2.38	59.9		
C3-8	7.95	5.93	9.26	2.39	60.1		

Appendix B3 Example calculation of relative density of pre-sintered body at temperature of 1300°C for 2 h as function of furnace type

Sintering condition	Code	Dry weight (g)	Weight in water (g)	Wet weight (g)	Density (g/cm^3)	Relative density (%)	AVG.	S.D.
Vacuum	SV1	6.73	4.91	6.74	3.67	92.3	92.1	0.3
	SV2	6.63	4.82	6.64	3.65	91.8		
	SV3	5.92	4.30	5.92	3.65	92.0		
Atmosphere	SA1	5.23	3.91	5.23	3.95	99.6	97.1	4.4
	SA2	5.20	3.89	5.20	3.96	99.7		
	SA3	4.67	3.44	4.72	3.65	92.0		

Appendix B4 Example calculation of grain size distribution of pre-sintered body at temperature of 1300°C for 2 h in atmosphere

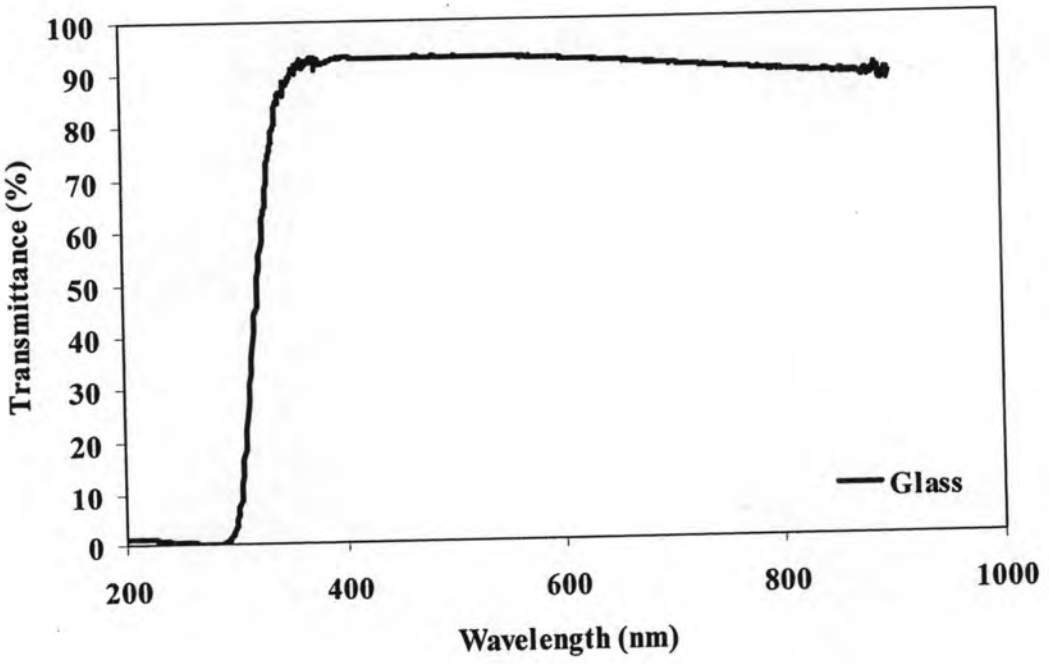




Appendix B5 Transmittance of alumina specimen after HIPed at 1300°C for 2 h under 150 MPa in argon atmosphere

Wavelength (nm)	% Transmittance				
	A1	A2	A3	B1	B2
900	81.87	68.74	72.95	74.78	71.91
850	80.68	67.85	71.77	73.75	71.05
800	80.45	66.89	71.43	73.09	69.77
750	79.91	65.91	70.67	72.11	68.50
700	79.46	64.91	69.88	71.07	67.02
650	78.97	63.85	69.08	69.87	65.39
600	78.39	62.68	68.13	68.44	63.46
550	77.55	61.19	66.93	66.68	61.11
500	76.72	59.68	65.68	64.78	58.72
450	75.85	58.31	64.52	62.85	56.40
400	75.43	57.43	63.90	61.13	54.49
350	78.64	60.80	68.11	63.48	56.87
300	78.49	57.87	68.38	61.42	56.21
250	45.91	21.42	29.71	24.61	19.47
200	23.83	9.11	13.84	11.23	7.32

Appendix B6 Transmittance of transparent glass



VITA

Miss Chutinan Promdej was born on February 17, 1984 in Bangkok, Thailand. She studied in primary and secondary educations at Chonpratarnvittaya School and Pakkred Secondary School, respectively. In 2005, she received the Bachelor Degree of Engineering (Chemical Engineering) from Chulalongkorn University. After that, she continued to study in Master program in Center of Excellence in Particle Technology (CEPT) at Chemical Engineering Department, Faculty of Engineering, Chulalongkorn University with the master thesis entitled "Transparent alumina ceramic with complicated shape prepared by slip casting".