Dentistry
ISSN: 2161-1122

Vaderhobli and Saha, Dentistry 2015, 5:7
DOI: 10.4172/2161-1122.1000316

Research Article Open Access

Microwave Sintering of Ceramics for Dentistry: Part 2

R Vaderhobli[1]* and S Saha[2]

[1]Department of Preventive and Restorative Dentistry, UCSF School of Dentistry, San Francisco, CA 94143, USA
[2]Department of Orthopaedic Surgery and Rehabilitation Medicine, SUNY-Downstate Medical Center, Brooklyn, NY 11203, USA

Abstract

Fourteen dental copings (zirconia stabilized with 3 mol% yttria) were layered with Noritake dental glass-ceramic and sintered in the microwave furnace with various sintering temperatures. Sintered zirconia rectangular beams measuring 50 mm in length, 4 mm in width and 0.75 mm in height were coated with the dental glass-ceramic and sintered in the microwave furnace. These were subjected to a four-point bend test to calculate modulus of rupture. Sintering temperature of 800°C in the microwave furnace with ramp rates in excess of 100°C per minute was sufficient to attain good sintered crowns. Indentation hardness with 200g loads and 500g loads for the microwave-sintered teeth resulted in hardness values of 0.685 ± 0.0245 GPa and 6.56 ± 0.4 GPa. The indentation fracture toughness values with 200g and 500g were calculated to be 2.26 ± 0.8 MPa(m)$^{0.5}$ and 0.97 ± 0.1 MPa(m)$^{0.5}$ respectively and agreed well with the published values [1]. The failure load for the layered beams in the bend test was 81.8 ± 17.7 N and the resulting modulus of rupture was 632 ± 105 MPa.

Keywords: Microwave sintering; Dental copings; Dental ceramics

Introduction

In our previous paper we attempted to explain the principles of microwave heating or sintering when compared to traditional conventional sintering methods. The next step was to explore the feasibility of making an actual sintered/processed restoration in the microwave oven. Dental porcelains are glass powders with a particle size varying from 1-80 microns. They essentially consist of vitreous (glass-like) silica, which is chemically modified to yield improved mechanical and chemical properties. The introduction of Zirconia as a dental biomaterial became very popular as a core material on which porcelain could be enamelled and sintered. It has remarkable strength (around 900 MPa) and high fracture toughness (6-9 MPa.m$^{0.5}$). This translucent ceramic (sintered) offers a bio-compatible and aesthetic solution with maximum reliability for all crown and bridge restorations [2]. Utilizing the similar MHH principle, the effects of fast and slow sintering of the porcelain powders on the zirconia copings and comparing the post-sintering properties with conventionally sintered crowns and to the published values were explored in this work. Our results may act as a basis for obtaining ideal microwave time-temperature-power profiles to standardize further investigations.

Materials and Methods

A multi-magnetron 2 kW research microwave oven having a stainless steel cavity and equipped with dual mode stirrers, a platinum sheated S-type thermocouple and Omega controller was procured from Microwave Research and Applications, INC (Model number BP 210/211) for the microwave sintering experiments. The operating frequency was 2.45 GHz. A thermal and a fibrous alumina cylinder containment box was made using fibrous alumina boards one and a half inches thick. Hybrid microwave heating was employed as the method of sintering with three Silicon carbide rings (25 mm diameter) as preheaters. These were sandwiched between the reticulated zirconia plates which also acted as the floor upon which the samples rested.

Six anterior (canines) and ten posterior (molars) sintered zirconia stabilized with 3 mol % yttria copings were obtained from Town and Country Dental Laboratory (Freeport, NY) and Nobel Biocare (Yorba Linda, CA) respectively. Town and Country Dental laboratory also supplied the dental glass-ceramic (Noritake, Japan) which was the base, dentine, enamel and the luster powders that was compatible with the ceramic copings. The mixing ratio was one part of powder to one part of the forming liquid and was mixed and applied to the coping as per manufacturer's instructions.

Each tooth was then placed upon the reticulated zirconia in the thermal containment box and the technique of Microwave Hybrid Heating (MHH) was applied. The temperature measurements were taken every two minutes. We attempted to sinter the teeth in the microwave furnace with both fast and gradual heating rate (per minute) by manipulating the power rate. The power was increased gradually for few samples and for some samples, the power was increased rapidly (rapid bursts). The sintering temperature was 800°C with a hold of one minute. However, one tooth was fired to approximately 700°C with a hold of one minute. Two teeth samples (anterior) were sintered with rapid bursts of power. The cooling temperature profile was recorded every two minutes. Accordingly, the posterior tooth copings were enameled with the layers of the dental glass-ceramic. They were fired with the power-temperature profile that worked well with the anterior teeth copings. Two individual teeth were utilized in each run with the ideal power-profile. The remaining six tooth samples were placed one at a time on the reticulated zirconia in the thermal containment box and fired according to the gradual power-time profile to approximately 800°C with a thermal time histrory as depicted in Figure 1. Once the samples were sintered, they were removed from the furnace, bagged and numbered for further measurements.

Two posterior teeth samples layered with the dental glass-ceramic were sintered in the conventional furnace (Dentsply Ceramco, PA) one at a time as per manufacturer's instructions and fired incrementally with each layer. Yttria stabilized sintered zirconia substrates were obtained from Coors Tek (Golden, Colorado) measuring 50 mm (length) × 4 mm (breadth) × 0.71 mm (thickness) with parallel edges. The layers of dental ceramic, shade base, dentine, enamel and the luster shades

***Corresponding author:** R. Vaderhobli, 707 Parnassus Avenue, Room D 1028, San Francisco, CA 94143-0758, USA, Tel: 415-476-4469; Fax: 415-476-0858; E-mail: ram.vaderhobli@ucsf.edu

Received May 27, 2015; **Accepted** June 24, 2015; **Published** June 29, 2015

Citation: Vaderhobli R, Saha S (2015) Microwave Sintering of Ceramics for Dentistry: Part 2. Dentistry 5: 316. doi:10.4172/2161-1122.1000316

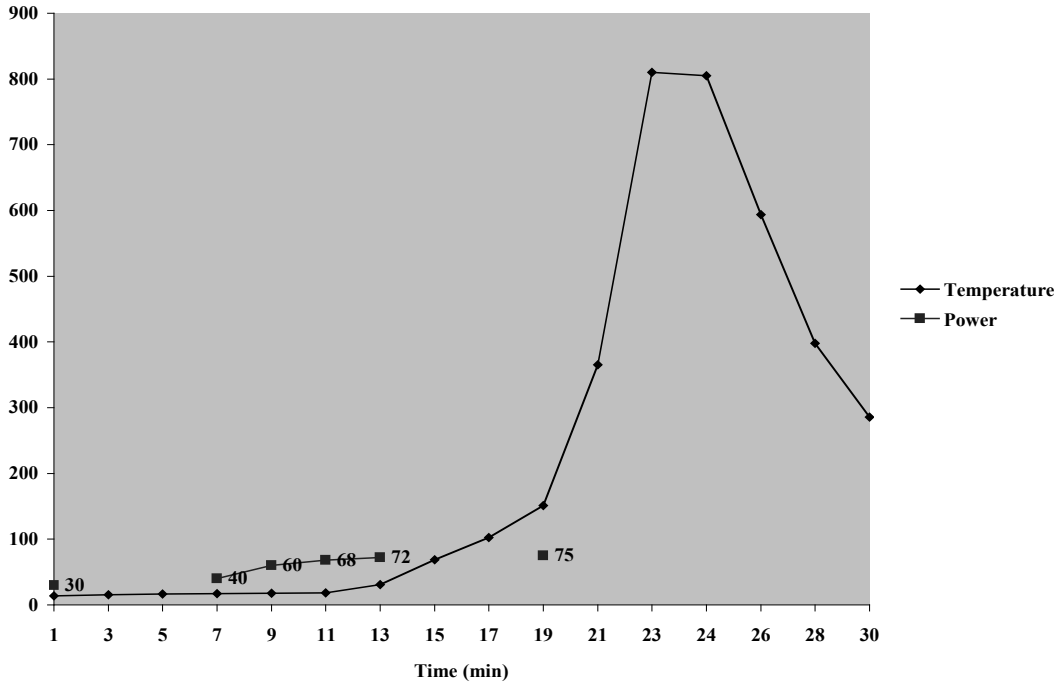

Figure 1: Time-temperature-power profile that was found to be ideal to obtain best mechanical and microstructural properties in the sintered dental crowns. The horizontal asis indicates time in minutes and the vertical axis the temperature in °C.

were layered or enameled onto the zirconia substrates. Twenty such substrates were coated; ten for conventional and ten for microwave sintering. The total thickness of the coating was 0.5-0.8 mm. The ideal firing schedule utilized for sintering teeth was utilized for microwave sintering of these substrates. The power was increased as dictated by the temperature requirement. The remaining ten samples were sintered in the conventional furnace. Thus, the layered beams were formed with layers of veneering porcelain and core materials.

A Vickers micro-hardness indenter (Micromet-II) was used to measure the Vickers Indentation number and the Indentation fracture toughness. The diamond indenter with maximum load of 200 grams was utilized to indent five teeth samples and the indenter was held in position for a period of ten seconds. Three teeth samples were indented with a load of 500 grams and held in position for a period of ten seconds. When a good indentation mark was made, the software (Image Pro) was utilized to capture the image. The mean diagonal length and the crack length were measured using the software. Subsequently the Vickers hardness number and the Indentation fracture toughness were calculated.

The dental glass-ceramic powders were analyzed using x-ray diffraction (XRD,Siemens D500) 40 kV of accelerating potential, 30 mA current. A 2-theta angle scanning range was set for 10°-70°, with a step size of 0.04° and a dwell time of 10 seconds. The spectral peaks obtained were analyzed using the JADE computer program. XRD data of the microwave-sintered tooth was obtained using a Siemens (Kristalloflex 810) low angle reflectometer. A 2-theta angle scanning range was set for 10°-150°.

The specimens were loaded in a four-point bend test fixture (with the coated surface loaded in tensile direction) and tested on a universal testing machine (Instron Corp., Canton, Massachusetts, model 8532) with a crosshead speed of 0.5 mm per minute. Four-point bend test was also conducted on three zirconia substrates without the coating for comparisons between the results. The flexure strength or the modulus of rupture was calculated by using the following formula:

$$\text{Flexure Strength } (S) = 3WI/2bd^2$$

Where I pertains to the distance between the supports; b is the width of the specimens; d is the depth or thickness of the specimen and W is the maximal load before fracture.

Results

The temperature-power-time profile as depicted in Figure 1 shows the initial ramp rate to be around 40°C per minute. At 200°C the power was increased and a ramp rate, in excess of 270°C per minute allowed the sample to achieve the sintering temperature. A dwell period of one minute was required. The temperature remained around 800°C for the stipulated one minute, and the microwave power was turned off. The cooling rates were also not controlled; but were approximately in the range of 110°C per minute. The entire sintering process was completed in less than 25 minutes. The restoration exhibited an excellent finish with natural translucent surface.

The thermal schedule for the sample sintered at 700°C follows the ideal schedule with a pre-heating period of fifteen minutes to evaporate the water in the layered restoration. Sixty eight percent power was sufficient to achieve a temperature up to 700°C with a dwell of one minute and the microwave switched off after the dwell period. The cooling rates were similar to prior schedules. The restorations were removed when the temperature was below 150°C. The finished restorations, when compared to the earlier firing schedule, did not have a good finish and was porous in appearance. For fastest sintering, a maximum power at 75 percent was used. There was no preheat period and the sintering temperature was 800°C. The sintering temperature was achieved within five minutes with a ramp rate of more than 275°C

per minute. The cooling rate was 100°C per minute and the samples exhibited improper sintering with very rough surfaces.

For the posterior teeth, power was raised to 75% in order to increase the temperature after the initial pre-heat or soak period was completed. This was required due to the larger load that required more power. The maximum temperature was around 810°C, and over a period of twenty seconds the temperature dropped to 798°C. The microwave was switched off after the one-minute dwell period. All the restorations were completely sintered and had an excellent translucent look as shown in Figure 2. The use of faster sintering in the conventional furnace resulted in cracking and porosity of the samples. The standard procedure of slow heating these dental crowns was followed and this took approximately 30 minutes for the application of the basic shades.

Leucite crystals ($KAlSi_2O_6$), wollastonite ($CaSiO_3$) and calcium aluminum silicate ($CaAlSiO_3$) were precipitated in the glassy matrix of the material (some elongated and some circular) as determined by X-ray Diffraction. The crystals were dispersed in the glass matrix as shown in Figure 3A and averaged 10-15 µm in size**.** The SEM picture of the tooth sample fired by rapid power is also shown in Figure 3B and the crystals may be identified being dispersed in the uneven glassy matrix. Few crystals can be seen protruding from the glassy matrix.

The XRD patterns matched well with the PDF #06-0266 and #38-1423 indicating that the glass-ceramic was probably analogous to a Leucite and Zircon tetragonal phase.

Indentation data were obtained from at least three specimens (eighteen indents) for the ideal microwave sintering profile, two specimens for the conventionally sintered sample (nine indents), and one tooth from the rapidly fired group (five indents). There was

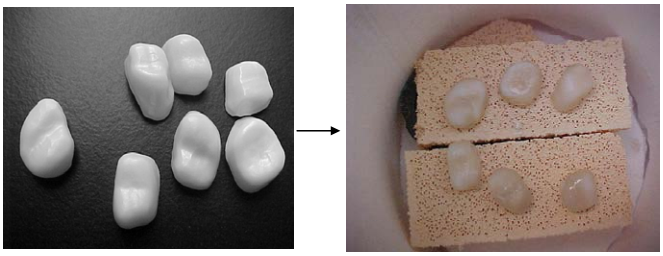

Figure 2: Using the ideal sintering profile the presintered copings (left) were sintered and restorations with optimum esthetics and completed sintering were obtained (right).

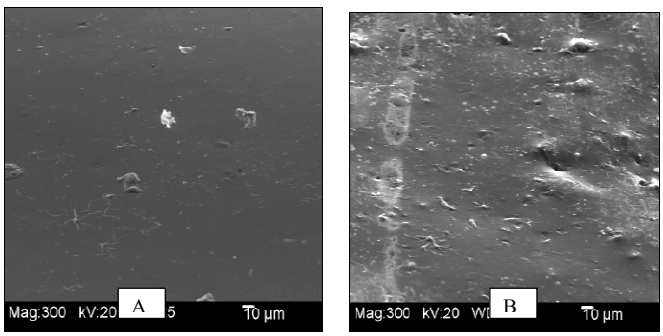

Figure 3: The figure A depicts the glassy microstructure of samples sintered according to the ideal thermal profile and when faster sintering were attempted in both conventional and microwave furnace there was a distinctively poor glass phase with improper crystal precipitation.

Materials	VHN (500 g load) ± S.D.
Ceramco	5.71 ± 0.23
Vita VMK	5.74 ± 0.11
Excelso	5.64 ± 0.20
Will-Ceram	5.84 ± 0.20
Vitadur-N	6.62 ± 0.58
Biodent-NBK100	6.66 ± 0.74
M/W Sintered Sample	6.56 ± 0.4

Table 1: Hardness Values for the teeth sampes sintered by M/W at VHN 500 g compared well with and available published values at the same load.

little difference between these values for the microwave sintered teeth samples with the ideal thermal profile and the conventionally sintered samples. The indentation hardness with 200g load resulted in hardness values of 0.685 ± 0.0245 GPa. The indentation hardness number was comparable with those published in the literature [3]. Based on twelve indents at a load of 500g, the Vickers hardness number as calculated was 6.56 ± 0.4 GPa. This agreed with the published values for other dental glass-ceramics as shown in Table 1 [3].

The fracture toughness value for the microwave-sintered samples was 2.26 ± 0.08 $MPa(m)^0$.

The modulus of rupture values obtained from the microwave-sintered beams were compared with the conventionally sintered beams as published in the literature and shown in Figure 4 [4-6]. The reference ceramic materials used were ICZ (Inceram Zirconia), IC (Inceram Alumina), and ICS (Inceram Magnesium Aluminum Spinel). The modulus of rupture (MOR) for the beams without the coatings were strongest and they were 2659 ± 413 MPa (n=3) when compared to the microwave-sintered layered beams which were 632 ± 105 MPa (n=6).

Discussions

Strict adherence on clinical procedures and cooperation by the dentist and the technician is vital in creating beautiful tooth restorations [7-9]. In the conventional method of firing of layered dental ceramics over the copings, the sintering temperatures are usually 900-960°C with dwell times of three minutes. The primary reason for using microwave processing technology was to use lower temperatures and for a shorter period. In addition, the ability to fire many crowns at once was advantageous. Microwave sintering of zirconia at high temperatures enabled a protocol with an ideal power-temperature profile for sintering teeth in the microwave furnace. An initial soak period at low temperatures ensured that the water in the layered dental ceramic was removed. This procedure can be compared to conventional methods, where copings with layered dental ceramic are placed in the muffle of a pre-heated furnace for few minutes before they are placed inside to remove water [10]. Purpose of the firing was to ensure the fusion of the powders to completely form the restoration. The method of cooling of the porcelain restorations from firing temperatures to room temperature has been the subject of considerable controversy [11-13]. Cooling rates in excess of 100°C per minute had no deleterious effect (cracking) on the sintered restorations. This was one of the major advantages for the use of microwave sintering in making these restorations. Literature reports that some chemical reactions do occur at prolonged firing times and this is of importance if the dental glass-ceramic has more of the leucite content [14-19]. If the temperature had been raised too quickly before the layers of the ceramic vitrified in a short span of time, then complete sintering could not be accomplished and would ultimately lead to cloudiness in the completed restorations. This was one of the main reasons for the failure of sintering with high power (around 75%). The attempt of completing

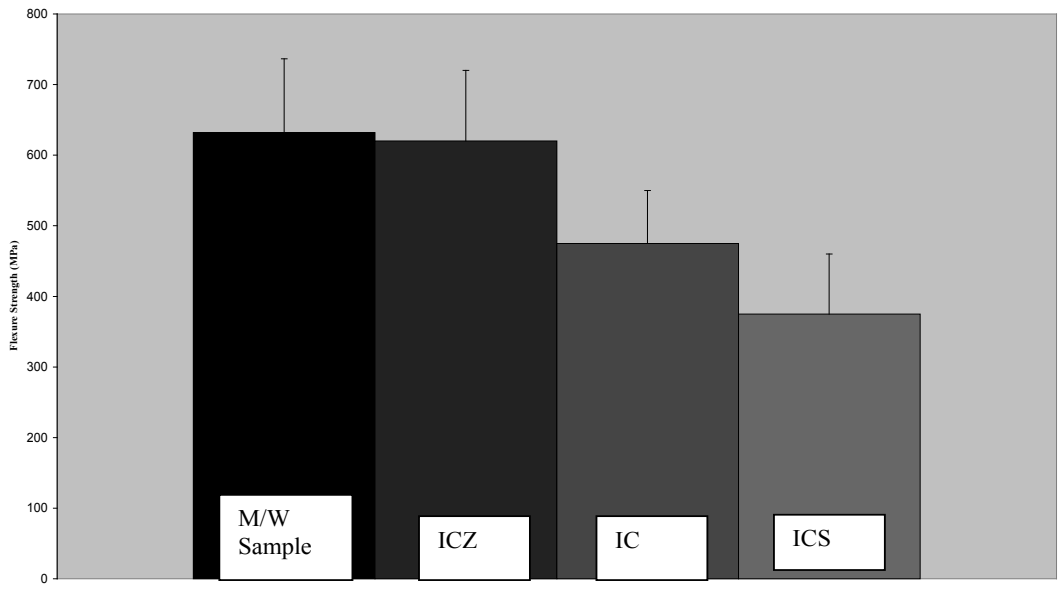

Figure 4: The modulus of Rupture for the M/W sintered samples were 632 ± 105MPa and were compared against the published values of Inceram Zirconia (ICZ), Inceram Alumina (IC) and Inceram Magnesium Aluminum Spinel (ICS).

these thermochemical reactions without the dwell periods was unsuccessful and we concluded that thermochemical reactions have to be completed to allow for complete vitrification [16,20]. As a part of the observable variable, the aesthetic properties were visually assessed and were found to be excellent. Faster firing in the conventional oven (to mimic the microwave firing parameters) resulted in cracking of the samples owing to the generation of thermal stresses. Literature reports conversion of glass to a partially crystalline glass as creaming [4,10,18]. However, for the microwave sintering if larger amounts of crystals are to be formed, dwell periods have to be increased from one to two minutes. The poor microstructure for samples sintered without the dwell periods or a faster sintering (power in excess of 70%) can be considered to be the result of incomplete precipitation of the crystals that were entrapped within the glassy matrix due to the rapid sintering which resulted in insufficient time for complete precipitation.

The edges of the indents for the porcelain were not as sharp as those seen on the zirconia cylinders. This may be due to the high indent loads used and the sample preparation that resulted in well-defined indents. The cracks were also straighter and longer suggesting a different structure and even greater brittleness for this material. As a result of using the Image Pro software program to measure the flaw sizes, the values obtained were accurate. The indentation hardness and the fracture toughness values obtained compared well with published values [3].

Important test parameters for the determination of the strength of brittle dental materials by flexural testing are the specimen thickness, the contact zone at loading, the homogeneity and porosity of the material, and the loading rate [21]. ISO standards (ISO 6872, 1995) are used by many researchers and require a three-point or a four-point bending test for the evaluation of the modulus of rupture for dental porcelain. Tensile failures of crowns made of zirconia layered with porcelain have been clinically observed [5]. The beams, which had the weakest material, Noritake dental glass-ceramic on the tensile surface and the strongest material, Zirconia, on, the compressive surface tended to undergo failure initially on their tensile surfaces, and then underwent partial delamination prior to a secondary failure of the stronger zirconia substrate. This type of failure occurred in all microwave-sintered samples. The conventionally sintered beam samples exhibited porosity, insufficient coating thickness, delamination during the testing and these data were not included in this comparative study. However, the modulus of rupture values obtained from the microwave-sintered beams was compared with the conventionally sintered beams as reported in the literature [4-6].

It is desirable to build an ideal microwave furnace suited for the dental laboratory or the office which has a vacuum capability, an optical pyrometer for temperature measurements and feedback power control which automatically increases the power when the temperature begins to drop. Dentists and dental technicians need to be aware of the rapidly changing field of dental ceramic processing technology. Based on this work of microwave sintering of all ceramic crowns, the sintering technology could be expanded further to porcelain-fused-to-metal crowns. We hope that based on our work, new industries related to microwave sintering specially for dental ceramics would be setup in the future.

Acknowledgements

Our special thanks to Coors Tek (Golden, CO) and Chand Kare (Boston, MA) for providing and machining the zirconia substrates which were used for the flexure testing. We are indebted to Town and Country Dental Laboratory (Freeport, NY) and Nobel Biocare (Yorba Linda, CA) for providing us the copings.

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