ORIGINAL ARTICLE

The effects of sintering temperature and duration on the flexural strength and grain size of zirconia

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Abstract

Objective: This study investigated the effect of different sintering temperatures and times on the flexural strength and grain size of zirconia.

Material and methods: Zirconia specimens (In-Coris ZI, In-Coris TZI, 120 samples) were prepared in a partially sintered state. Subsequently, the specimens were randomly divided into three groups and sintered at different final sintering temperatures and for various durations: 1510 °C for 120 min, 1540 °C for 25 min and 1580 °C for 10 min. Three-point flexural strength (for 120 samples, 20 samples per group) was measured according to the ISO 6872: 2008 standards. The grain sizes were imaged by scanning electron microscopy (SEM) and the phase transitions were determined by X-ray diffraction (XRD). The data were analyzed using one-way ANOVA and Duncan tests ($p < 0.05$).

Results: The highest flexural strength was observed in ZI and TZI samples sintered at 1580 °C for 10 min. The differences between the ZI samples sintered at 1510 °C for 120 min and those sintered at 1540 °C for 25 min were statistically insignificant. Also, TZI samples sintered at 1510 °C for 120 min and those sintered at 1540 °C for 25 min also did not show any statistically significant differences. There were no visible differences in the grain sizes between the ZI and TZI specimens. The XRD patterns indicated similar crystalline structure for both materials subjected to the three different procedures.

Conclusions: The results of this study showed that experimented high sintering temperature and short sintering time combination increases the flexural strength of zirconia.

Introduction

Zirconia is used for dental restoratives, such as crowns, bridges, implant fixtures and implant abutments [1] due to its suitable properties for dental prostheses.[2–4] The excellent mechanical properties of zirconia are attributed to the stress-induced transformation toughening mechanism, similar to that encountered in quenched steel.[5–7] Zirconia fixed partial dentures are used to replace posterior teeth because of the high flexural strength and fracture toughness of zirconia, which is used as the framework material.[8–10] Fractures of zirconia frameworks have rarely been reported.[11–15] In contrast, chipping of the veneering ceramic is a frequent complication.[11–15] From a clinical point of view, the stability of the system consisting of both the zirconia framework and the veneering ceramic is important.

To decrease the costs and simultaneously overcome the chipping issue, monolithic zirconia fixed dental prostheses without veneering ceramic are produced. Such restorations are esthetically unsuitable due to their high opacity. Sintering parameters have an effect on the crystalline content.[16–19] It

has been shown that the holding time during sintering affects the grain growth in the material.[20] As the grain size increases, zirconia becomes less stable and more susceptible to spontaneous tetragonal-to-monoclinic phase transformations, which may result in a gradual strength decrease.[20]

The monoclinic phase is stable up to 1170 °C; above this temperature, it transforms into the tetragonal phase, which remains stable up to 2370 °C. The cubic phase of zirconia on the other hand, exists up to the melting point, 2680 °C.[21,22] The tetragonal form of metastable zirconia could be achieved at room temperature by alloying zirconia with other oxides (stabilizers), such as CaO,[23] MgO,[24] Y_2O_3,[25,26] and CeO_2.[27] Y_2O_3 is the most widely used stabilizer for dental zirconia.[21] In response to tensile stresses at the crack-tips, the stabilized tetragonal zirconia transforms to the more stable monoclinic phase with a local volume increase of approximately 4–5%.[27] The differences in sintering parameters of zirconia can directly affect its microstructure and properties.[28] The extent of this effect have become of interest in the field of dental research especially after the introduction of

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short sintering cycles by manufacturers. Several authors have studied the effect of the changes in sintering time and temperature on the translucency, grain size and biaxial flexural strength of zirconia ceramics; however, the effect of these changes on the properties of zirconia remains in question.[29–32]

Computer-aided design and computer aided manufacturing (CAD/CAM) technologies enable the milling of zirconia into reconstructions with complex geometries. Two types of zirconia milling processes are currently available, i.e. soft-milling (partially sintered state) and hard-milling (full sintered). Soft-milled frameworks are subsequently sintered to full density. Different sintering parameters may show a strong influence on the properties of the zirconia frameworks.

Computer-aided design and computer aided manufacturing (CAD/CAM) chair side systems have reduced the operation times significantly and allowed for the production of most prosthetic restorations in one visit, although zirconia needs a sintering procedure, which takes several hours. Rapid sintering procedures, which can be carried out in minutes render the production of zirconia-based restorations possible in one visit and enhance its clinical use. Hence, translucent zirconia used for monolithic restorations was also included in this study. Use of translucent zirconia has the potential to eliminate delamination of the veneering ceramic, which has been known to be a common clinical problem and also reduce the amount of tooth preparation required.[33]

The aim of this study was to investigate the effect of different sintering temperatures and durations on the flexural strength, grain size and phase transformation of zirconia. The tested null hypothesis was that the decrease in final sintering time would decrease the flexural strength.

Materials and methods

Sixty zirconium oxide and 60 highly translucent zirconium oxide bar specimens (In-Coris ZI and In-Coris TZI, Sirona Dental Systems GmbH, Bensheim, Germany) were cut using a low speed diamond saw (Isomet 1000, Buehler, IL). Then, the ZI and TZI specimens were randomly divided into three subgroups (with each group containing $n = 20$ samples) according to the sintering time and temperature. The employed parameters belong to fixed program numbers 1 – superspeed for groups C and F; 2 – speed for groups B and E; 5 – classic for groups A and D of the sintering furnace (inFire HTC Speed, Sirona, Bensheim Germany). The test groups are listed below.

Group A (ZI): Slow sintering program (regular); sintered at 1510 °C with a dwell time of 120 min. Total time is approximately 8 h.

Group B (ZI): Faster sintering program (speed); sintered at 1540 °C with a dwell time of 25 min. Total time is approximately 2 h.

Group C (ZI): Rapid sintering program (super speed); needs preheating to 1580 °C, starts at 1580 °C with a dwell time of 10 min. At the end of dwell time, furnace opens and material is immediately removed. Total time is 10 min.

Group D (TZI): Slow sintering program (regular); sintered at 1510 °C with a dwell time of 120 min. Total time is approximately 8 h.

Group E (TZI): Faster sintering program (speed); sintered at 1540 °C with a dwell time of 25 min. Total time is approximately 2 h.

Group F (TZI): Rapid sintering program (super speed); needs pre-heating to 1580 °C, starts at 1580 °C with a dwell time of 10 min. At the end of dwell time, furnace opens and material is immediately removed. Total time is 10 min (Table 1).

Three-point flexural strength tests

Three-point flexural strength (total number of specimens, $N = 120$; number of samples per group $n = 20$) was measured according to the ISO 6872:2008 standards.[34] After sintering, the final dimensions of all the specimens were 1.2 mm $\times$ 4 mm $\times$ 25 mm. Before the flexural strength test, the dimensions of the specimens were measured with a digital micrometer (Absolute Digimatic Caliper, Mitutoyo, Tokyo, Japan) to an accuracy of 0.01 mm. The specimens were then placed in the appropriate sample holder and loaded in a Universal testing machine (Shimadzu Model AGS-X, Kyoto, Japan) at a crosshead speed of 1 mm/min until failure. The specimens were tested dry at room temperature. The flexural strength was calculated according to the following formula.

$$\sigma = 3Nl/2bd^2$$

σ is the flexural strength, N is the fracture load (N), l is the distance between the supports (mm), b is the width of the specimen (mm) and d is the thickness of the specimen (mm).

Zirconia grain size

After sintering, the surfaces of all the specimens ($N = 6$, $n = 1$ per group) were polished up to a thickness of 1 μm with a diamond suspension (Struers, Bellerup, Denmark) and ultrasonically cleaned in isopropanol. Then, the specimens were dried at 50 °C for 24 h (Nüve Incubator EN 120, Ankara, Turkey) and coated with 9 nm gold-palladium particles (Cressington sputtercoater 108 auto, Cressington MTM-20, Elektronen-Optik-Service, Dortmund, Germany) and the

Table 1. Classification of the zirconia bar specimens to the groups according to sintering time.

Group	Material	Sintering temperature	Dwell time (min)	Total time
A	In-Coris ZI	1510 °C	120	8 h
B	In-Coris ZI	1540 °C	25	2 h
C	In-Coris ZI	1580 °C	10	10 min[a]
D	In-Coris TZI	1510 °C	120	8 h
E	In-Coris TZI	1540 °C	25	2 h
F	In-Coris TZI	1580 °C	10	10 min*

[a]Applied in pre-heated furnace while the others start from room temperature.

surface topography was evaluated using a scanning electron microscope (Evo LS10, Carl Zeiss, Oberkochen, Germany) at magnifications of 5000× and 15,000×.

Crystal structure analysis

Three specimens were selected randomly from each subgroup for X-ray diffraction (XRD) surface analysis to detect the amount of tetragonal and monoclinic phases present. The specimens were placed in the holder of a diffractometer (Bruker D8 advance-Lynxeye detector) and irradiated with

Cu Kα. The spectrum was recorded within the range of 0–80° at a scan time per step of 1 min. The voltage and current were set to 40 kV and 40 mA, respectively.

Statistical analysis

The data collected was checked for normal distribution and analyzed using one-way analysis of variance (ANOVA), followed by Duncan tests (SAS; Statistical Analysis System, SAS Institute Inc., Cary, NC) at a significance level of $p < 0.05$ to determine the effect of sintering time and temperature on each of the variables tested.

Results

The mean flexural strength values (MPa) and standard deviations for each group are presented in Table 2.

One-way ANOVA showed statistically significant differences between the groups ($p < 0.05$). The mean flexural strength of group C (ZI, 1580 °C sintering temperature and total 10 min sintering time) was significantly higher than group A and B. Also, the mean flexural strength of group F (TZI, 1580 °C sintering temperature and total 10 min sintering time) was significantly higher than group D and group E (Table 2).

Table 2. Mean and standard deviations (SD) values of all tested groups.

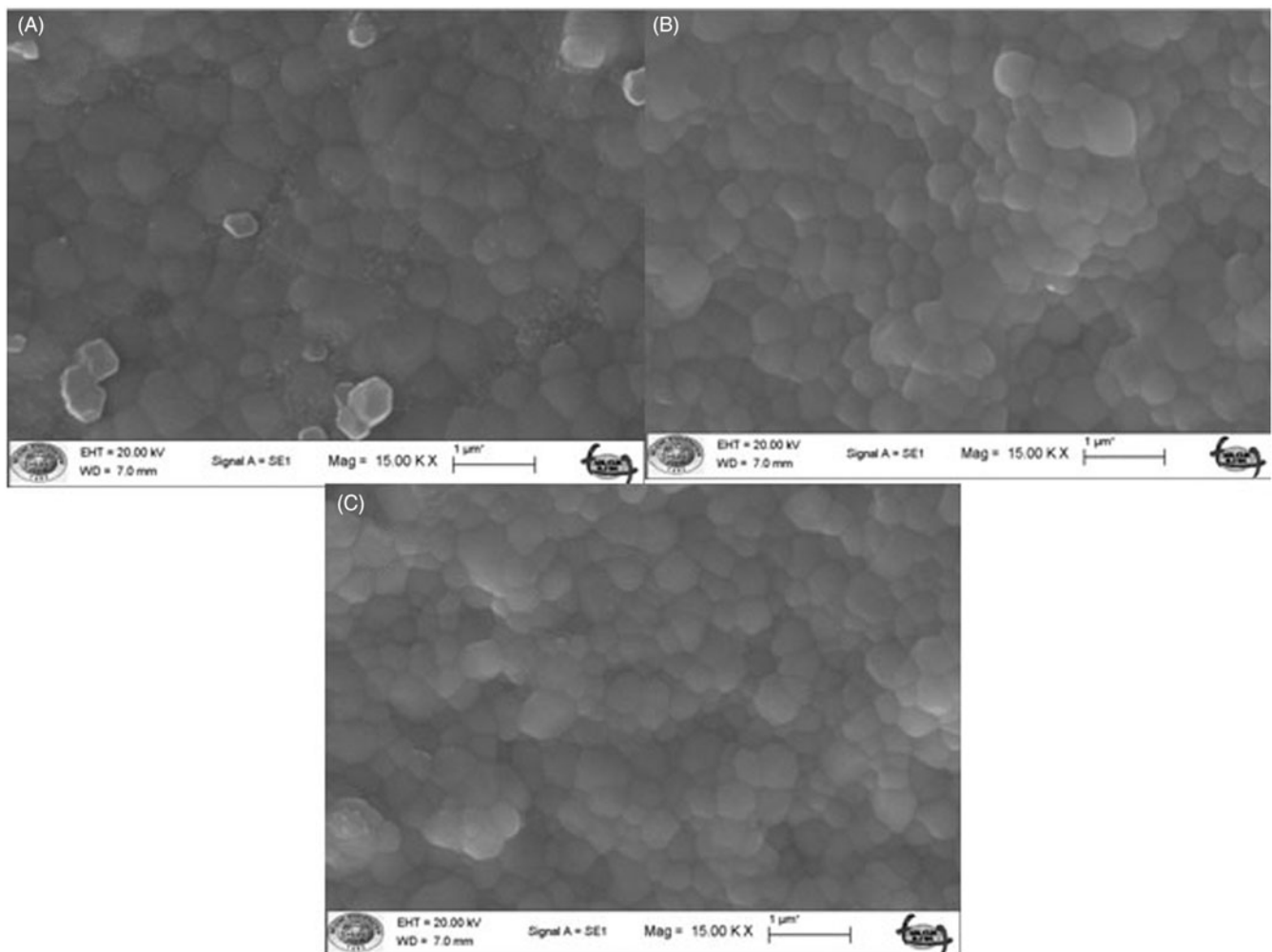| Group	Mean (MPa)	SD
Group A	700.3[a]	125.3
Group B	662.1[a]	77.8
Group C	871.8[b]	108.8
Group D	579.7[a]	140.6
Group E	622.3[a]	82.7
Group F	904.2[b]	115.7

Letters rendered in superscripts represent a significant difference.

Figure 1. In-Coris ZI grain size after different sintering temperatures and times (15,000×): (A) 1510 °C with a dwell time of 120 min; (B) 1540 °C with a dwell time of 25 min; (C) 1580 °C with a dwell time of 10 min.

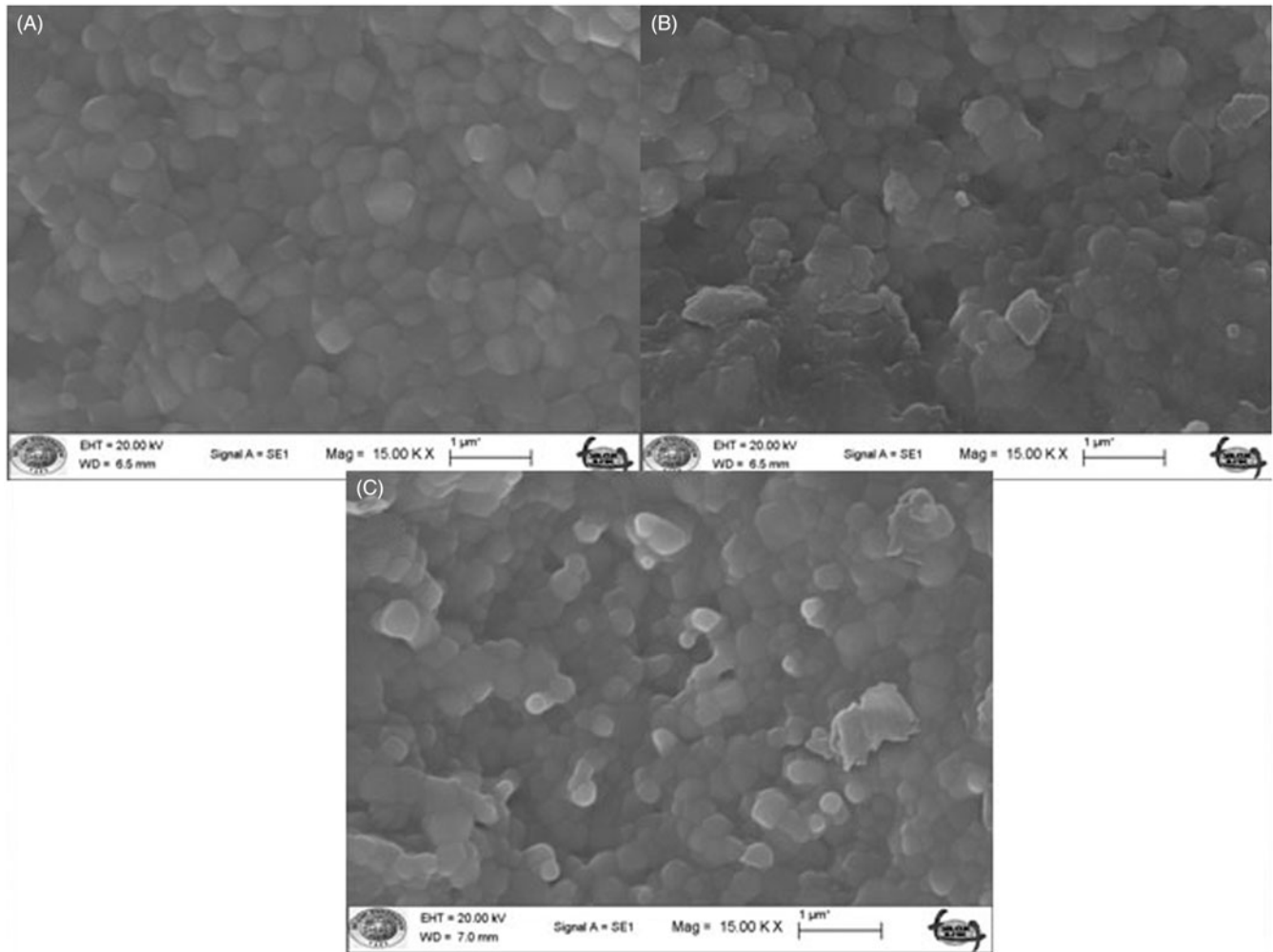Figure 2. In-Coris TZI grain size after different sintering temperatures and times (15,000×): (A) 1510 °C with a dwell time of 120 min; (B) 1540 °C with a dwell time of 25 min; (C) 1580 °C with a dwell time of 10 min.

Scanning electron microscopy (SEM) images were used to observe zirconia grain sizes. (Figures 1 and 2). The difference in grain size caused by different sintering procedures is generally small and it is difficult to see if there is a difference. In this study, no quantitative analysis was done, for this reason we could not say there were no visible difference in the grain size between the ZI and TZI groups.

The microstructure analysis of the specimens using XRD revealed that the peak positions for the spectra of the samples matches the corresponding ICSD card for tetragonal phase for ZrO_2 within the resolution of the data (PDF# 01-089-6976; (Figures 3–5).

Discussion

The 1580 °C–10 min sintering program (C and F groups) led to the highest flexural strength values for both materials. For groups A, B, D and E, the sintering temperature and time combinations did not have a significant effect on the flexural strength of zirconia. Hence, the null hypothesis is rejected.

In our study, SEM images were used to observe the grain sizes visually. However, since the visual observation alone is not enough for comparing grain sizes, quantitative analyses should be done for the determination of differences. This study was undertaken to demonstrate the possible change in strength of sintered green bar-shaped zirconia specimens by varying the sintering conditions. The main motivation behind undertaking this study was that the properties of some green milled zirconia can reach higher strengths when the sintering temperature and time are altered. Also, questioning the total phase transformation from monoclinic to tetragonal during the 10 min sintering process is also of interest. Clinically, shorter sintering times would be also beneficial for the rapid manufacturing of zirconia-based prostheses.

It is to be noted that the mechanical analysis in this study uses static loading tests and dynamic (fatigue) tests would more closely resemble clinical masticatory forces. However, there is a correlation between static and fatigue properties, analogous to studies, which describe different damage modes or strength degradation, while comparing results of cyclic loading and monotonic loading tests.[35,36] Itinoche et al. [37] found marginal differences in the flexural strength of zirconia obtained by static and cyclic loading tests, although the differences were statistically insignificant.

Crystal structural analysis revealed that all subgroups contained typical tetragonal phase grains. All specimens were completely sintered to the tetragonal phase and did not transform back to the monoclinic phase. This was predicted in the light of the absence of any physical or thermal treatment on the specimens.

Pattern: PDF 01-089-6976   Radiation: 1.54060   Quality: Indexed

Formula	Zr O2
Name	Zirconium Oxide
Name (mineral)	
Name (common)	

Lattice:	Tetragonal	Mol. weight =	123.22
S.G.:	P42/nmc (137)	Volume [CD] =	69.83
		Dx =	5.86
		Dm =	
		I/Icor =	10.150

a =	3.64000	alpha =	
b =		beta =	
c =	5.27000	gamma =	
a/b =	1.00000	Z =	2
c/b =	1.44780		

ANX: AX2
Analysis: O2 Zr1
Formula from original source: Zr O2
ICSD Collection Code: 51051
Calculated Pattern Original Remarks: Stable from 1273 to 2573 K (2nd ref., Tomaszewski), below P21/c, m.p. 2950 K. Cell of Zr.76 Y.12 Ta.12 O2 (3rd ref
, Kim & Hubbard): 3.6107(4), 5.2323(14); of Zr.66 Y.17 Ta
17 O2: 3.6177(3), 5.2513(11)
Minor Warning: No e.s.d reported/abstracted on the cell dimension. No R factors reported/abstracted
Wyckoff Sequence: d b(P42/NMCZ)
Unit Cell Data Source: Powder Diffraction

Structure
Publication: J. Solid State Chem.
Detail: volume 139, page 225 (1998)
Authors: Sham, E.L., Aranda, M.A.G., Farfan-Torres, E.M., Gottifredi, J.C., Martinez-Lara, M., Bruque, S.
Primary Reference
Publication: Calculated from ICSD using POWD-12++

Radiation:	CuKα1	Filter:	Not specified
Wavelength:	1.54060	d-spacing:	
SS/FOM:	999.9 (0,33)		

d	2θ	I	h	k	l
2.99503	29.807	999	1	0	1
2.63500	33.995	82	0	0	2
2.57387	34.828	129	1	1	0
2.13444	42.310	11	1	0	2
1.84123	49.462	329	1	1	2
1.82000	50.079	172	2	0	0
1.72030	53.201	1	2	0	1
1.58207	58.273	116	1	0	3
1.55535	59.374	217	2	1	1
1.49751	61.913	55	2	0	2
1.38489	67.589	3	2	1	2
1.31750	71.559	17	0	0	4
1.28693	73.533	40	2	2	0
1.23885	76.893	2	1	0	4
1.19401	80.352	77	2	1	3
1.18240	81.305	37	3	0	1
1.17278	82.115	29	1	1	4
1.15638	83.538	22	2	2	2
1.15107	84.011	20	3	1	0
1.10211	88.683	1	3	0	2
1.06722	92.404	30	2	0	4
1.05482	93.818	69	3	1	2
1.02411	97.557	2	2	1	4
1.01241	99.080	22	1	0	5
0.99834	100.992	21	3	0	3
0.99152	101.952	41	3	2	1
0.96279	106.274	1	3	1	3
0.94273	109.590	1	3	2	2
0.92062	113.591	21	2	2	4
0.91000	115.662	12	4	0	0
0.89251	119.326	1	3	0	4
0.88474	121.069	34	2	1	5
0.87833	122.566	5	0	0	6
0.87530	123.294	34	3	2	3
0.87070	124.428	33	4	1	1
0.86684	125.404	28	3	1	4
0.86015	127.156	12	4	0	2
0.85796	127.749	11	3	3	0
0.85383	128.890	1	1	0	6
0.83710	133.912	1	4	1	2
0.83126	135.840	15	1	1	6
0.81580	141.547	22	3	3	2
0.81393	142.310	31	4	2	0
0.80134	147.999	1	3	2	4

Figure 3. ICSD card for tetragonal phase for ZrO$_2$.

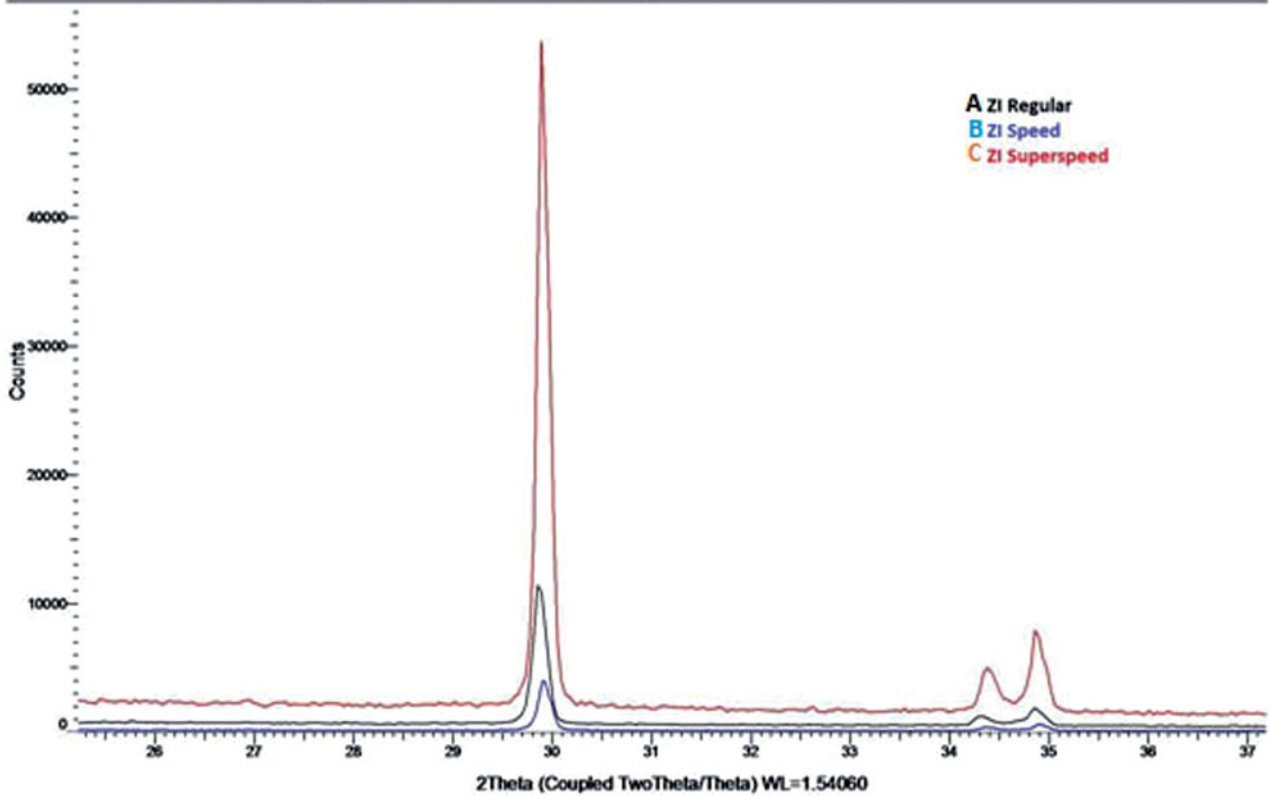**Commander Sample ID (Coupled TwoTheta/Theta)**

Figure 4. X-ray diffraction analysis of In-Coris ZI.

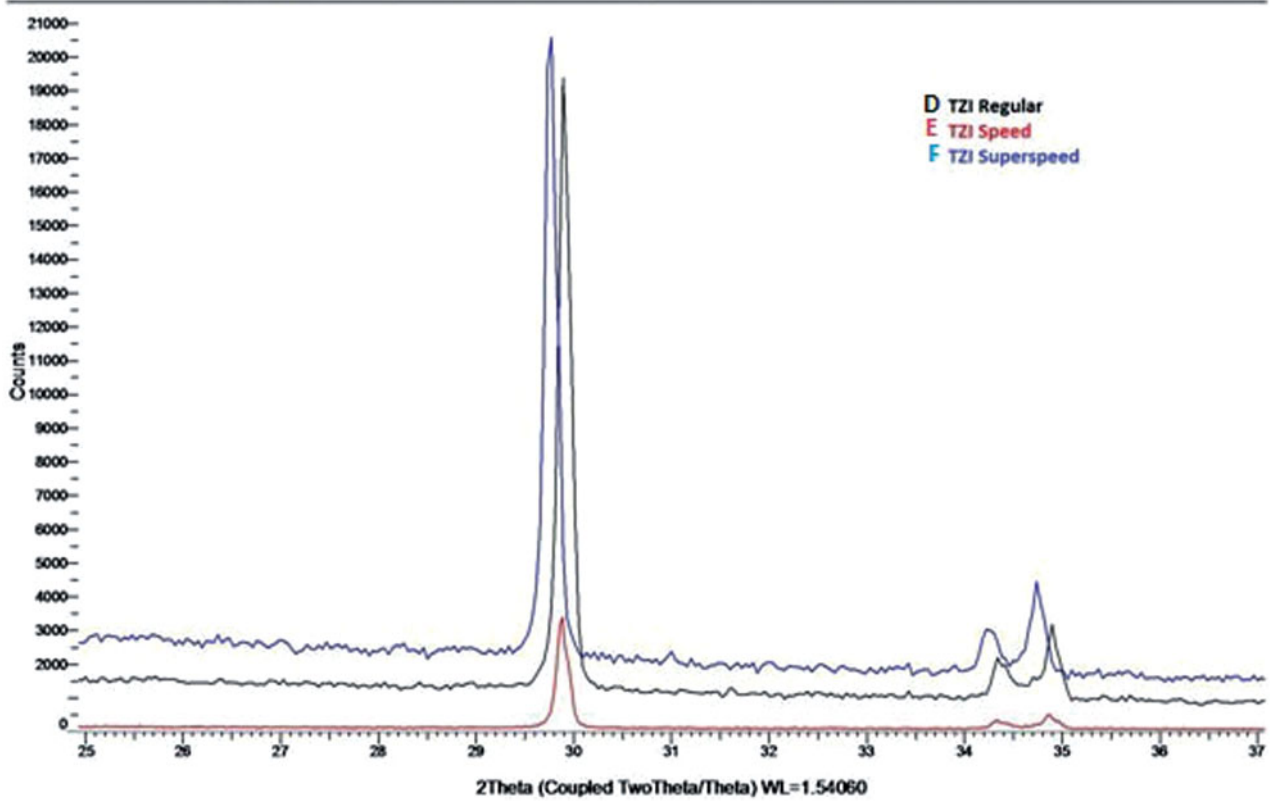**Commander Sample ID (Coupled TwoTheta/Theta)**

Figure 5. X-ray diffraction analysis of In-Coris TZI.

Stawarczyk et al. [30] reported that the grain size of zirconia increased with increase in sintering temperatures. They also reported that the sintering temperature showed a significant negative correlation with flexural strength and concluded that the sintering temperature for zirconia should be limited below 1550 °C. Our findings contradicts with their results, because we achieved higher flexural strength values with 1580 °C sintering. This difference can be explained by several reasons: the varying brands of zirconia used in both studies, our narrower temperature range and altering the sintering times with changing temperatures. It is to be noted that all our specimens were sintered as-milled and were unpolished; thus, comparing the flexural strength values obtained in this study to the values reported in other studies using polished specimens is questionable.

The flexural strength of the groups sintered at 1580 °C with a dwell time of 10 min (speed groups) were found to be significantly higher than that exhibited by the other groups (speed and regular groups) used in this study. Hjerppe et al. [38] investigated the interaction between the sintering time and static biaxial flexural strength of zirconia. According to their results, shorter sintering times did not affect the biaxial flexural strength, while having correlation to the surface composition of the samples. This can affect the durability of zirconia after water exposure, which can be clinically significant for monolithic zirconia restorations uncovered by porcelain intraorally. In our study, shorter sintering times affected flexural strength. We did not analyze the surface composition of samples; this analysis can be helpful for explaining such difference.

This study has limitations. First, only one brand of zirconia was used. The results may not be applicable for other brands with different grain sizes and different manufacturers may have special recommendations for sintering zirconia. Sintering with shortened dwell time was also influenced the density of the material but in the current study the effect of sintering on the density of the material was not studied. Further, we have used static *in vitro* tests; however, dynamic fatigue tests are more representative of clinical masticatory forces and further *in vitro* and *in vivo* tests are required. Kim et al. [29] recommended that the physical properties and marginal fitness of the coping are to be analyzed in relation to the sintering method, grain size and light transmittance. We have studied the physical properties related to the sintering method and the marginal fit and light transmittance of zirconia in correlation to the sintering method may require further investigations.

Conclusions

Based on the results obtained in this study, the following conclusions can be drawn.
(1) Zirconia samples tested showed the highest flexural strength when sintering was carried out at 1580 °C for 10 min.
(2) All experimented sintering parameters have provided full sinterization for green zirconia.

Acknowledgements

Ethical approval: This article does not contain any studies with human participants or animals performed by any of the authors.

Declaration of interest

Nuri Murat Ersoy, Hasan Murat Aydoğdu, Beyza Ünalan Değirmenci, Neslihan Çökük, Müjde Sevimay declare that they have no conflict of interest.

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