Evaluation of some properties of an opaque porcelain fired simultaneously with the body porcelain

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Recently, a porcelain-fused-to-metal opaque porcelain was introduced that does not require a separate firing before application of the body porcelain. The objective of this study was to determine the properties of this new opaque porcelain and its ability to bond to metal. The properties studied included flexural strength, linear firing shrinkage, coefficient of thermal expansion, powder particle size, and ability to bond to body porcelain and dental alloys. Sintering of this opaque porcelain was complete when fired at 1760° F (960° C) with a linear firing shrinkage of 13.1% ± 0.2%. No boundary between the opaque and body porcelains could be found with a scanning electron microscope after firing at 1760° F (960° C). The mean flexural strengths were 99 ± 7 and 101 ± 8 MPa respectively, for this opaque porcelain and a conventional opaque porcelain, and were not significantly different as assessed with Student's *t*-test ($p = 0.548$). The coefficient of thermal expansion for this opaque porcelain was $13.3 \pm 0.2 \times 10^{-6}$/° C. Particle size analysis showed a 63% increase in the particles below 5 μm for this opaque porcelain and bonding to two alloys was adequate as indicated by its cohesive failure. Simultaneous firing of this special opaque porcelain and body porcelain produced satisfactory sintering, strength, and bonding to metal. (J PROSTHET DENT 1994;72:414-9.)

Porcelain-fused-to-metal restorations are fabricated from at least three porcelains to form an esthetic layered structure. An opaque porcelain is applied to the metal to mask the metal oxide color. After the opaque porcelain is fired, subsequent layers of body (gingival) and incisal porcelain are added for a minimum of three firings. Because each firing and cooling takes 20 to 25 minutes, the process is time-consuming and labor intensive. An opaque porcelain is now available that does not require a separate firing before application of the body porcelain (No-Bake Opaque, Excelco International Inc., Deerfield Beach, Fla.). The technician applies the No-Bake Opaque porcelain to the metal and then builds up the body porcelain layer and fires them both together. Although this procedure only saves approximately 20 to 25 minutes, the savings for the millions of these crowns made annually would be considerable. This study determined the properties of a new opaque porcelain that does not require a separate firing and its ability to bond to metal.

MATERIAL AND METHODS

No-Bake Opaque porcelain (shade A3, lot No. 1791) was used throughout this study. A standard opaque porcelain (Brush-O-Paque, DB-A4, lot No. 8015050, Excelco International Inc.) was used when comparisons were made. The body porcelain used was Gingival DG-A4 porcelain (lot No. 8012030, Excelco International Inc.). The two alloys used were Jelenko Olympia (JF Jelenko, Armonk, N.Y.) and Will-Ceram W1 (Williams Dental, Amherst, N.Y.) alloys.

Flexural strength

The flexural strength in three-point bending was determined[1] for six specimens. The bars were prepared by pressing the powder into a steel mold (25 mm × 5 mm × 2 mm) with a load of 500 kg and firing to 1800° F (982° F). A tensile testing apparatus was used to fracture the samples (Model HTC, John Chatillon & Sons, New York, N.Y.). The flexural strength of five conventional opaque porcelain samples (Brush-O-Paque, Excelco International Inc.) was also measured for comparison. A statistical evaluation of the means of these two porcelains was performed according to Student's *t*-test with two-tailed *p* value.

Sintering

The degree of densification caused by sintering was determined by the change in linear firing shrinkage as a function of temperature and by scanning electron microscope (SEM) analysis (1000B, AmRay Inc., Bedford, Mass.).

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Rectangular bars were prepared by pressing the powder into a steel mold (25 mm × 5 mm × 2 mm) with a load of 500 kg. Three bars were fired at each of the following temperatures: 1360° F (738° C), 1460° F (793° C), 1560° F (849° C), 1660° F (904° C), and 1760° F (960° C). The linear firing shrinkage was estimated by measuring the change in dimensions of the tablets produced by sintering. The change in linear firing shrinkage *(LS)* was calculated by use of the following formula

$$LS = \frac{Lm - Ls}{Lm} \times 100$$

where *Lm* is the length of the mold cavity, and *Ls* is the length of the sample after firing.[1] A one-way analysis of variance was computed with respect to the effect that firing temperature had on linear shrinkage. Paired comparisons were run with simultaneous confidence intervals.

Bonding

Bonding to body porcelain. The bonding between the No-Bake Opaque and body (Excelco International Inc.) porcelains was assessed on layered tablets prepared in a steel mold (9.5 mm in diameter). A dual layered tablet was made by pressing moistened body porcelain in the mold with a load of 500 kg to produce a 2 mm thick layer; the plunger was removed, and moistened No-Bake Opaque porcelain was added to the mold. The plunger was placed in the mold, and the No-Bake Opaque porcelain was pressed on top of the body porcelain with a load of 500 kg to produce a 2 mm thick layer. Firing was carried out at 1460° F (793° C), 1560° F (849° C), 1660° F (904° C), and 1760° F (960° C), followed by fracture in diametral tension to obtain a fracture perpendicular to the interface. This fractured surface was examined with an SEM to qualitatively observe the nature of the interface between the two porcelains and determine if any voids or porosity were present or whether the bond was continuous along this interface.

Bonding to metals. Alloy bonding samples were prepared by applying a thin layer of No-Bake Opaque porcelain to six degassed flags (16 mm × 13 mm × 1 mm) of two commercial alloys. Unfired tablets (9.5 mm in diameter and 4 mm thick) of the body porcelain were then placed on top of the No-Bake Opaque porcelain, and the specimens were fired to 1800° F (982° C). The cooled samples were tested for bonding with the use of a drop ball tester (Model 170041, American Optical Corp, Southbridge, Mass.). The porcelain disks were fractured by a falling steel ball, and the interfacial fractures were examined with an SEM and classified according to the fracture surfaces formed.[2]

Coefficient of thermal expansion

The coefficient of thermal expansion of No-Bake Opaque porcelain was measured on four rectangular bars (6 mm × 6 mm × 51 mm) with a single-push dilatometer (Model TDA-H1-MP6, Harrop Laboratories, Columbus, Ohio). The dilatometer was calibrated on the cooling curve with an alumina standard. The coefficient of thermal expansion value was determined with the cooling curve between 25° and 500° C with a heating and cooling rate of 4° C/min. The coefficient of thermal expansion of the body porcelain was also measured for comparison. A statistical evaluation of the means of these two porcelains was performed according to Student's *t*-test with two-tailed *p* value.

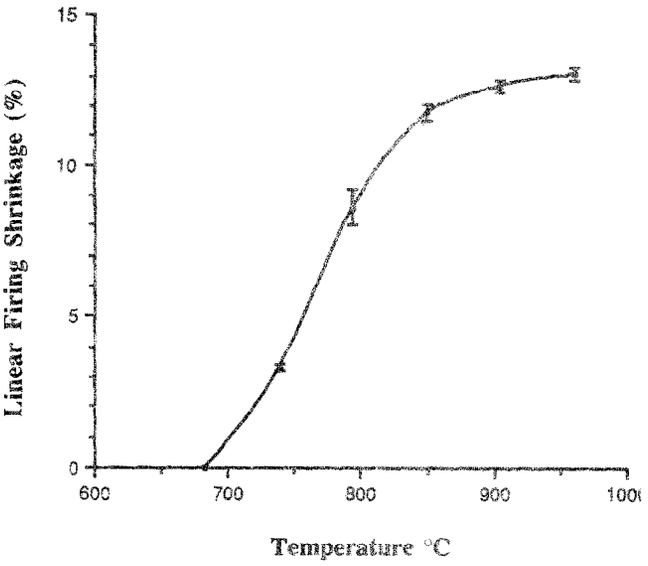

Fig. 1. Linear firing shrinkage as function of temperature for No-Bake Opaque porcelain.

Particle size analysis

The particle size distribution of No-Bake Opaque porcelain was determined by measuring the sedimentation rate of the powder in a solution of 0.05% tetrasodium pyrophosphate (Huron Valley Steel, Trenton, Mich.) in distilled water by means of a particle size analyzer (Sedigraph 5000D, Micromeritics Instrument Corp, Norcross, Ga.). The particle size distribution of a conventional opaque porcelain was also measured for comparison. The Kolmogorov-Smirnov two-sample two-tailed test with 70 intervals was used to test for statistical differences between the distributions.

RESULTS

Flexural strength

The flexural strengths were 99 ± 7 MPa for No-Bake Opaque and 101 ± 8 MPa for the conventional opaque porcelain and were not statistically different according to Student's *t*-test (t statistic = −0.623, p = 0.548). These are excellent results for an opaque porcelain, well above the International Organization for Standardization (ISO) requirement[1] of 55 MPa.

Sintering

The relationship between firing temperature and linear firing shrinkage is shown in Fig. 1 and Tables I and II. An

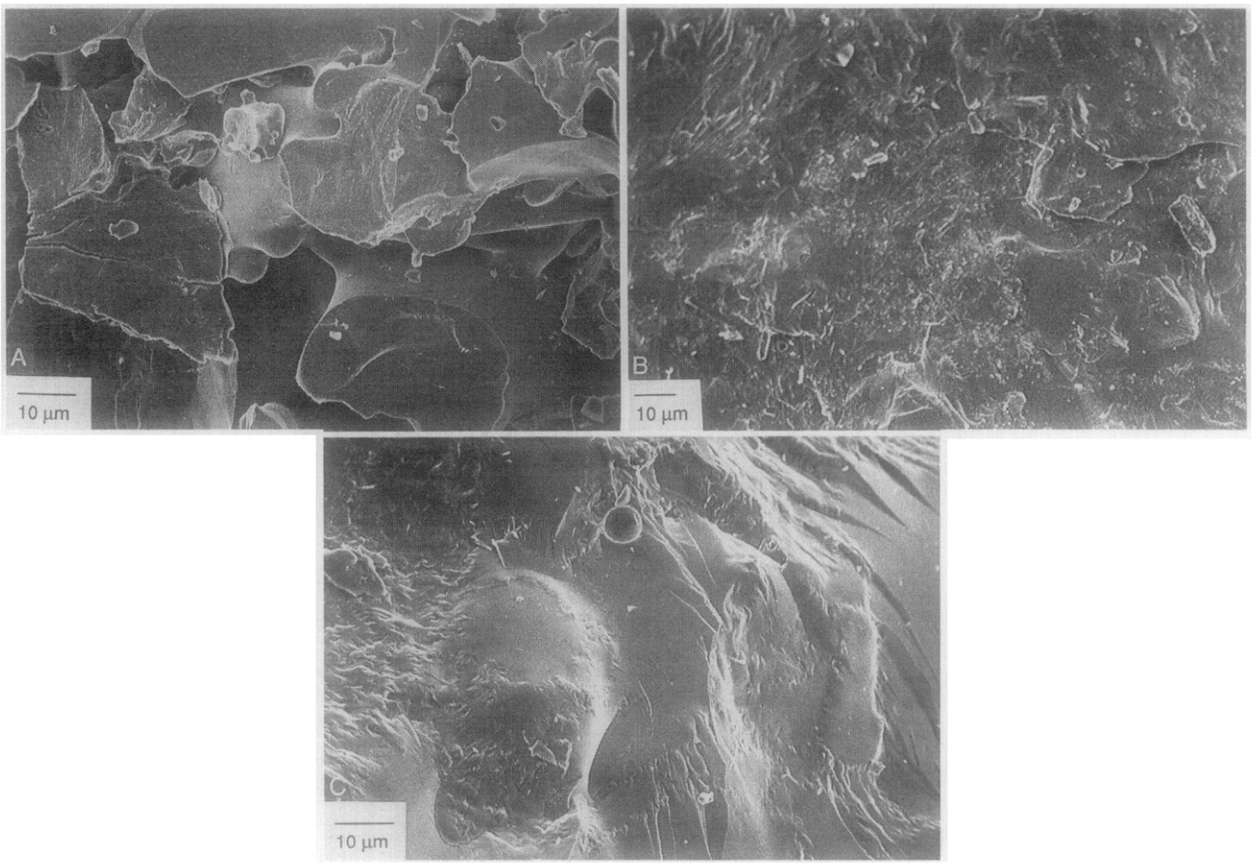

Fig. 2. SEM photomicrographs of No-Bake Opaque porcelain fracture sintered at **(A)** 1560° F (849° C), **(B)** 1660° F (904° C), and **(C)** 1760° F (960° C).

Table I. Relationship between firing temperature and linear shrinkage for No-Bake Opaque porcelain

Firing temperature (°F)	Linear firing shrinkage (%)	
	Mean	SD
1360	3.3	0.08
1460	8.6	0.56
1560	11.8	0.31
1660	12.7	0.15
1760	13.1	0.20

Groups joined by vertical lines are not significantly different with Scheffé F-test at the 95% confidence level

Table II. Statistical analysis

Source	One-way analysis of variance				
	df	Sum of squares	Mean square	F-test	*p* Value
Between groups	4	154.5	38.6	369.7	0.0001
Within groups	9	0.941	0.105		
Total	13	155.5			

analysis of variance showed that a significant effect of temperature occurred on linear firing shrinkage ($p < 0.0001$). No significant difference occurred in linear shrinkage between the temperatures of 1560° F (849° C) and 1660° F (904° C) or between 1660° F (904° C) and 1760° F (960° C). The linear firing shrinkages at the latter two temperatures were 12.7% ± 0.2% and 13.1% ± 0.2%, respectively, which is well within the ISO limit[1] of 16% max. Fig. 2 illustrates representative SEM photomicrographs of the No-Bake Opaque porcelain fired at 1560° F (849° C), 1660° F (904° C), and 1760° F (960° C). The No-Bake Opaque porcelain appears to be fully sintered at 1760° F (960° C) (Fig. 2, *C*).

Bonding

Fig. 3 shows representative SEM photomicrographs of No-Bake Opaque/body porcelain interfaces produced by firing at 1560° F (849° C), 1660° F (904° C), and 1760° F (960° C). These photomicrographs show that the bonding of the No-Bake Opaque porcelain to the body porcelain was nearly complete at 1660° F (904° C) and was completed by 1760° F (960° C) (Fig. 3, *C*).

Photomicrographs of the fractured samples of No-Bake Opaque porcelain and tablets of body porcelain fired on

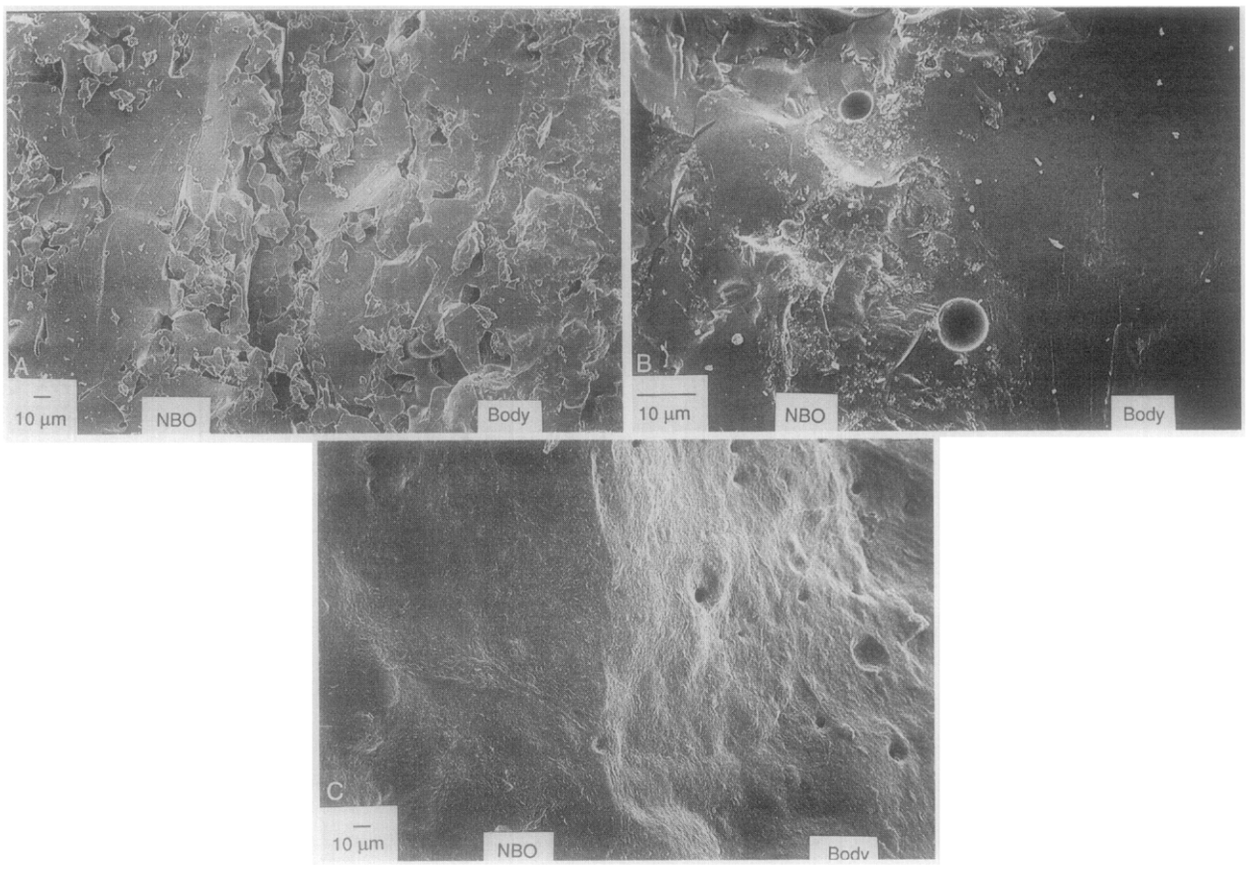

Fig. 3. SEM photomicrographs of No-Bake Opaque *(NBO)*/body porcelain interface produced by firing at **(A)** 1560° F (849° C), **(B)** 1660° F (904° C), and **(C)** 1760° F (960° C).

Will-Ceram W1 and Jelenko Olympia alloys are shown in Fig. 4. It appeared that the bonding failure was mostly cohesive within the porcelain as evidenced by the porosity that is typical of these porcelains. Also, smaller areas of failure may exist within the porcelain-metal oxide diffusion zone or within the metal oxide as evidenced by the smoother regions at some locations along the periphery.

Coefficient of thermal expansion

The coefficient of thermal expansion was $13.3 \pm 0.2 \times 10^{-6}$/° C (between 25° and 500° C) for No-Bake Opaque porcelain and $13.6 \pm 0.2 \times 10^{-6}$/°C (between 25° and 500° C) for the body porcelain. Student's t-test showed that no significant difference existed between these two porcelains (t statistic −1.667, p 0.147).

Particle size analysis

A particle size analysis found that 33.5% by weight of No-Bake Opaque particles were under 5 μm, compared with 20.6% by weight for the conventional opaque. No-Bake Opaque porcelain also had a small percentage of particles over 50 μm, which was not found with the conventional opaque porcelain (Table III). Fig. 5 shows the particle size distribution of No-Bake Opaque and Brush-O-Paque porcelains. These distributions were significantly different according to the Kolmogorov-Smirnov two-sample two-tailed test (maximum difference 0.329, p 0.05).

Table III. Particle size analysis of No-Bake Opaque and Brush-O-Paque porcelains

Equivalent spherical diameter	Cumulative mass (%)	
	No-Bake Opaque	Brush-O-Paque
<5 μm	33.5	20.6
<10 μm	49.5	38.2
<15 μm	57.0	49.5
<20 μm	63.1	60.0
<25 μm	68.2	69.5
<30 μm	74.5	77.0
<35 μm	78.5	84.2
<40 μm	83.5	91.2
<45 μm	88.0	96.5
<50 μm	91.5	100.0
<55 μm	94.5	100.0
<60 μm	96.2	100.0
<65 μm	97.5	100.0
<70 μm	98.4	100.0

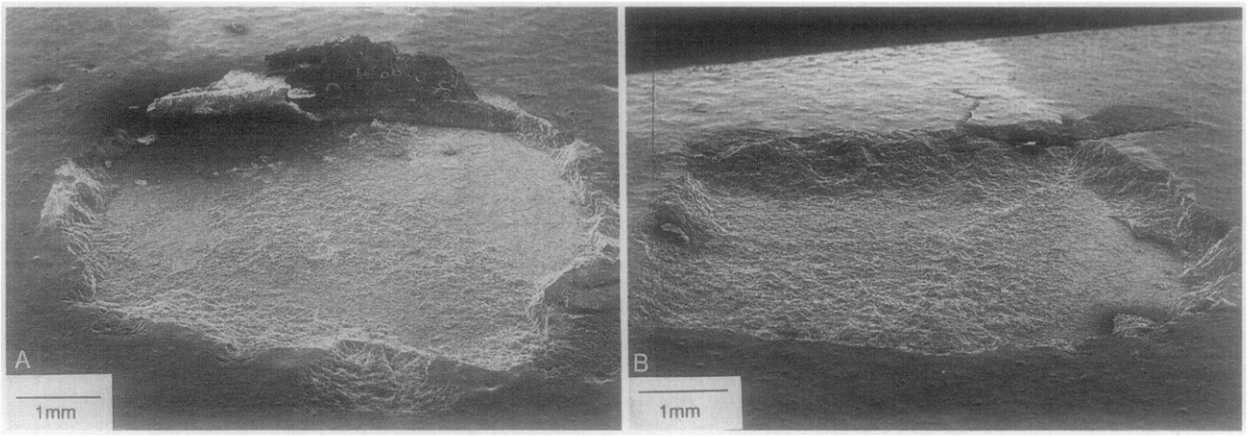

Fig. 4. SEM photomicrographs of No-Bake Opaque alloy fracture interface showing cohesive failure for **(A)** Will-Ceram W1 and **(B)** Jelenko Olympia alloys. Bonding failure was mostly cohesive within porcelain as evidenced by porosity that is typical of these porcelains. Smaller areas of failure may exist within porcelain-metal oxide diffusion zone or within metal oxide as evidenced by smoother regions at some locations along periphery.

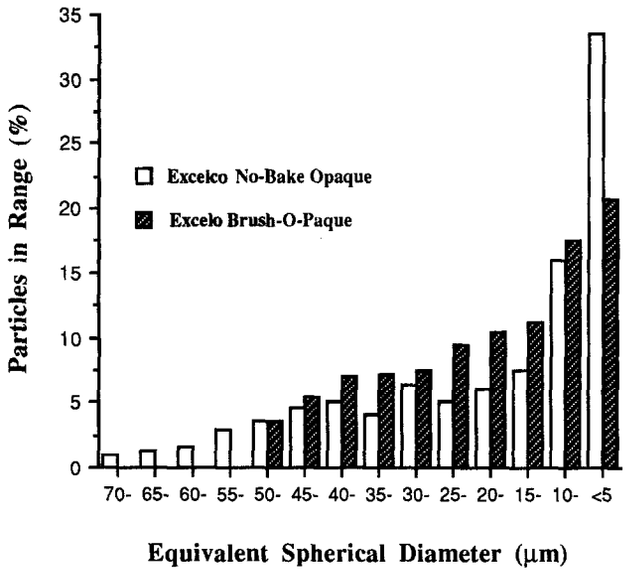

Fig. 5. Particle size distribution of No-Bake Opaque and Brush-O-Paque porcelains.

DISCUSSION

An opaque porcelain is used in metal-ceramic restorations to mask the color of the alloy, to provide the basis for the internal color of the restoration, and to bond the metal and the porcelain.[3] Usually two thin applications of opaque porcelain are recommended.[4-6] The No-Bake Opaque porcelain does not require a separate firing before application of the body porcelain. Therefore, it would save the dental technician the time required for the two opaque firing cycles and for the application of the second opaque layer during the preparation of a porcelain-fused-to-metal restoration.

The flexural strength of No-Bake Opaque porcelain was not statistically different when compared with that of a conventional opaque porcelain and was well above the ISO requirement[1] of 55 MPa. This opaque porcelain appeared to be fully sintered at 1760° F (960° C), and the linear firing shrinkage was well within the ISO limit[1] of 16% max. Photomicrographs showed that the bonding of the No-Bake Opaque porcelain to the body porcelain was complete when fired at 1760° F (960° C). Bonding of this opaque porcelain to a palladium-silver alloy and a gold-palladium (silver-free) alloy was adequate as indicated by the cohesive failure of the opaque when examined by SEM. No significant difference occurred between the coefficient of thermal expansion for this opaque porcelain and the body porcelain manufactured to be used with it. A particle size analysis found 33.5% less than 5 μm and 3.8% greater than 50 μm for the opaque porcelain evaluated in this study. Opaque porcelains should consist of particles of several different sizes.[3] A fine grain and variation in grain size of the porcelain powder will improve packing, giving the material a high green or wet strength, will reduce shrinkage, and will have an effect on sintering rates[6, 7] because smaller particles undergo fusion more rapidly.[8]

Additional studies on its performance in general production would be useful.

SUMMARY

Simultaneous firing of this special opaque porcelain and body porcelain produced satisfactory sintering, strength, and bonding to metal. The linear firing shrinkage and the flexural strength of this porcelain were well within the ISO requirements for dental ceramics.[1]

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