Effect of Conventional and Microwave Glazing on Surface Roughness of Metal Ceramics: An Atomic Force Microscopy Analysis

Abstract

Purpose: This study investigated and compared the surface roughness achieved by glazing porcelain samples in a conventional and a microwave oven. Materials and Methods: Two commercial brands of metal ceramics were used, VITA VMK MASTER and IPS CLASSIC. Sixty samples were fabricated, 30 for each type of ceramic. The samples were sintered in the conventional oven and hand-polished to remove any irregularities. Samples (n = 10) from each type of ceramic were further divided into three groups as follows: hand-polished (Group A), conventional oven glazed (Group B), and microwave glazed (Group C). Each specimen was evaluated for surface roughness by atomic force microscope. Data were statistically analyzed using two-way analysis of variance (ANOVA) and Tukey’s post hoc test (α = 0.05). Results: Two-way ANOVA indicated a highly significant difference in surface roughness based on the type of glazing (P < 0.001), there was a significant difference based on the metal ceramics (P = 0.002). There was also a significant interaction between the type of glazing and metal ceramics (P = 0.009). The images obtained from the atomic force microscope corroborated the measured values. Conclusions: All the results indicate that microwave glazing can be a feasible option for glazing porcelain specimens. It was concluded that surface topography is influenced by surface treatment and microwave glazed ceramic is superior to conventional oven glazed ceramic and hand-polishing showed greater surface roughness when compared to glazing. IPS CLASSIC ceramic showed relatively smooth surface when compared to VITA VMK MASTER irrespective of the surface treatment.

Keywords: Atomic force microscope, dental ceramic, glazing, microwave sintering, surface roughness

Introduction

Over the past four decades, there has been a technological evolution in the field of dental ceramics as new materials and processing methods are being introduced steadily. An improvement in their properties has made it possible to expand their range of indications from long-span fixed partial prostheses to implant abutments. One of the most common fabrication techniques for dental ceramics is sintering. Sintering is the process of heating the ceramic to ensure densification. Sintering of dental ceramic in a standard dental conduction oven is not a uniform heating process. Heat is applied to the surface of the ceramic and reaches the core by thermal conduction. This results in the production of high-temperature gradients and stress within the ceramic material.[1]

High sintering temperatures are said to achieve greater densification but result in increased sintering time. The high sintering temperature and long sintering time required for the consolidation of ceramic powders often result in extreme grain coarsening and decomposition of the ceramic causing deterioration of the mechanical properties of ceramics. These factors potentially produce an inferior product with higher failure rates.[2] Microwave processing has emerged as a unique sintering method for many conventional ceramics. It has been found that microwave sintering has several benefits over conventional sintering. The advantages include more precise and controlled volumetric heating, faster ramp-up temperature, lower energy consumption, and improved properties of the ceramic materials.

Microwave process yields better mechanical properties because the grain size formed is comparatively finer. The shapes of the porosities, if present, are different from those produced...
during conventional heating. In microwave-processed specimens, porosities observed were round edged with higher ductility and toughness. Microwave sintering is capable of producing ceramics which are superior when compared to conventional sintering.[3] The microwave is a term given to electromagnetic radiation that corresponds to frequencies of approximately 1–300 GHz that lies below visible light range in the electromagnetic spectrum. Microwaves are coherent and polarized and can be transmitted, absorbed, or reflected depending on the material type. The nature of the interaction between microwaves and ceramics is complex and is dependent on the dielectric property of the ceramic. Ceramics have excellent dielectric property.[4]

In conventional heating, the material’s surface is first heated followed by the heat moving inward. This means that there is a temperature gradient from the surface to the inside. However, microwave heating generates heat within the material first and then heats the entire volume.[5] A microwave includes various components inclusive of the furnace, an insulation box and susceptors. The insulation box is a small chamber made up of low-density rigid insulation board because low density and very low dielectric loss are required for the box to make it microwave transparent. Microwaves pass through the material with little interaction, allowing the contents to get heated up. Thus, the insulation box itself acts like an oven within the furnace to heat up the contents.[6]

The purpose of glazing dental porcelain is to seal the open pores in the surface of fired porcelain. Surface gloss of dental porcelain can be achieved by either self-glazing or over glazing. Both approaches produce a smooth surface with significantly less flaws and higher gloss level.[7] Glaze fills small porosities and surface irregularities enhancing the surface character. Studies advocate glazing ceramics for superior surface and strength. Unglazed porcelain may cause inflammation of the soft tissues. Researchers determined that glazing was necessary after porcelain adjustment in the clinical setting. Glazed porcelain also allows for easy removal of plaque from its surface.[8] Although the effects of microwave glazing on dental porcelain are still under exploration, the superior qualities of microwave technology are well-known in the industrial settings.

The purpose of this study was to compare and evaluate the surface roughness obtained by conventional and microwave oven glazing of two different porcelains. The null hypothesis of this study was that microwave glazing is not superior to conventional oven glazing and that there is no difference between the 2 metal ceramics.

**Materials and Methods**

Two commercial dental porcelains were used, VITA VMK MASTER (Vita VITA Zahnfabrik, Bad Sackingen, Germany) and IPS CLASSIC (Ivoclar Vivadent, Liechtenstein). A total of sixty samples were made, 30 for each type of porcelain in accordance with ADA specification number 69 for dental ceramics.

**Preparation of customized mold**

Modelling wax (Hindustan Dental Products, Hyderabad) was cut into rectangular blocks of dimensions $22 \pm 0.5 \text{ mm} \times 6 \pm 0.5 \text{ mm} \times 3 \pm 0.5 \text{ mm}$ and a rectangular base of the same dimension was prepared. The wax mold was sprued, invested in a phosphate bonded investment material (Bellasun, Bego). Lost wax technique was followed and casted using centrifugal casting machine. It was casted into nickel-chromium alloy (WIRON 99, Bego).

**Specimen preparation**

The porcelains were mixed with sculpting liquid and placed into the mold. Excess moisture was absorbed with a tissue. Four samples were fired at one time at a temperature of 930°C in the ceramic furnace (Multimat NTX Press) according to manufacturer’s instructions. Due to the small dimensions of the specimens, a single firing cycle for each specimen was adequate to achieve sintering. Specimens with defects were discarded. The samples were allowed to cool and were finished to maintain specified dimensions with medium grit diamond on both sides to remove all the irregularities and polished using ceramic finishing and polishing kit (Shofu Dental GmbH). The dimensions of the specimens were verified with a digital Vernier Caliper and the thirty samples for each type of porcelain were further divided into three groups of 10 samples each for surface treatment.

**Surface treatment**

The control group samples (Group A) were hand polished, and the test group samples were glazed using a conventional oven (Group B) and microwave oven (Group C) under vacuum according to the manufacturer’s instructions. The glaze powder and the glaze liquid (Vita Akzent plus) were mixed together and applied over Group B and Group C uniformly. Group B samples were glazed using oven furnace (Multimat NTX Press) in the conventional method under manufacturer’s instructions.

The samples of Group C were subjected to glazing in a 6 kW microwave furnace (Cober Electronics Inc., Norwalk, Conn) and used a minimum microwave power of 0.4 kW at a frequency of 2.45 GHz. Ten specimens of each type of porcelain were glazed in the microwave oven at the recommended glazing temperature according to the manufacturer’s instructions. A custom-built heating chamber was constructed for the penetration of microwave radiation that acted like an insulation box. The temperature and heat rate were set and monitored using the controller box and the pyrometer. It took approximately 9 min at 903°C to microwave the samples with a holding time of 1 min.
The AFM roughness analysis software (Nanoscope Analysis Version 1.5, Bruker Innova.co, UK) was used to evaluate the surface roughness. Surface roughness is expressed as average Ra value (arithmetical average value of all absolute distances of the roughness profile) which increases with increasing surface roughness. Significance was evaluated at $P < 0.05$ for all tests. All the computational work was performed by means of Statistical Package for Social Software (SPSS) 16.0 Version (SPSS Inc, Chicago, IL, USA) was used for statistical analysis.

Results

The surface roughness of untreated group and treated groups were compared [Table 1]. Mean values and standard deviations of surface roughness (Ra) in nm for control and experimental groups are listed in Table 2. According to the two-way analysis of variance, surface treatment and their interactions were statistically significant [Table 3].

The highest mean value reported was 92.42 nm or 0.0924 μm and lowest was 10.56 nm or 0.0105 μm. The glazed surfaces produced smoother surfaces for both brands of metal ceramics when compared to the control or unglazed group. The hand polished Group (A) presented with higher surface roughness followed by conventional and microwave glazed Group (B and C). Microwave glazing produced smoother surface when compared to other two surface treatments. For the materials tested, VITA VMK MASTER presented with greater surface roughness when compared to IPS CLASSIC. Post hoc Tuckey’s test [Table 4] showed a significant difference between conventional and microwave glazing ($P = 0.007$) but a highly significant difference between conventionally glazed and hand-polished specimens. Furthermore, a highly significant difference between microwave glazed and hand-polished specimens was observed ($P < 0.001$). The surfaces of unglazed specimens revealed greater surface roughness when compared to glazed surfaces.

AFM images of ceramic surfaces after the surface treatments are shown in Figures 1-6.

Discussion

From the results of this in vitro study, the null hypothesis was rejected since microwave glazing was found to be superior to conventional oven glazing and that there is a difference between the 2 metal ceramics.

Metal-ceramic restorations were introduced as a fixed prosthetic option in the year 1962 which exhibited the strength of metal and esthetics of ceramic. A major prerequisite for a good restoration is that the material should be created with a smooth exposed surface. Various authors advise polishing and glazing to enhance the esthetic appearance of these restorations. The key objective to finish and polish a ceramic restoration is to achieve a highly smooth surface that provides satisfactory patient comfort, clinically acceptable esthetics and slows the rate of plaque formation that helps to maintain oral hygiene.
Klausner et al.\[9\] in 1992, compared glazing with different polishing techniques in relation with surface roughness and demonstrated that Shofu polishing kit was capable of producing a smooth surface as glazed porcelain, whereas Vieira et al.\[10\] in 2013 evaluated the roughness of the ceramic surfaces submitted to different finishing and polishing methods and concluded that mechanical finishing and polishing methods were not able to provide a surface as smooth as the glazed surface ceramics.

In vivo studies suggest that a patient’s tongue is susceptible to surface roughness on the restoration. As per Van Noort\[11\] in 1983, a patient can discern surface roughness scratches as small as 20 μm. Jones et al.\[12\] in 2004 concluded that when finishing restorations the surface should have a maximum roughness 0.50 μm if it is not to be detected by the patient. Bollen et al.\[13\] in 1997 reviewed a comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention and suggested an ideal threshold of 0.2 μm.

Roughness can be measured in several ways, the most commonly used in dentistry is the Ra value. Research on surface roughness in dentistry involved qualitative methods such as optical and scanned electron microscopy and quantitative methods such as contact profilometry. Contact diamond and noncontact laser modes as well as laser

Table 4: Results of post hoc Tuckey’s test for surface roughness

| Type of glazing   | Type of glazing | Mean difference | SE       | Significance | 95% CI        |
|------------------|-----------------|-----------------|----------|--------------|---------------|
| Conventional     | Microwave       | 9.4560          | 2.8340   | 0.007*       | 2.3786 - 16.5334 |
| Conventional     | Handpolished    | −62.9500        | 2.8340   | <0.001**     | -70.0274 - 55.8726 |
| Handpolished     | Microwave       | 72.4060         | 2.8340   | <0.001**     | 65.3286 - 79.4834 |

CI=Confidence interval, SE=Standard error, *P < 0.05 significant difference, **P < 0.001 highly significant difference
reflectivity measuring systems are also commonly applied for surface profile measurements. Several shortcomings with respect to the sensitivity of these methods have been well-described by Tholt de Vasconcellos et al.\cite{14} in 2006. The Ra value given by a contact profilometer is a two-dimensional data relating to roughness height but does not provide any information regarding the surface profile. To obtain this information regarding the surface topography, an image of the surface is necessary which can be acquired by means of optical and scanned electron microscopy. However, this does not provide qualitative data in 3 dimensions. Bessing and Wiktorsson\cite{15} in 1983 theorized that some profilometer readings of the ceramic surface can be deceptive due to the presence of voids on the ceramic surface that creates inaccuracy in the readings.

A previous study done by Prasad et al.\cite{16} in 2009 showed that the surface character of microwave porcelain was superior to oven glazed porcelain and polishing alone was comparable to microwave or conventional glazing when attempted with a contact profilometer and scanning electron microscopy analysis. An AFM analysis was carried out for this present investigation, and surface roughness was measured as Ra value. Two commonly available commercial brands of metal ceramics VITA VMK MASTER and IPS CLASSIC were used because of their ease of availability and widespread use for making a metal-ceramic restoration.

When the surface roughness mean values were examined, in a progression from smoothest to roughest, the smoothest surfaces were obtained with microwave glazing followed by conventional glazing and roughest with hand-polishing. The results obtained by statistical tests indicated that there was a significant difference in surface roughness based on the type of glazing and metal ceramic used and also there was a significant interaction between the surface treatment and porcelain. The study results were in accordance with Prasad et al.\cite{16} in 2009 showed a significant difference between conventional oven glazed and microwave glazed specimens. However, a highly significant result was obtained between glazed and unglazed specimens that is conflicting with the previous study. This discrepancy in results may be accredited to the fact that glazing reduces the number of surface porosities and flaws and particularly microwave energy results in greater densification and better grain distribution.

On visual examination, all the specimens of control and experimental groups appeared clinically acceptable. However, a detailed image analysis under AFM corroborated that microwave glazed specimens presented with a surface profile characterized by rounded, flat surfaces with shallow valleys compared to hand-polished specimens that presented highly elongated, sharp peaks, and deep valleys.

The image analysis appreciably seen propose that surface roughness for VITA VMK MASTER porcelain type is higher than that of IPS CLASSIC. It may be due to the difference in the leucite content between the two feldspathic porcelain. The difference in leucite content affects the surface roughness of the dental porcelain has been specified by Sasahara et al.\cite{17} in 2006. The results of Yılmaz et al.\cite{18} in 2010 further support our study wherein the best material in terms of surface smoothness was found to be IPS CLASSIC.

**Conclusions**

Within the limitations of the study, it can be concluded that:

1. Surface topography is influenced by surface treatment. Microwave glazed ceramic is superior to conventional oven glazed ceramic and hand-polishing shows greater surface roughness when compared to glazing.

2. IPS CLASSIC ceramic shows relatively smooth surface when compared to VITA VMK MASTER irrespective of the surface treatment.
Financial support and sponsorship
Nil.

Conflicts of interest
There are no conflicts of interest.

References
1. Katz JD. Microwave sintering of ceramics. Annu Rev Mater Sci 1992;22:153-70.
2. Tang CY, Uskokovic PS, Tsui CP, Veljovic DJ, Petrovic R, Janackovic DJ. Influence of microstructure and phase composition on the nano-indentation characterization of bio-ceramic materials based on hydroxyapatite. Ceram Int 2009;35:2171-8.
3. Clark D, Sutton WH. Microwave processing of materials. Annu Mater Sci 1996;26:299-331.
4. Sutton WH. Microwave processing of ceramics. Am Ceram Soc Bull 1989;68:376-86.
5. Yadoji P, Peelamedu R, Agrawal R, Roy R. Microwave sintering of Ni-Zn ferrites: Comparison with conventional sintering. Mater Sci Eng 2003;98:269-78.
6. Khalil KA. Advanced Sintering of Nano-Ceramic Materials, Ceramic Materials - Progress in Modern Ceramics, Prof. Feng Shi (Ed.), ISBN: 978-953-51-0476-6, InTech, 2012. Available from: http://www.intechopen.com/books/ceramic-materials-progress-in-modern-ceramics/advanced-sintering-ofnano-ceramic-materials.
7. Motro PF, Kursoglu P, Kazazoglu E. Effects of different surface treatments on stainability of ceramics. J Prosthodont 2012;10:231-7.
8. Anusavice KJ, Phillips RW. Phillip’s Science of Dental Materials. 11th ed. St. Louis: Elsevier; 2003. p. 660-72.
9. Klausner LH, Cartwright CB, Charbeneau GT. Polished versus autoglazed porcelain surfaces. J Prosthet Dent 1982;47:157-62.
10. Vieira AC, Oliveira MC, Lima EM, Rambob I, Leite M. Evaluation of the surface roughness in dental ceramics submitted to different finishing and polishing methods. J Indian Prosthodont Soc 2013;13:290-5.
11. Van Noort R. Controversial aspects of composite resin restorative materials. Br Dent J 1983;155:380-5.
12. Jones CS, Billington RW, Pearson GJ. The in vivo perception of roughness of restorations. Br Dent J 2004;196:42-5.
13. Bollen CM, Lambechts P, Quirynen M. Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: A review of the literature. Dent Mater 1997;13:258-69.
14. Tholt de Vasconcellos B, Miranda-Junior WG, Prioli R, Thompson J, Oda M. Surface roughness in ceramics with different finishing techniques using atomic force microscope and profilometer. Oper Dent 2006;31:442-9.
15. Bessing C, Wiktorsson A. Comparison of two different methods of polishing porcelain. Scand J Dent Res 1983;91:482-7.
16. Prasad S, Monaco EA Jr., Kim H, Davis EL, Brewer JD. Comparison of porcelain surface and flexural strength obtained by microwave and conventional oven glazing. J Prosthet Dent 2009;101:20-8.
17. Sasahara RM, Ribeiro Fda C, Cesar PF, Yoshimura HN. Influence of the finishing technique on surface roughness of dental porcelains with different microstructures. Oper Dent 2006;31:577-83.
18. Yilmaz C, Korkmaz T, Demirköprüli H, Ergün G, Ozkan Y. Color stability of glazed and polished dental porcelains. J Prosthodont 2008;17:20-4.