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Effect of Sintering Temperature on Flexural Strength of Two Types of Zirconia

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A B S T R A C T

Objectives: Any change in the sintering process can directly affect the micro-structure and properties of zirconia. This study sought to assess the effect of sintering temperature on flexural strength of IPS e.max ZirCAD MO Ivoclar (EZI) and CopraSmile White Peaks Symphony (WPS) zirconia blocks.

Materials and Methods: In this in vitro, experimental study, 30 EZI and 30 WPS zirconia blocks measuring 10 x 10 x 1 mm were milled and sintered at 1440, 1500 and 1530°C in three subgroups. The flexural strength of the specimens was measured by a testing machine with piston-on-3-ball method according to ISO2015. Data were analyzed using one-way ANOVA.

Results: The mean flexural strength was 1.31±0.49, 1.09±0.24 and 1.29±0.48 MPa in 1440, 1500, and 1530°C subgroups of EZI, and 1.44±0.61, 1.18±0.35, and 1.33±0.54 MPa in 1440, 1500, and 1530°C subgroups of WPS zirconia, respectively. Two-way ANOVA revealed that the effects of zirconia type (P=0.484), temperature (P=0.258) and their interaction (P=0.957) on flexural strength were not significant.

Conclusion: Increasing the sintering temperature from 1440°C to 1530°C did not increase the flexural strength of EZI or WPS zirconia.

Keywords: Zirconium Oxide; Flexural Strength; IPS e.max Press

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INTRODUCTION

Advances in dental science have led to an increase in use of ceramics with optimal esthetics, biological properties, and biomechanics [1,2]. The novel ceramics have more favorable esthetics, biocompatibility, wear resistance, and chemical stability [3,4]. Despite the great advances in composition of ceramics, some ceramic types still have poor mechanical properties and high brittleness, which limit their application in posterior areas [5]. Nonetheless, introduction of monolithic zirconia ceramics eliminated the existing problems related to ceramic strength to some extent, and enabled their application in high stress-bearing areas [6]. Zirconia ceramics are increasingly used in

different parts of dentition due to their optimal biomechanics and favorable esthetics [7]. Use of zirconia as a core improves the mechanical properties of all-ceramic restorations [8,9]. Moreover, advent of computer-aided design/ computer-aided manufacturing technology significantly increased the use of zirconia as the first option for fixed prosthetic restorations [10]. Monolithic zirconia restorations are highly popular due to their high flexural strength (+1000MPa), which exceeds the maximum occlusal loading during normal mastication. They have even shown flexural strength over 2000N [11]. Optimal restoration color, minimal wear of the opposing teeth, conservative preparation, and high long-term clinical success rate are among the favorable properties of these restorations [12].

In the process of fabrication of crowns, bridges, frameworks, and other types of zirconia-based restorations, they are sintered in a dental furnace at high temperatures. Sintering can greatly affect the micro-structure and properties of zirconia. In the process of sintering, heat is transferred from the surface to the core to obtain matured sintered zirconia [13]. Sintering temperature is an important parameter affecting the size of zirconia particles and their density, which directly impact on the rate of porosities and growth of particles. The sintering temperature can also affect the strength and resistance of zirconia. In the process of sintering, zirconia undergoes significant shrinkage. Thus, variations in sintering parameters of zirconia can directly affect its properties [14,15].

Flexural strength is a physical property that shows resistance of a material against deformation [16]. The available studies on the effects of sintering temperature on strength of zirconia have reported controversial results [17-23]. Thus, this study aimed to assess the effect of sintering temperature on flexural strength of IPS e.max ZirCAD MO Ivoclar (EZI) and CopraSmile White Peaks Symphony (WPS) zirconia blocks.

MATERIALS AND METHODS

This in vitro, experimental study evaluated 30 EZI (Ivoclar Vivadent, Schaan, Liechtenstein) and 30 WPS (White Peaks Dental, Germany) zirconia blocks. The sample size was calculated to be 10 in each subgroup according to a previous study [21], assuming 95% confidence interval, 80% study power, mean difference of 1, and standard deviation of 0.75. The study was approved by the ethics committee of Hamadan University of Medical Sciences (IR.UMSHA.REC.1398.279, IR.UMSHA.REC.1398.333). Zirconia blocks measuring 10×10mm with 1mm thickness were milled out of semi-sintered zirconia blocks. Considering 20% shrinkage, the specimens were first milled in 12×12mm dimensions by a milling machine (NEMO Co, Mashhad, Iran) and after shrinkage, reached 10 x 10 mm dimensions. After staining with A3 shade, EZI specimens were first dried at 170° for 30 min, and were then sintered in three subgroups at 1440°C, 1500°C and 1530°C temperatures [10,11] in a furnace (Ceramill Therm; Amann Girrbach, Germany) for 2 h, as recommended by the manufacturer. WPS specimens were first dried in a furnace (Ceramill Therm, Amann Girrbach, Germany) at 1400°C for 30 min, and were then sintered in three subgroups at 1440, 1500 and 1530°C temperature as recommended by the manufacturer [24]. After sintering, the zirconia specimens underwent flexural strength test in a testing machine (SFA-50; Santam, Iran) using piston-on-3-ball method at room temperature according to ISO2015 standard. For this purpose, the specimens were placed on 3 steel balls with 2.3mm diameter. Next, a piston with 1.4mm diameter applied load to the center of specimens at a speed of 0.5mm/min until fracture [25]. The two inferior metal balls had 1 cm distance, and the upper middle ball had 5 cm distance from each side. The load at fracture was recorded in megapascals (Mpa) and biaxial flexural strength was calculated using the following formulae:

$$(1)\ S = -\frac{0.2387P\ (X-Y)}{d2}\ ;$$

$$(2)\ X = (1+\nu)\ \ln(\frac{r_2}{r_3})^2 + (\frac{[1-\nu]}{2})(\frac{r_2}{r_3})^2;\ \text{and}$$

$$(3)\ Y = (1+\nu)\ (1+\ln(\frac{r_1}{r_3})^2) + (1-\nu)(\frac{r_1}{r_3})^2$$

Where S is the biaxial flexural strength, P is the load at fracture, d is the disc thickness at the fracture site, v is the Poisson's ratio, r1 is the radius of the supporting circle, r2 is the radius of the loaded area, and r3 is the radius of the specimen [25]. Also, the specimens were inspected under an electron microscope (VEGA XMU; Tescan, Czech Republic). For this purpose, they were first glued on a glass side with aluminum adhesive and coded. Next, they were coated with 200Å silver and connected to the stage of the device with aluminum foil to ensure adequate conductivity [26]. They were then inspected under the electron microscope.

Data were analyzed using SPSS version 21. Normal distribution of data was confirmed by the Kolmogorov-Smirnov test. Thus, comparisons were made using one-way analysis of variance and the level of significance was set at 0.05.

RESULTS

Table 1 presents the measures of central dispersion for the flexural strength of EZI and WPS zirconia groups. For the EZI group, the minimum flexural strength was noted at 1500°C and the maximum flexural strength was noted at 1440°C subgroups.

Table 1. Measures of central dispersion for the flexural strength of zirconia groups

Zirconia	Temperature (°C)	Mean	SD
White Peaks Symphony	1440	1.44	0.61
	1500	1.18	0.35
	1530	1.33	0.54
IPS e.max ZirCAD MO Ivoclar	1440	1.31	0.49
	1500	1.09	0.24
	1530	1.29	0.48

SD: standard deviation

For the WPS group, the minimum flexural strength was noted in 1500°C and the maximum flexural strength was noted in 1440°C subgroups. Two-way ANOVA revealed that the effects of zirconia type (P=0.484), temperature (P=0.258) and their interaction (P=0.957) on flexural strength were not significant. Figure 1 illustrates the electron microscopic micrographs of the surface of EZI specimens sintered at three different temperatures. As shown, the smallest particles were seen in specimens sintered at 1440°C. The particle size increased as the sintering temperature increased, and the largest particles were seen in 1530°C subgroup. Figure 2 illustrates the electron microscopic micrographs of the surface of WPS specimens sintered at three different temperatures. As shown, the smallest particles were seen in specimens sintered at 1440°C. The particle size increased as the sintering temperature increased, and the largest particles were seen in 1530°C subgroup.

DISCUSSION

This study assessed the effect of sintering temperature on flexural strength of EZI and WPS zirconia blocks. Two-way ANOVA revealed that the effects of zirconia type (P=0.484), temperature (P=0.258), and their interaction (P=0.957) on flexural strength were not significant. Thus, it appears that increasing the sintering temperature from 1440°C to 1530°C has no significant effect on flexural strength of EZI and WPS zirconia.

However, scanning electron microscope images of both zirconia brands showed that the size of zirconia particles increased and their orientation became more regular as the sintering temperature increased from 1440°C to 1530°C. The results of previous studies on this topic have been controversial. Amat et al. [25] sintered zirconia discs at 1400°C, 1450°C, 1500°C, 1550°C, and 1600°C for 120 min and measured their flexural strength. They found that increasing the sintering temperature increased the flexural strength of zirconia. The maximum flexural strength was noted in specimens sintered at 1500°C.

Ersoy et al. [27] evaluated 120 In-Coris ZI zirconia specimens sintered in three groups: at 1510°C for 120 min, 1540°C for 25 min, and 1580°C for 10 min. They measured the flexural strength of specimens, and reported maximum flexural strength in specimens sintered at 1580°C for 10 min. Also, the flexural strength of specimens sintered at 1540°C was significantly higher than that of specimens sintered at 1510°C.

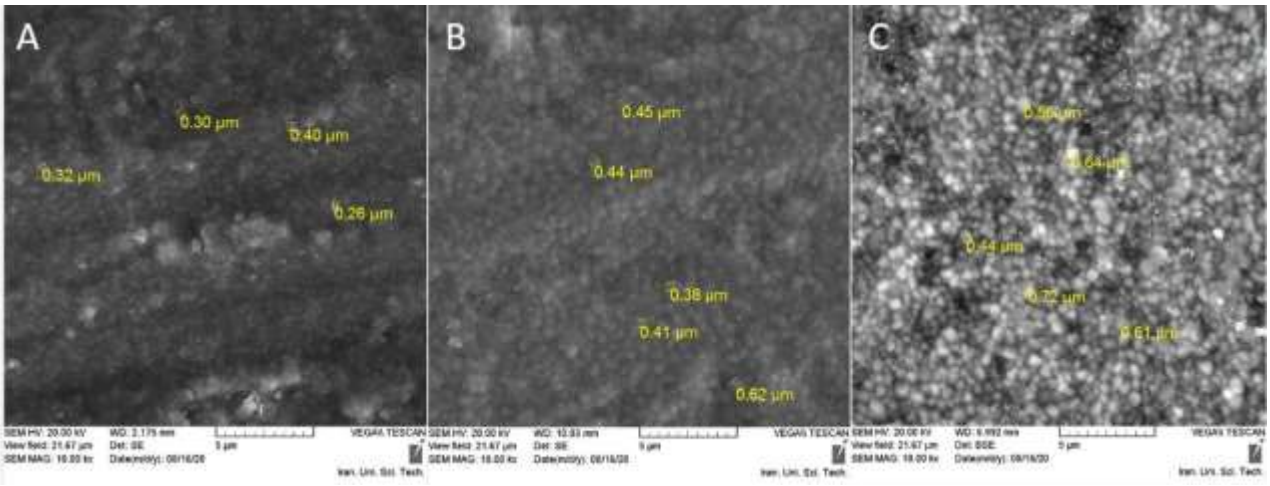

Fig. 1. Electron microscopic micrographs of the surface of EZI specimens sintered at three different temperatures at x10,000 magnification: (A) surface of specimen sintered at 1400°C; (B) surface of specimen sintered at 1500°C; (C) surface of specimen sintered at 1530°C

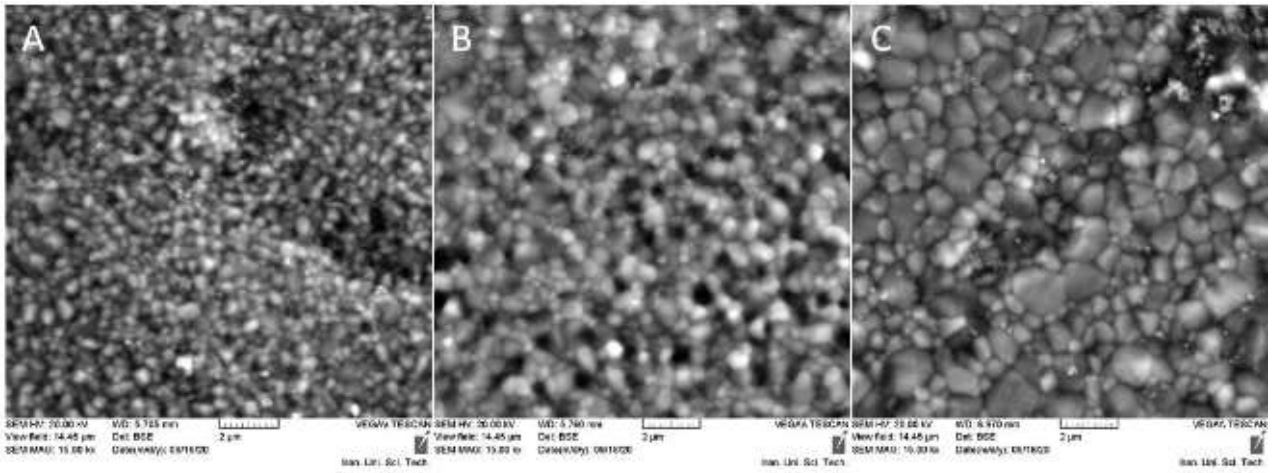

Fig. 2. Electron microscopic micrographs of the surface of WPS specimens sintered at three different temperatures at ×15,000 magnification: (A) surface of specimen sintered at 1400°C; (B) surface of specimen sintered at 1500°C; (C) surface of specimen sintered at 1530°C

The results of the abovementioned two studies were different from our findings, which may be attributed to the use of different zirconia brands and sintering protocols including temperatures and durations. Moreover, the abovementioned two studies measured the flexural strength according to ISO2008 standard while we applied the ISO2015 standard for this purpose. Furthermore, duration of sintering was different in the three subgroups in the study by Ersoy et al, [27] while duration of sintering was the same in the three subgroups of each zirconia group in our study.

In contrast to our study, Juntavee and Attashu [28] indicated the positive effect of increasing the sintering temperature and duration on flexural strength of zirconia.

They sintered the specimens at 1350°C, 1450°C, and 1550°C temperatures for different time periods and measured the flexural strength with piston-on-3-ball method according to ISO2015 standard. Difference between their findings and ours may be attributed to different sintering protocols and thickness of specimens. The thickness of specimens was 1 mm in our study and duration of sintering was the same in all

three subgroups; while, they evaluated specimens with 1.5 mm thickness and sintered the specimens for different time periods. Evidence shows that use of specimens with 4 mm thickness for measurement of flexural strength may mask the possible effects of phase transformation on flexural strength of zirconia. Thus, the recommended thickness for specimens is 1±0.2 mm [29]. Unlike our study, Stawarczyk et al. [22] showed the negative effect of increasing the sintering temperature on flexural strength of zirconia. They evaluated 1300°C, 1350°C, 1400°C, 1450°C, 1500°C, 1550°C, 1600°C, 1650°C and 1700°C sintering temperatures and measured the flexural strength using the piston-on-3-ball method according to ISO2008 standard. They reported the maximum flexural strength in specimens sintered at temperatures between 1400°C and 1500°C. They found a significant inverse correlation between the flexural strength and sintering temperature such that the flexural strength of zirconia decreased in sintering temperatures higher than 1500°C. Disagreement between their results and ours may be due to the use of different zirconia brands and ISO standards.

Our results were in agreement with those of Sen et al, [21] who evaluated the flexural strength of Vita YZ HT zirconia specimens with 1 mm thickness sintered at 1350°C, 1450°C and 1600°C temperatures using the same technique as ours. They found no significant difference in flexural strength of the three groups, and reported that increasing the sintering temperature had no significant effect on flexural strength.

Electron microscopic micrographs of specimens in the present study revealed that increasing the sintering temperature increased the size of zirconia particles, despite causing no significant change in flexural strength.

The relationship of zirconia particle size and mechanical properties has been previously investigated, and size of particles is believed to play a fundamental role in toughness, strength, and resistance of zirconia [30].

In line with our findings, Tekeli and Erdogan [31] showed that sintering conditions affected the density and mechanical properties of zirconia. In contrast, some others failed to show the significant effect of sintering conditions on particle size. For instance, Kim et al. [26] indicated that zirconia specimens sintered under different conditions had similar density with no significant difference. Hjerppe et al. [32] demonstrated that sintering conditions had insignificant effect on the size of zirconia particles and no effect on mechanical properties. Cottom and Mayo [33] showed that faster sintering resulted in smaller particle size. Although it has been reported that larger particles confer higher flexural strength [31], the current results revealed no significant difference; thus, some factors other than particle size may affect the flexural strength. One limitation of this study was that specimens evaluated in this study were flat and had no surface irregularities; whereas, tooth crowns have a complex curved morphology. Use of specimens with tooth morphology can increase the accuracy of the obtained results. Also, this study only assessed two zirconia brands available in the market. Future studies are required to compare the flexural strength of different zirconia brands. Moreover, the flexural strength of monolithic zirconia should be compared with metal-ceramic and other types of all-ceramic restorations. Last but not least, future studies should better simulate the clinical setting to obtain more generalizable results.

CONCLUSION

Increasing the sintering temperature from 1440°C to 1530°C did not increase the flexural strength of EZI or WPS zirconia although the particle size increased.

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CONFLICT OF INTEREST STATEMENT

None declared.

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