Post-hot isostatic pressing: A healing treatment for process related defects and laboratory grinding damage of dental zirconia?

EMPA Materials Science & Technology

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Objective

Processing parameters (powder granulation, compaction, debinding, greenbody shaping, sintering) and post-sinter rough, even fine grinding are influencing the final mechanical properties of 3Y-TZP. The hypothesis of this study was that post-sinter hot isostatic pressing (post-HIP) would be beneficial for improving reliability and strength of both sintered and coarse ground sintered zirconia by closing or reducing surface and/or small volume defects.

Methods

75 sintered bars of an experimental 3Y-TZP (3 mm × 4 mm × 45 mm) with chamfered edges and 15 µm diamond surface finish were provided by the manufacturer (Ivoclar Vivadent) and randomly distributed in five groups of $N=15$ each. G1 served as control (as received); G2 was post-HIPed at 1400 °C and G3 at 1350 °C, both using a pressure of 195 MPa in Ar for 1 h; G4 was coarse ground with 120 µm diamond disk grain size; G5 was ground 120 µm and post-HIPed at 1350 °C at 195 MPa, 1 h in Ar. The specimens were fractured in air in 4 point-bending. Weibull characteristic strength (σ_0), m parameter (reliability) and confidence intervals (CI) at 90% confidence level are reported. Identification of the critical flaw was performed by SEM on the fractured surface of all specimens and XRD performed in all groups.

Results

G5 had a significantly lower σ_0 than G1. No significant differences were seen in the reliability (m) among the groups. Fractography revealed critical intrinsic subsurface flaws of 10–60 µm present in all groups resulting from the processing parameters. No «healing» (i. e. closing of defects by densification) resulted after post-HIP. Grinding sintered zirconia with 120 µm diamond disks induced radial cracks of 10–20 µm and an important pseudo-cubic phase transformation (56 wt%) that was not completely removed after post-HIP. Post-HIP increased slightly the relative density by 0.1% but without improving the strength and reliability.

Conclusion

- Post-sinter HIP could not close existing critical processing subsurface flaws in the size range of 10–60 µm.
- Surface grinding with 120 µm diamond grain sizes introduced radial cracks which were smaller than the processing defects of this experimental ceramic.
- With the post-sinter and post-grinding, it is possible to partially transform the pseudo-cubic phase to tetragonal.

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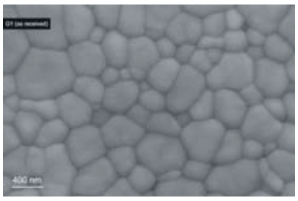
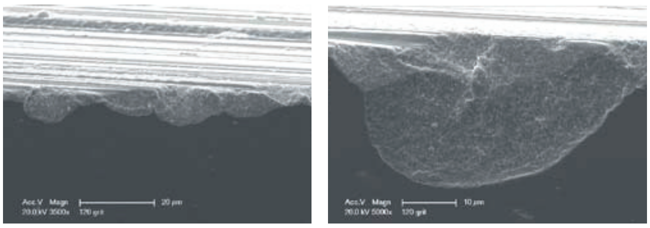
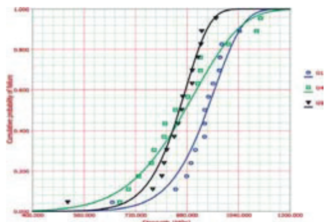


	Density (g/cm³)	Rel. density (% TD)	Grain size (nm)
G1	6.067 ± 0.002	99.62 ± 0.04	476 ± 73
G2	–	–	499 ± 68
G3	6.073 ± 0.001	99.72 ± 0.02	475 ± 65
G4	6.066 ± 0.001	99.60 ± 0.02	–
G5	6.074 ± 0.002	99.74 ± 0.03	–

SEM images of mirror polished and thermally etched specimen LG1.

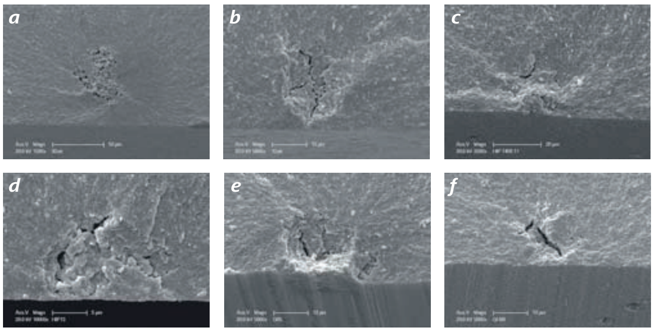


120 grit grinding damage (120 µm diamond bur) on 3Y-TZP. Damage in the form of chips (radial cracks) are between 10 and 20 µm in depth.

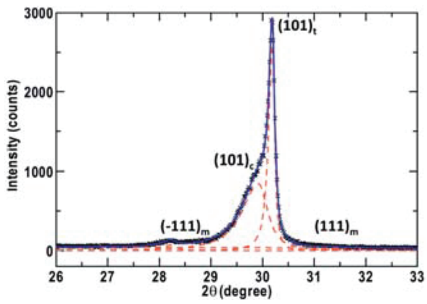


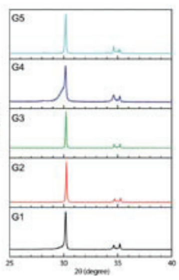
	Weibull (σ_0) + CI (MPa)	m (reliability) + CI
G1	973 (932–1016)	10.6 (7.45–15.1)
G2	930 (871–995)	6.90 (4.87–9.9)
G3	898 (848–952)	7.94 (5.6–11.4)
G4	921 (857–991)	6.35 (4.48–9.11)
G5	881 (847–918)	11.4 (8.03–16.3)

Weibull cumulative probability of failure distribution (left). Statistically significant difference between G1 and G5 for σ_0 but not m.



a, b: processing (i. e. pressing and sintering) flaws of G1 in the order of 20–40 µm.
c, d: processing flaws which were not closed by HIP (G2: 1400 °C; G3: 1350 °C).
e: flaw nearby a ground 120 µm surface (G4) which is related to powder pressing and sintering.
f: subsurface processing flaw in G5 (grinding 120 µm followed by HIP1350 °C).





(rigth) XRD patterns. The composition changes among the samples G1, G4, G5 is clearly visible. Grinding (G4) broadened the base of the tetragonal peak dramatically as expressed by the large amount of pseudo-cubic phase (56 wt%). HIP at 1350 °C after grinding lowered the amount of pseudo-cubic to 10 wt%.

(left) Fitting example for G4 using asymmetric Lorentzian functions with linear background function.

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