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Effect of Conventional and Rapid Sintering on the Mechanical and Hydrothermal Aging Properties of 5Y-TZP Dental Zirconia

Yena Jang, Jae Chul Bang*

Department of Materials Science and Engineering, Soonchunhyang University, 646 Eupnaeri, Asan, Chungnam 31538, Republic of Korea

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Abstract

This study investigated the effects of sintering methods on the mechanical properties and hydrothermal aging resistance of 5 mol% yttria-stabilized tetragonal zirconia polycrystals (5Y-TZP), a material widely used for dental restorations due to its aesthetic translucency. Specimens were fabricated using both conventional sintering (CS) and rapid sintering (RS) at two temperatures (1 450 °C and 1 600 °C), and their flexural strength, fracture toughness, and Vickers hardness were compared. X-ray diffraction (XRD) and SEM analyses confirmed that all sintered specimens exhibited dense microstructures with no monoclinic phase detected even after low-temperature degradation (LTD) testing for up to 20 h. CS specimens consistently demonstrated superior mechanical properties, particularly in fracture toughness and flexural strength, and exhibited higher reliability based on Weibull analysis. While RS offered shorter processing times, it showed slightly inferior mechanical properties. The results indicate that, although both sintering methods produced LTD-resistant 5Y-TZP, optimizing the RS parameters is necessary to bridge the performance gap with CS, especially for long-span prosthetic applications.

Keywords: Yttria-stabilized tetragonal zirconia polycrystals, rapid sintering, conventional sintering, low-temperature degradation, flexural strength, Weibull analysis

I. Introduction

Yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) ceramics are widely utilized in clinical applications and dental restorations due to their excellent mechanical properties and biocompatibility [1–5]. Early generations of Y-TZP ceramics, with lower yttria content (e.g. 3 mol%), offered robust mechanical performance but were limited in their aesthetic qualities due to high opacity and a gray-white appearance. This led to the development of veneering porcelain to enhance translucency. However, complications such as chipping and fractures of the veneering porcelain highlighted the need for alternatives, spurring the adoption of monolithic translucent Y-TZP restorations [2, 6–9].

Recently, 5 mol% yttria-stabilized zirconia (5Y-TZP) has gained popularity thanks to its superior translucency, which addresses aesthetic demands, despite its appreciably lower mechanical properties compared to lower yttria-content zirconia [10]. In parallel, advancements in sintering technology have introduced rapid sintering (RS) methods, enabling faster fabrication of zirconia [4, 11–13]. This innovation supports single-visit dental prosthetic treatments or chairside applications, making it a preferred manufacturing approach [13–15]. However, limited research exists on comparison of the properties of zirconia produced by means of RS with those fabricated with conventional sintering(CS) methods [16, 17].

A persistent challenge with zirconia ceramics is the phenomenon of aging or low-temperature degradation (LTD). This involves tetragonal-to-monoclinic (t→m) phase transformation under hydrothermal conditions, leading to deterioration in mechanical properties, reduced light transmission, and the formation of internal stress and microcracks [18–23]. These effects compromise the long-term stability and structural integrity of zirconia restorations [24–28].

This study aims to address these gaps by investigating the mechanical properties – such as flexural strength, fracture toughness, and hardness of 5Y-TZP zirconia fabricated by means of RS compared to CS. Furthermore, the study explores the effects of LTD on these materials to evaluate their suitability for clinical applications.

II. Materials and Methods

(1) Material preparation

The raw material used for 5Y zirconia was Zpex Smile.m (Tosoh Corp., Japan), which contained 8.89 wt% Y_2O_3 and less than 0.005 wt% Al_2O_3. The powder was placed in a circular mold and uniaxially pressed (first pressing) at 75 bar to fabricate a disc with a diameter of 102 mm and a thickness of 8.5 mm. The formed specimen was then subjected to cold isostatic pressing (CIP) at 1 600 bar for

* Corresponding author: bangj@sch.ac.kr

300 s to complete the second pressing. Pre-sintering was performed at 980 °C for 5 h. The green body was then machined into a rectangular cross-section for a three-point bending test using a CAD/CAM device (ZX5SD, Manix Corp., Korea) in accordance with ISO 6872 standards. The dimensions of the green body were designed with provision for approximate 19-% shrinkage during final sintering, resulting in final specimen dimensions of 4 mm in width, 1.5 mm in thickness, and 40 mm in length.

The sintering process was conducted using two methods: CS and RS, with sintering temperatures of 1 450 °C and 1 600 °C. CS was performed using a box furnace (DS-SK-EV-500, Daesung Corp., Korea). The heating time to the sintering temperature was 3 h, corresponding to an average heating rate of 7.9 K/min for 1 450 °C and 8.7 K/min for 1 600 °C. The dwell time at the sintering temperature was 1 h, and cooling occurred within the furnace, taking approximately 15 h to reach room temperature, which corresponds to an average cooling rate of approximately 1.6 K/min. RS was carried out in a specialized high-speed sintering furnace (AUSTROMAT 674i, Dekema Corp., Germany) with a two-step heating process: (1) 60 K/min up to 1 000 °C, followed by (2) 50 K/min up to the sintering temperature (1 450 °C or 1 600 °C). The dwell time at the sintering temperature was 15 min. Cooling was conducted at 100 K/min to 500 °C in a closed furnace, then at 200 K/min to room temperature with the furnace open. The thermal schedules for both CS and RS are summarized in Fig. 1.

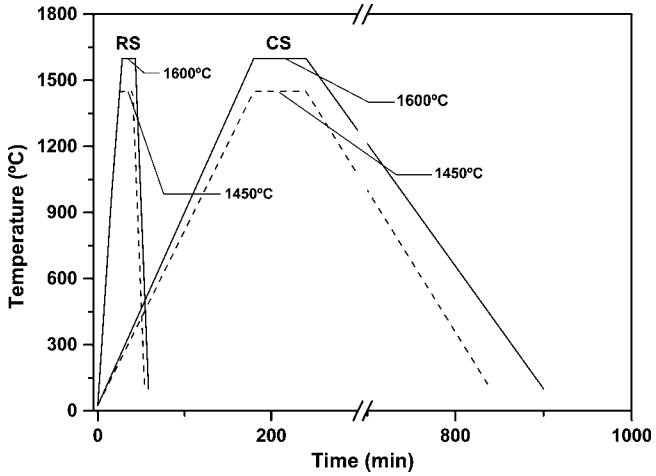

Fig. 1: Sintering profile for conventional sintering (CS) and rapid sintering (RS).

For LTD testing, *in vitro* hydrothermal aging was conducted in accordance with ISO 13356 [29]. The specimens were treated in an autoclave (3.5L-M35n, Hongik Medical Systems Corp., Korea) at 134 °C and 2 bar for 5 h and 20 h [16, 30–32], followed by drying under ambient conditions for 24 h.

(2) Phase and microstructural characterization

The crystalline phase analysis of the starting powder and sintered specimens was performed using X-ray diffraction (XRD; D/Max-2200PC, Rigaku Corp., Japan) with CuK_α radiation. The scanning 2θ range was 20° to 90° with a step size of 0.02° and a scan speed of 7 °/min [6, 16, 33, 34]. For the LTD specimens, an additional analysis was performed within the 27° to 33° 2θ range at a scan speed of 1°/min [6, 15, 35]. The microstructure of the specimens was characterized using field emission scanning electron microscope (FE-SEM; Sigma 630, ZEISS Corp., Germany). Prior to analysis, the specimens were mirror-polished, thermally etched at 1 450 °C for 20 min, and sputter-coated with platinum [36]. The microstructural features were observed at an accelerating voltage of 5 kV and a magnification of 15 000 ×. Grain size and porosity were measured using the image processing software ImageJ [37]. Grain size was determined based on the line-intercept method in accordance with ASTM E112, with a minimum of 1 000 grains counted for each condition. The porosity of the specimens was evaluated following ASTM E2109. For each condition, ten low-magnification FE-SEM images were analyzed. Pore regions were identified based on grayscale thresholding followed by particle analysis, and the pore area fraction (%) was calculated. The porosity was determined using the following equation:

$$\text{Porosity } \% = \frac{A_p}{A_t} \times 100 \quad (1)$$

where A_p is the pore area and A_t is the total analyzed area.

(3) Mechanical property characterization

The three-point flexural strength was measured for 20 specimens per sintering condition following the ISO 6872 standard. A universal testing machine (Electronic Type Universal Testing Machine (ST-1001), SALT Corp., Korea) was used with a crosshead speed of 1 mm/min (equivalent to a loading rate of 0.1634 N/s). The flexural strength (σ) was calculated using the following equation:

$$\sigma = \frac{3Pl}{2wb^2} \quad (2)$$

where P is the breaking load [N], l is the length of the support span [mm], w is the width of the specimen [mm], and b is the thickness of the specimen [mm].

The micro-hardness (H_V) and indentation fracture toughness (K_{IC}) of mirror-polished samples were measured using the indentation method with a Vickers microhardness tester (HV-120B, Mitutoyo Corp., Japan). Surface hardness was measured with a 2.94 N load (corresponding to the maximum load without crack formation) applied for 10 s, while indentation toughness was measured with a 9.81 N load (sufficient to induce crack formation) applied for 10 s. Three specimens were tested per condition, and the average value was obtained from five measurements per specimen. The equations used for H_V and K_{IC} were as follows [38, 39]:

$$H_v = 1.854 \times \frac{P}{d^2} \quad (3)$$

where P is the applied load [N] and d is the mean diagonal length of the diamond indenter [μm].

$$K_{IC} = 0.16 \times \left(\frac{c}{a}\right)^{-1.5} \times H_V \times a^{0.5} \quad (4)$$

where a is half the diagonal length of the diamond indenter [μm], and c is the distance from the center of the indenter to the crack tip [μm].

III. Results and Discussion

As described in the experimental methods, two sintering temperatures were applied in this study: 1 450 °C and 1 600 °C. The temperature of 1 600 °C was selected based on the manufacturer's recommendations, whereas 1 450 °C was chosen on account of preliminary experiments indicating superior mechanical properties. Both temperatures also align with the optimal sintering temperature range reported by various researchers studying Y-TZP ceramics [14, 16, 17, 38].

Fig. 2 presents the XRD phase analysis results of the raw material powder before sintering. Tetragonal, cubic, and monoclinic phases were observed. The Zpex Smile.m powder used in this study contained 8.89 wt% Y_2O_3 and less than 0.005 wt% Al_2O_3, which corresponds to a slightly reduced amount of the stabilizer Y_2O_3 and a minimized amount of Al_2O_3 compared to the previous commercial version, Zpex Smile (Tosoh Corp., Japan), which contained 9.26 wt% Y_2O_3 and 0.048 wt% Al_2O_3. This adjustment was aimed at increasing the amount of the tetragonal phase to enhance strength, while minimizing Al_2O_3, which contributes opacity [6, 20, 33, 37], thus maintaining translucency. However, since Al_2O_3 is known to improve resistance to LTD [37, 40, 41], aging tests were necessary to evaluate potential degradation.

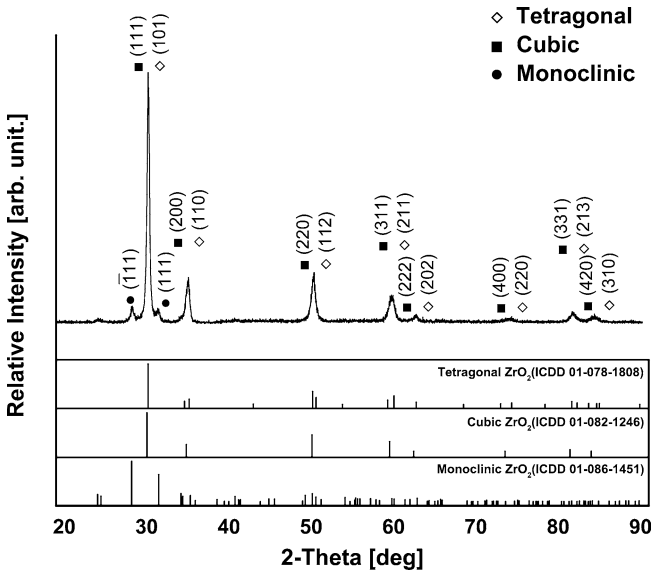

Fig. 2: XRD patterns of the raw material powder before sintering.

Fig. 3 shows the XRD phase analysis results for specimens fabricated by means of CS and RS at both sintering temperatures, before and after aging. In the figure, specimens are labeled as follows: sintering temperature and holding time are denoted as temperature-time, and LTD-treated specimens as LTD-time. The same

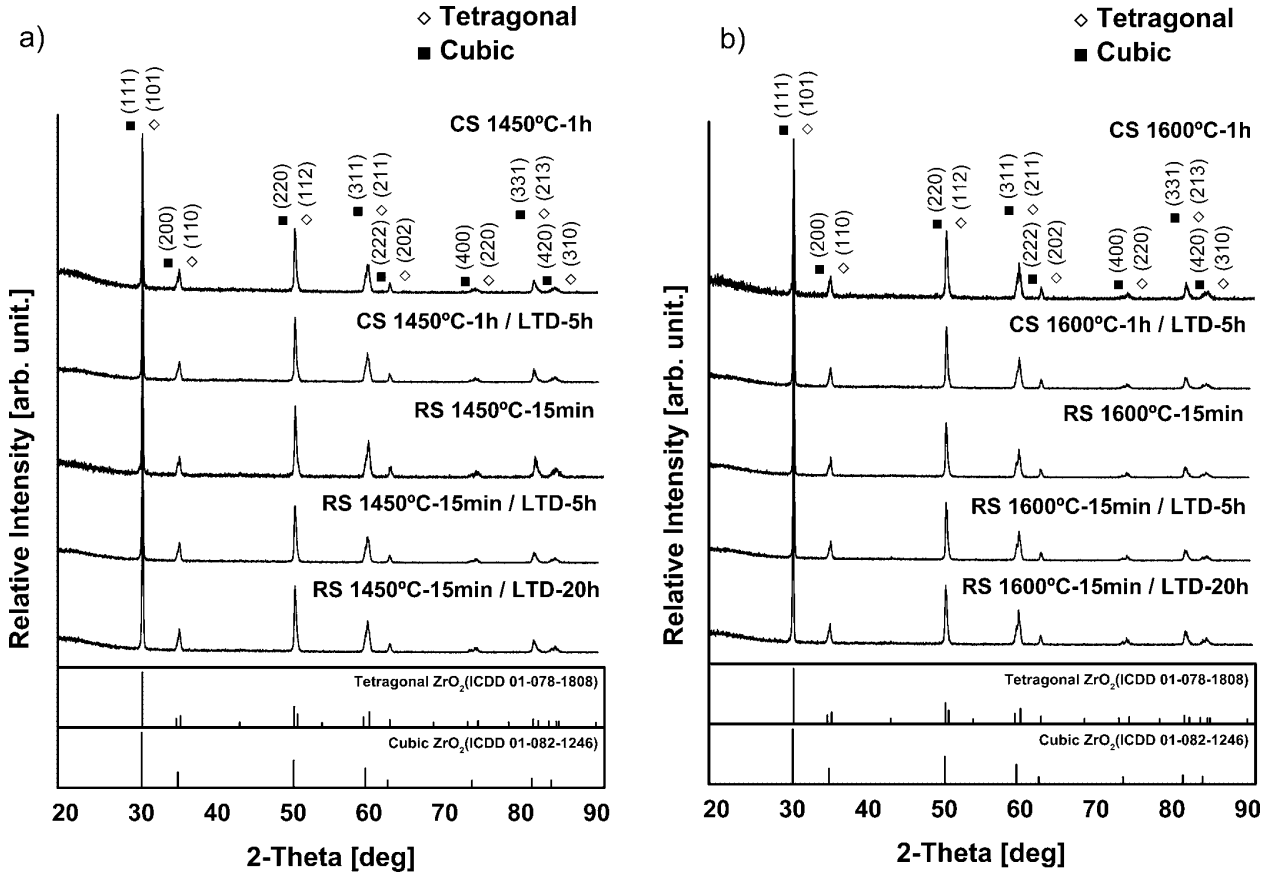

Fig. 3: XRD patterns of CS and RS specimens sintered at (a) 1 450 °C and (b) 1 600 °C, before and after LTD.

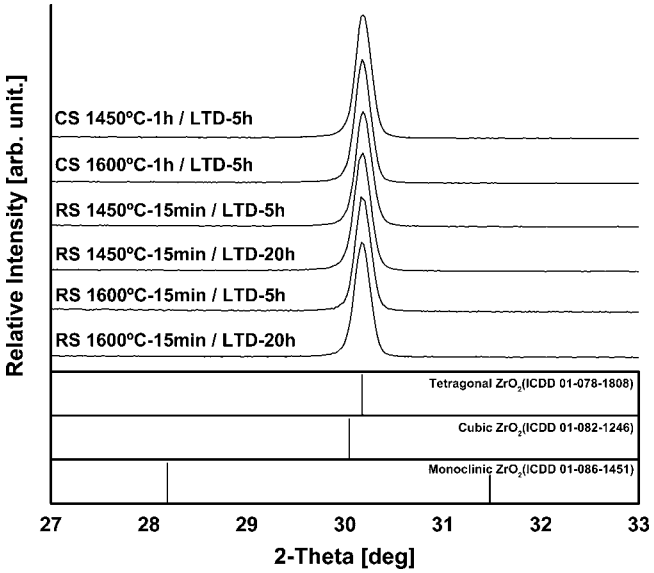

Fig. 4: XRD patterns of LTD-treated specimens fabricated by CS and RS at two sintering temperatures – 1 450 °C and 1 600 °C – focused on the 2θ range of 27 – 33°.

notation is used throughout the subsequent tables and figures. All specimens exhibited identical phase compositions, a mixture of tetragonal and cubic phases, with no monoclinic phase observed for raw material powder, regardless of sintering temperature, method (CS or RS), or aging status. Monoclinic phase formation would indicate hydrothermal degradation through tetragonal-to-monoclinic phase transformation, correlating with degradation severity. Notably, even RS specimens subjected to extended aging (20 h) showed no monoclinic phase. Additional analysis was performed with reduced scan speed over the 2θ range of 27°– 33°, as depicted in Fig. 4. No diffraction peaks corresponding to monoclinic phase at 2θ values of 28.190° (-111) and 31.482° (111) were detected. According to Garvie and Nicholson, the volume fraction of the monoclinic phase can be estimated from the diffraction intensities of the (-111) and (111) monoclinic peaks and the (101) tetragonal peak [41]. However, since no monoclinic peaks were observed, such calculations were not applicable. Considering 1 h of aging at 134 °C and 2 bar steam equates roughly to 4 years *in vivo* at body temperature (37 °C) [43, 44], the 20-h LTD tests conducted correspond approximately to 80 years *in vivo*. Therefore, it can be concluded that the current composition is highly resistant to hydrothermal degradation, independent of sintering temperature or method (CS or RS).

Fig. 5 presents SEM micrographs of specimens processed by CS and RS at two sintering temperatures, before and after LTD. All specimens displayed dense microstructures with no observable microcracks. Alumina precipitates were not detected, consistent with the extremely low Al_2O_3 content added to the 5Y-TZP raw material. Alumina typically segregates at grain boundaries [45], and while 8 mol% yttria-stabilized zirconia reportedly has an alumina solubility of approximately 1 wt% [46], the significantly lower alumina content (< 0.005 wt%) used here suggests complete dissolution within grain boundaries.

Grain size and porosity values measured from FE-SEM images (Fig. 5) are summarized in Table 1. Significant grain growth was observed at 1 600 °C compared to 1 450 °C, with more pronounced growth in the CS specimens. At identical sintering temperatures, CS consistently exhibited larger grain sizes, particularly at 1 600 °C. These results differ from previous reports by Nonaka *et al.* [48] and Cokic *et al.* [13], who observed larger grains in RS specimens compared to CS [49]. The porosity showed a slight decrease at 1 600 °C compared to 1 450 °C; however, when compared at the same sintering temperature, the RS specimens exhibited nearly twice the porosity of the CS specimens. Since microstructural characteristics, particularly grain size and porosity, are highly sensitive to sintering parameters such as temperature, heating rate, holding time, and the heating method (e.g. SiC or microwave furnaces) [9, 50–54], the discrepancies observed in certain microstructural aspects among studies are most likely attributable to differences in the furnace and sintering conditions employed. Meanwhile, no significant changes in grain size or porosity were observed after LTD treatments.

Table 1: Grain size and porosity of CS and RS specimens sintered at 1 450 °C and 1 600 °C, before and after LTD.

	Grain size (μm) (Mean ± SD)	Porosity (%) (Mean ± SD)		Grain size (μm) (Mean ± SD)	Porosity (%) (Mean ± SD)
CS 1450 °C-1h	0.45 ± 0.03	0.55± 0.02	CS 1600 °C-1h	1.19 ± 0.12	0.44± 0.02
CS 1450 °C-1h/LTD-5h	0.43 ± 0.03	0.54± 0.03	CS 1600 °C-1h/LTD-5h	1.19 ± 0.12	0.45± 0.03
RS 1450 °C-15min	0.43 ± 0.05	0.93± 0.03	RS 1600 °C-15min	0.97 ± 0.12	0.89± 0.02
RS 1450 °C-15min/LTD-5h	0.42 ± 0.04	0.94± 0.04	RS 1600 °C-15min/LTD-5h	0.98 ± 0.11	0.90± 0.04
RS 1450 °C-15min/LTD-20h	0.42 ± 0.02	0.93± 0.04	RS 1600 °C-15min/LTD-20h	0.97 ± 0.12	0.89± 0.03

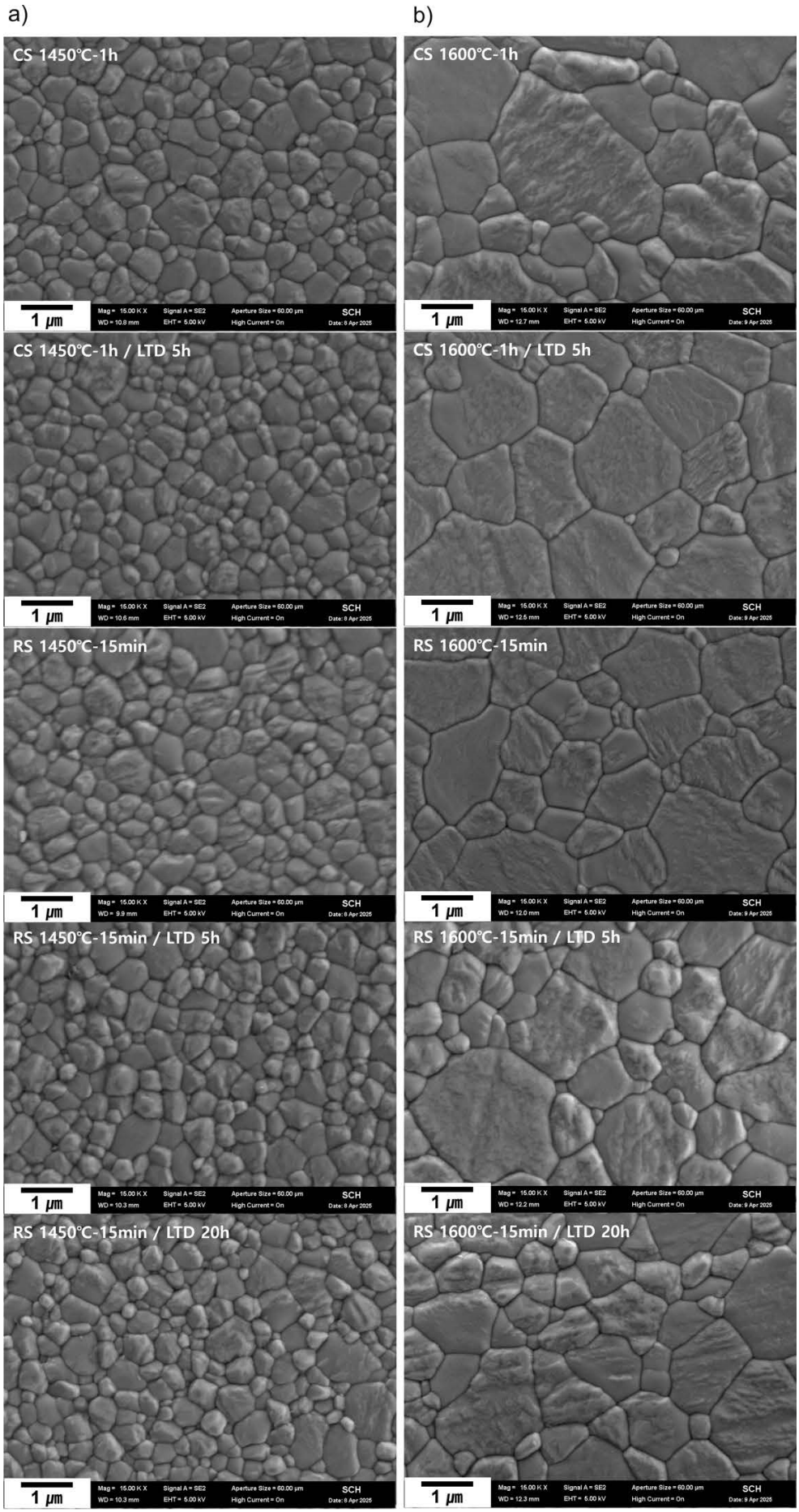

Fig. 5: FE-SEM micrographs of polished and thermally etched surfaces of CS and RS specimens sintered at (a) 1 450 °C and (b) 1 600 °C, before and after LTD.

Table 2 summarizes Vickers hardness and fracture toughness measurements for CS and RS specimens at both sintering temperatures, before and after LTD. Hardness values were slightly higher in RS specimens, with no apparent correlation to sintering temperature or LTD treatment. Fracture toughness was greater in CS specimens, showing negligible variation with sintering temperature, but demonstrated a slight decrease after LTD.

Fig. 6 presents the three-point flexural strength results of CS and RS specimens at both sintering temperatures, before and after LTD, including the average flexural strength (σ), Weibull modulus (m), and characteristic strength (σ_0) at a fracture probability of 63.2 %. The corresponding values are summarized in Table 3. Overall, all measured parameters (σ, m, and σ_0) were higher for the CS specimens and for the specimens sintered at the lower temperature (1 450 °C). A slight decrease in all parameters was observed after LTD treatment. These differences can be primarily attributed to the porosity characteristics summarized in Table 1. At identical sintering temperatures, the CS specimens exhibited nearly half the porosity of the RS specimens, which likely resulted in a reduced population of critical defects and consequently higher flexural strength and reliability. In contrast, for specimens sintered at 1 600 °C, both CS and RS showed reduced mechanical performance compared to those sintered at 1 450 °C, which is likely associated with grain growth accompanied by the coarsening of residual pores, leading to a deterioration in mechanical properties. These results are consistent with previous studies by Nonaka *et al.* [48] and Too *et al.* [16], which demonstrated that not only the amount but also the size of residual pores plays a critical role in governing the mechanical and optical (translucency) properties of TZP ceramics.

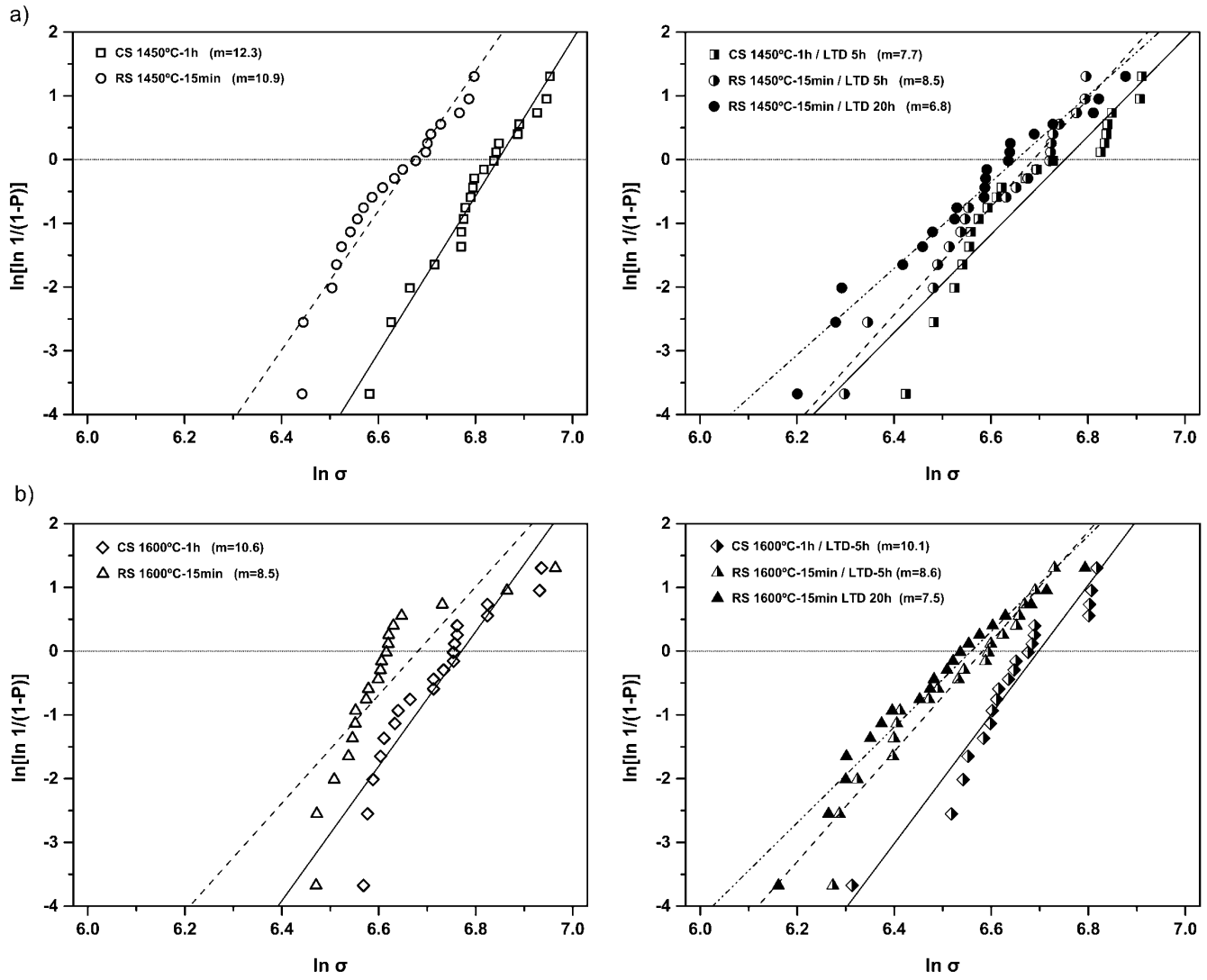

Fig. 6: Weibull modulus for the flexural strength of specimens sintered at (a) 1 450 °C and (b) 1 600 °C, before and after LTD.

Table 2: Vickers hardness (H_V) and fracture toughness (K_{IC}) of specimens sintered at 1 450 °C and 1 600 °C, before and after LTD.

	H_V **(Mean ± SD)**	$K_{IC}(MPa\sqrt{m})$ **(Mean ± SD)**		H_V **(Mean ± SD)**	$K_{IC}(MPa\sqrt{m})$ **(Mean ± SD)**
CS 1450 °C-1h	1343 ± 31.7	3.97 ± 0.2	CS 1600 °C-1h	1348 ± 21.2	3.90 ± 0.1
CS 1450 °C-1h/LTD-5h	1411 ± 12.2	3.73 ± 0.1	CS 1600 °C-1h/LTD-5h	1402 ± 25.4	3.53 ± 0.2
RS 1450 °C-15min	1400 ± 16.6	3.31 ± 0.2	RS 1600 °C-15min	1378 ± 15.3	3.41 ± 0.1
RS 1450 °C-15min/LTD-5h	1431 ± 35.8	3.48 ± 0.2	RS 1600 °C-15min/LTD-5h	1389 ± 1.0	3.22 ± 0.1
RS 1450 °C-15min/LTD-20h	1379 ± 13.4	3.22 ± 0.1	RS 1600 °C-15min/LTD-20h	1360 ± 5.6	3.18 ± 0.1

Table 3: Flexural strength (σ), Weibull modulus (m), and characteristic strength (σ_0) of specimens sintered at 1 450 °C and 1 600 °C, before and after LTD.

	σ (MPa) (Mean ± SD)	**m**	**σ_0 (MPa)**		**σ (MPa) (Mean ± SD)**	**m**	**σ_0 (MPa)**
CS 1450 °C-1h	903 ± 88	12.3	932	CS 1600 °C-1h	831 ± 92	10.6	850
CS 1450 °C-1h/LTD-5h	805 ± 123	7.7	849	CS 1600 °C-1h/LTD-5h	722 ± 88	10.1	831
RS 1450 °C-15min	756 ± 82	10.9	810	RS 1600 °C-15min	751 ± 99	8.5	798
RS 1450 °C-15min/LTD-5h	758 ± 104	8.5	808	RS 1600 °C-15min/LTD-5h	683 ± 94	8.6	716
RS 1450 °C-15min/LTD-20h	723 ± 127	6.8	774	RS 1600 °C-15min/LTD-20h	663 ± 107	7.5	706

Although ISO 6872 does not specify explicit hardness requirements for dental ceramics, it does define minimum requirements for flexural strength and fracture toughness. According to ISO 6872, prostheses involving four or more units must exhibit a minimum flexural strength of 800 MPa and fracture toughness of 5.0 $MPa{\cdot}m^{1/2}$, while three-unit prostheses involving molars require at least 500 MPa and 3.5 $MPa{\cdot}m^{1/2}$, respectively. Consequently, the specimens in this study did not meet the requirements for prostheses involving four or more units. While CS specimens satisfied the criteria for three-unit prostheses involving molars, RS specimens fell slightly short in terms of fracture toughness. These findings suggest that while RS provides promising processing efficiency, further refinement of the sintering parameters is necessary to consistently achieve the desired balance between mechanical performance and hydrothermal stability in 5Y-TZP.

IV. Conclusions

This study compared the effects of conventional sintering (CS) and rapid sintering (RS) on the mechanical properties and hydrothermal aging behavior of a commercial 5Y-TZP dental zirconia. Two representative sintering temperatures (1 450 °C and 1 600 °C) were examined to evaluate the trade-offs between processing efficiency and mechanical reliability. Compared with RS, CS consistently produced higher flexural strength, Weibull modulus, and characteristic strength, particularly at the lower sintering temperature of 1 450 °C. These differences were primarily associated with microstructural characteristics, as CS specimens exhibited lower porosity at identical sintering temperatures, which likely resulted in improved mechanical reliability. At 1 600 °C, both CS and RS specimens showed reduced mechanical performance, which is likely associated with grain growth accompanied by the coarsening of residual pores, leading to a deterioration in mechanical properties. Low-temperature degradation (LTD) tests revealed no detectable phase transformation or significant microstructural changes, even after extended aging, indicating high resistance to hydrothermal degradation regardless of sintering method or temperature. Nevertheless, the slight decrease in mechanical properties after LTD suggests that the extent of aging cannot be adequately assessed based on phase analysis alone. From an application perspective based on ISO6872 criteria, CS specimens satisfied the requirements for three-unit prostheses involving molars, whereas RS specimens marginally failed to meet the fracture toughness requirement despite exhibiting comparable flexural strength. These results demonstrate that while RS offers substantial advantages in processing time and efficiency, careful optimization of sintering parameters is required to achieve a balance between mechanical performance, reliability, and hydrothermal stability in 5Y-TZP dental zirconia.

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