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ORIGINAL ARTICLE

Flexure Strength and Fatigue Durability of Ce-TZP/Al_2O_3 Nanocomposite with Different Sintering Process and Surface Treatment

Tomofumi Takano, Akinori Tasaka, Masao Yoshinari, Kaoru Sakurai

ABSTRACT

Purpose: Ce-TZP/Al_2O_3 nanocomposite (NANOZR) has not only higher strength, but also higher fracture toughness than conventional Y-TZP. NANOZR is generally machined from densely sintered block. However, it is more efficient to sinter densely after machining partially sintered block, if the mechanical strength of final product is enough. Moreover, as for NANOZR, it has been reported that mechanical strength changes with surface treatments. The purpose of this study is to evaluate the effect of sintering process and surface treatment on biaxial flexure strength and fatigue durability of NANOZR.

Material and methods: Two kinds of NANOZR specimens were prepared (Pre-sinter and Full-sinter). Pre-sinter and Full-sinter were given machine polish (MP) or shot-blast and fluoric acid etching (SB + HF). Biaxial flexure strength was measured as recommended by ISO 6872. The cyclic loading test of the load equivalent to 480 MPa was performed by the application of a cyclic load in the bending mode at a frequency of 10 Hz, for 10^6 cycles in distilled water at 37°C.

Results: Biaxial flexure strength of MP was 1244 ± 43 MPa and 1320 ± 50 MPa in Pre-sinter and Full-sinter, respectively. and it of SB + HF was 1070 ± 60 MPa and 1069 ± 12 MPa in Pre-sinter and Full-sinter, respectively. In the cyclic fatigue test, the durability over the 10^6 cycle load equivalent to 480 MPa was shown on all the conditions.

Conclusion: Even if NANOZR was densely sintered after machining partially sintered block, having satisfactory biaxial flexure strength and fatigue durability on clinical application as dental prosthetic material was shown.

Keywords: Clinical restorative dentistry, Prosthodontics, Impression material, Laboratory prescriptions.

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INTRODUCTION

Zirconia, which is stronger than previously used ceramics and superior in biocompatibility, has rapidly become used widely as an all-ceramic restoration material in dentistry. The current zirconia for dental use is combined with yttria. Yttria-partially stabilized tetragonal zirconia (Y-TZP), in which the crystal structure is stabilized in the tetragonal state at room temperature, is frequently used, and its fracture toughness is the highest among ceramic materials.[1-4] However, although Y-TZP shows the general chemical durability of ceramics, it is known to undergo low-temperature degradation (LTD) in the aqueous solutions encountered in the oral environment because the surface crystal structure transforms from the tetragonal to monoclinic state under a hydrothermal condition. LTD is initiated from isolated surface grains, between which water is incorporated into the zirconia lattice through dissolution of Zr-O-Zr bonds and filling of oxygen vacancies[1,5] and may result in a significant reduction in the strength and toughness of Y-TZP[6,7] a problem that is further exacerbated by exposure of a restoration to cyclic stresses such as chewing[8] Recently, Ce-TZP/Al_2O_3 nanocomposite (NANOZR) was reported to be resistant to LTD, offering not only higher strength, but also higher fracture toughness than Y-TZP.[9-11]

CAD/CAM technology is indispensable to clinical application of zirconia. The processing accuracy and time by CAD/CAM have a large portion depending on the character of the charge of a work material. Therefore, most CAD/CAM systems are sintered densely after machining partially sintered block. On the other hand, as for machining of NANOZR, the densely sintered block has been machined. However, it is more efficient to sinter densely after machining partially sintered block for shortening of machining time and extension of life-span of a tool, if the mechanical properties of machined products are enough. Moreover, when using it as an implant fixture, performing surface treatments, such as machine polish, and shot-blast and etching, is also assumed. The purpose of this study is to evaluate the effect of sintering process and surface treatment on biaxial flexure strength and fatigue durability of NANOZR in order to examine whether the mechanical strength of NANOZR which sintered densely after machining partially sintered block is satisfactory on clinical application.

MATERIALS AND METHODS

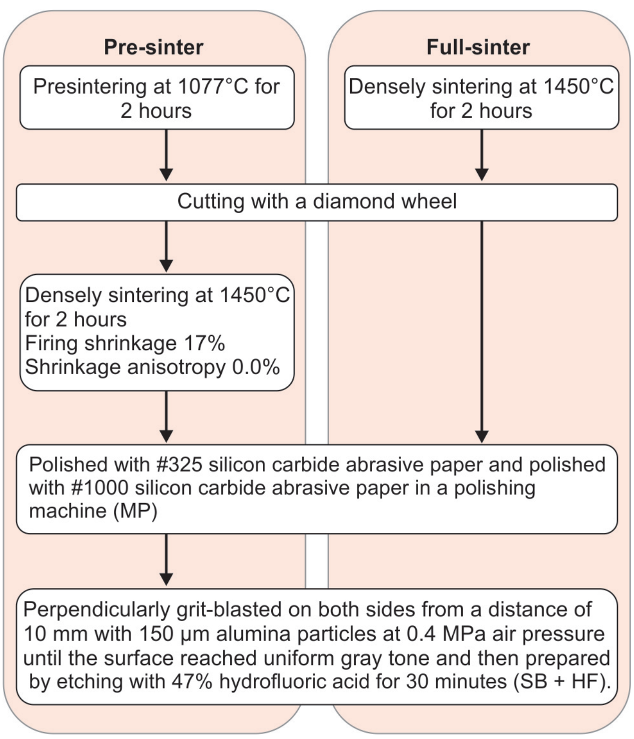
Schema of fabrication sequence of experiment samples is shown in Flow Chart 1. NANOZR (ZrO_2, 67.9 mass%; Al_2O_3, 21.5 mass%; CeO_2, 10.6 mass%; MgO, 0.06 mass%; TiO_2, 0.03 mass%) (C type; Panasonic Electric Works, Osaka, Japan) powders were processed in a cylindrical rod by a cold isostatic pressing method. Specimens of NANOZR cylinders were randomly divided into 2 groups Flow Chart 1. Pre-sinter was densely sintered after machining partially sintered block, Full-sinter was machined densely sintered block. Pre-sintered was firstly sintered at 1077°C for 2 hours and the they were prepared by machining with a diamond wheel and then densely sintered at 1450°C for 2 hours in air atmosphere. Full-sinter which was sintered at 1450°C for 2 hours in air atmosphere was prepared by machining with a diamond wheel. The size of both Pre-sinter and Full-sinter specimens was 13 mm in diameter and 0.5 mm in thickness.

Furthermore, each group was divided into 2 groups by surface treatment: machine-polished group (MP), shot-blasted and etched group (SB + HF). MP were prepared on both sides of each specimen polished with #325 silicon carbide abrasive paper and polished with #1000 silicon carbide abrasive paper. SB + HF were perpendicularly shot-blasted on both sides from a distance of 10 mm with 150 µm alumina particles at 0.4 MPa air pressure until the surface reached a uniform gray tone and then prepared by etching with 47% hydrofluoric acid for 30 minutes. These specimens were cleaned ultrasonically in acetone and distilled water for 10 minutes.

Surface Roughness and Microscopic Observations

Surface roughness of the specimens was analyzed by means of a surface roughness tester (Surfcom 130 A, Accretech, Tokyo, Japan). Four specimens, each measured at 3 points, were used for each condition. The arithmetic mean surface roughness (Ra) was determined under these conditions with a cut-off value of 0.8 mm, measurement length of 4.0 mm, and measurement speed of 0.6 mm/sec. Microscopic observation was conducted to characterize the microstructure on the surfaces, before and after cyclic loading, by scanning electron microscopy (SEM) (SU6600, HITACHI, Tokyo, Japan).

Flow Chart 1: Schema of fabrication sequence of experiment samples



Biaxial Flexure Test

In biaxial flexure test, 5 specimens from each group were used to measure biaxial flexure strength in a universal testing machine (AG-I 20kN, Shimadzu, Kyoto, Japan) with a cross-head speed of 0.5 mm/min in air at room temperature. Disk specimens were laced on 3 steel spheres positioned 120° apart in a circle (8.0 mm in diameter). A flat-end loading cylinder with a radius of 0.8 mm was then applied. Biaxial flexure strength was calculated according to the equation listed in ISO 6872, with a Poisson's ratio value of 0.25 for each specimen.

Cyclic Loading Test

Five specimens from each group were used in cyclic fatigue test. A servo-hydraulic universal testing machine (EHF-F05, Shimadzu, Kyoto, Japan) was used to apply cyclic load in the bending mode at a frequency of 10 Hz, for 10^6 cycles in distilled water at 37°C. The applied load was set at 480 MPa that was 150% of the required fatigue strength of Y-TZP in surgical implants as specified in ISO 13356. Finally, the durability was judged whether the specimen were broken under this cyclic fatigue test.

STATISTICAL ANALYSIS

Biaxial flexure strength were statistically analyzed with an ANOVA ($\alpha = 0.05$). Statistical analysis was performed using SPSS for windows, version 16.0 (SPSS Software, Munich, Germany).

RESULTS

The Ra values of Pre-sinter were 0.08 ± 0.01 in MP and 0.64 ± 0.05 in SB + HF. On the other hand, it of Full-sinter were 0.07 ± 0.06 in MP, and 0.65 ± 0.04 in SB + HF. The representative features of Pre-sinter and Full-sinter are shown in the SEM micrographs (Fig. 1). In SEM observation, the machining trace was observed in the field where Pre-sinter and Full-sinter are flat in MP, and the roughened surface was observed at SB + HF compared with MP. Moreover, there was no difference in a crystal grain size in Pre-sinter and Full-sinter.

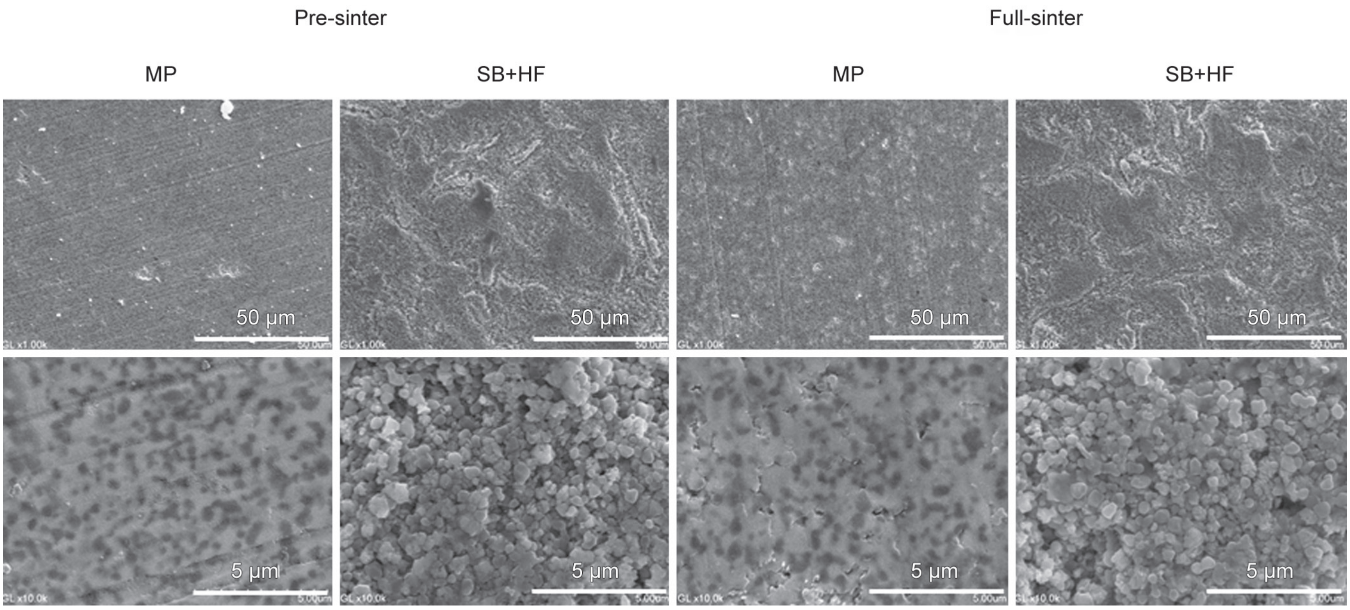


Fig. 1: Scanning electron micrographs (the upper stand magnification 1,000× and the lower stand 10,000×) of zirconia ceramic surfaces

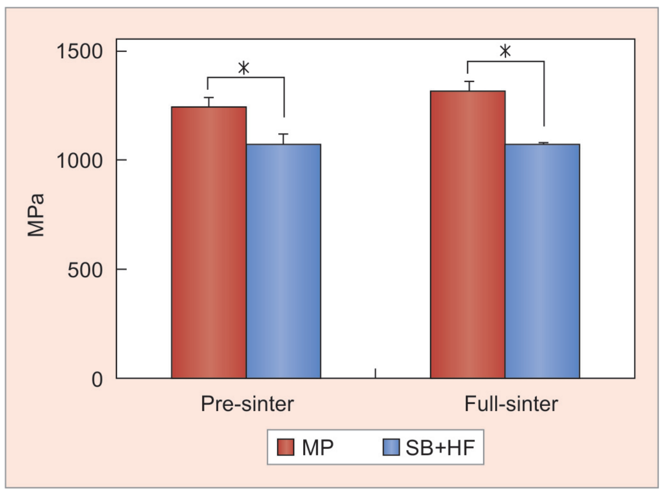
Biaxial flexure strengths of MP was 1244-1320MPa and those of SB + HF was 1069 to 1070 MPa (Graph 1). Two-way ANOVA revealed the effects of sintering process, surface treatment and their interaction (Table 1). As for biaxial flexure strengths, there was significant difference between MP and SB + HF. On the other hand, there was no significant difference between Pre-sinter and Full-sinter. Cyclic fatigue test showed the durability over the 106 cycle load of 480 MPa on all conditions.

DISCUSSION

Biaxial flexure strength of MP was 1094-1320 MPa, and that of SB + HF was 998-1070 MPa (Graph 1). The biaxial flexure strength of NANOZR was over 800 MPa which is the standards of the stability fixed prostheses substructure of four units based on ISO 6872 on all the conditions. There was significant difference between MP and SB + HF. It is thought that this reason is due to microcrack formed in the surface of NANOZR by the surface treatment. On the other hand, there was no significant difference between Pre-sinter and Full-sinter, suggesting that the biaxial flexure strength of NANOZR was not affected by firing process.

The difference in the sintering temperature of zirconia affects the grain size, and has influence on strength.[12] Pre-sinter performed twice sintering, and the Full-sinter was one time. Since the last sintering temperature of Pre-sinter is the same 1450°C as Full-sinter, the difference was not observed in the grain size as shown in SEM observation resulting no difference in the strength of Pre-sinter and Full-sinter.

It has been reported that cyclic fatigue strength of NANOZR was 667 to 881 MPa in the bending mode at a frequency of 10 Hz, for 10^6 cycles in distilled water at 37°C.[13] Cyclic fatigue test on all conditions showed the durability over the 10^6 cycle load of 480 MPa which is over the standards of the fatigue strength of Y-TZP in the implant for surgery determined by ISO 13356 .



Graph 1: Bi-axial flexure strength with different sintering process and surface treatment

Table 1: ANOVA for the main effects of sintering process and surface treatment, and their interaction on biaxial flexure strength

Source of variation	*Sum of squares*	*df*	*Mean square*	*F*	*p*
Corrected model	239275	3	79758	39	< 0.001*
Sintering process	7083	1	7083	3.5	0.081
Surface treatment	224717	1	224717	110	< 0.001*
Corrected model × surface treatment	7476	1	7476	3.7	0.74
Error	32645	16	2040		

Pre-sinter were densely sintered after machining partially sintered block. The machining time of partially sintered block is 1/6 of the machining time of densely sintered block. Shortening of machining time enables supply of efficient dental prostheses. However, if densely sintering is carried out after processing, 17% of firing shrinkage will arise. Therefore, the accuracy of the manufactured dental prosthesis is subject. On the other hand, Full-sinter is machined densely sintered block. There is no firing shrinkage after machining, manufacture of accurate dental prostheses are attained.

Shot-blast and etching processing to NANOZR may be performed as a pretreatment in the case of the application to dental implants and dental prostheses. It is clear from former research that this surface treatment has influence on mechanical strength of NANOZR.[13] Therefore, the biaxial flexural strength of NANOZR which performed shot-blast + etching processing in different fabrication sequence was examined supposing clinical application.

This time, it has become clear that the fatigue strength of Pre-sinter is equivalent to the fatigue strength of Full-sinter. This result leads to establishment of the efficient method of processing NANOZR, and can also expect broad clinical application of NANOZR.

CONCLUSION

The biaxial flexure strength of NANOZR was not affected by firing process. From the clinical point of view, the biaxial flexure strength of NANOZR treated by shot-blasting and etching was much higher than 800 MPa, the required flexure strength of substructure ceramic for prostheses involving 4 or more units specified in ISO 6872.

Furthermore, the durability over the cyclic load of NANOZR was 106 cycle and 480 MPa or more.

As mentioned above, even if NANOZR was densely sintered after machining a Pre-sintered block, having satisfactory biaxial flexure strength and fatigue durability on clinical application as dental prosthetic material was shown.

REFERENCES

1. Sato H, Yamada K, Pezzotti G, Nawa M, Ban S. Mechanical properties of dental zirconia ceramics changed with sandblasting and heat treatment. Dent Mater J 2008;27(3):408-414.
2. Andreiotelli M, Wenz HJ, Kohal RJ. Are ceramic implants a viable alternative to titanium implants? A systematic literature review. Clin Oral Implants Res 2009;20 Suppl 4:32-47.
3. Karakoca S, Yilmaz H. Influence of surface treatments on surface roughness, phase transformation and biaxial flexural strength of Y-TZP ceramics. J Biomed Mater Res B Appl Biomater 2009;91(2):930-937.
4. Nakazato T, Takahashi H, Yamamoto M, Nishimura F, Kurosaki N. Effect of polishing on cyclic fatigue strength of CAD/CAM ceramics. Dent Mater J 1999;18(4):395-402.
5. Guo X. Property degradation of tetragonal zirconia Induced by low-temperature defect reaction with water molecules. Chemistry of Materials 2004;16(21):3988-3994.
6. Hirano M. Inhibition of Low Temperature Degradation of Tetragonal Zirconia Ceramics A Review. British Ceramic Transactins and journal 1992;91(5):139-147.
7. Piconi C, Maccauro G. Zirconia as a ceramic biomaterial. Biomaterials 1999;20(1):1-25.
8. Studart AR, Filser F, Kocher P, Gauckler LJ. In vitro lifetime of dental ceramics under cyclic loading in water. Biomaterials 2007;28(17):2695-2705.
9. Tanaka K, Tamura J, Kawanabe K, Nawa M, Oka M, Uchida M, et al. Ce-TZP/Al_2O_3 nanocomposite as a bearing material in total joint replacement. J Biomed Mater Res 2002;63(3):262-276.
10. Ban S, Sato H, Suehiro Y, Nakanishi H, Nawa M. Biaxial flexure strength and low temperature degradation of Ce-TZP/Al_2O_3 nanocomposite and Y-TZP as dental restoratives. J Biomed Mater Res B Appl Biomater 2008;87(2):492-498.
11. Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Evaluation of a high fracture toughness composite ceramic for dental applications. J Prosthodont 2008;17(7):538-544.
12. Nakamura K, Adolfsson E, Milleding P, Kanno T, Ortengren U. Influence of grain size and veneer firing process on the flexural strength of zirconia ceramics. Eur J Oral Sci 2012;120(3):249-254.
13. Takano T, Tasaka A, Yoshinari M, Sakurai K. Fatigue strength of Ce-TZP/Al_2O_3 nanocomposite with different surfaces. J Dent Res 2012;91(8):800-804.

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