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The effect of silica sol infiltration on the properties of dental 3Y-TZP ceramics

Anastasia Samodurova

Study programme: Nanosciences and Nanotechnologies,
Jožef Stefan International Postgraduate School

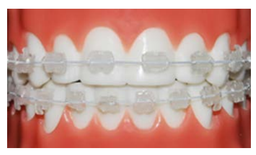
Supervisor: prof. dr. Tomaž Kosmač

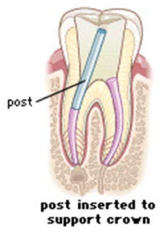
Jožef Stefan Institute, Jamova 39, 1000 Ljubljana

Dental zirconia

Early 1990s

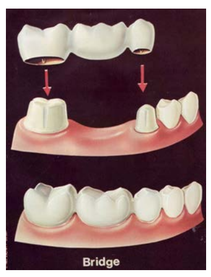
- Orthodontic brackets
- Root posts for anterior teeth





Nowadays

- Fixed partial dentures
- Implants



Why zirconia?

- High strength and fracture toughness
- Aesthetics
- Biocompatibility

Problems:

- Porcelain chipping
- Low temperature degradation (LTD), i.e. ageing

Aim

To enhance the ageing resistance of Y-TZP by infiltration with silica sol without decreasing mechanical properties

Materials and methods

TZ-3YB
TZ-PX-242A
(Tosoh, Japan)

→ Uni-axial dry pressing, 150 kN, Ø 20mm

→ Pre-sintering, 900°C

Infiltration with silica sol

Infiltration with silica sol, synthesized *in situ* by the sol–gel method through hydrolysis of dynasylan. Specimens were immersed into a mixture of absolute ethanol and dynasylan. The hydrolysis was carried out by dropwise adding of an aqueous ammonia (%) at room temperature.

→ Final sintering, 1450°C, 4h

→ Autoclaving in water: 134°C, up to 48h

→ Characterization XRD, TEM, SEM

Results

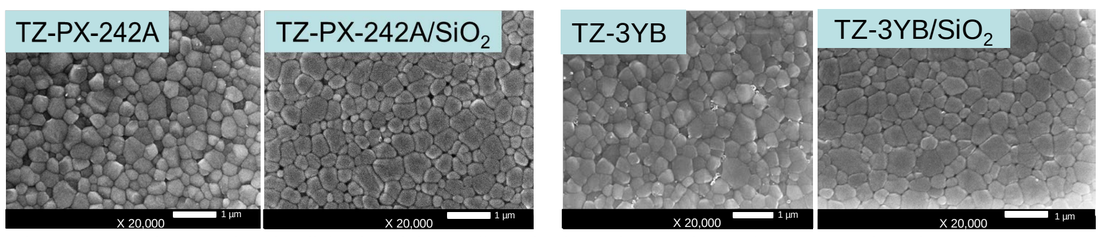


Fig. 1. SEM micrographs of polished and thermally etched specimens sintered at 1450 °C for 4 h.

Relative densities, %

	TZ-PX-242A	TZ-3YB
As sintered	98.6 ± 0.3	96.0 ± 0.7
SiO_2-doped	99.2 ± 0.5	96.5 ± 0.4

Average grain sizes, µm

	TZ-PX-242A	TZ-3YB
As sintered	0.29 ± 0.03	0.31 ± 0.04
SiO_2-doped	0.28 ± 0.02	0.32 ± 0.04

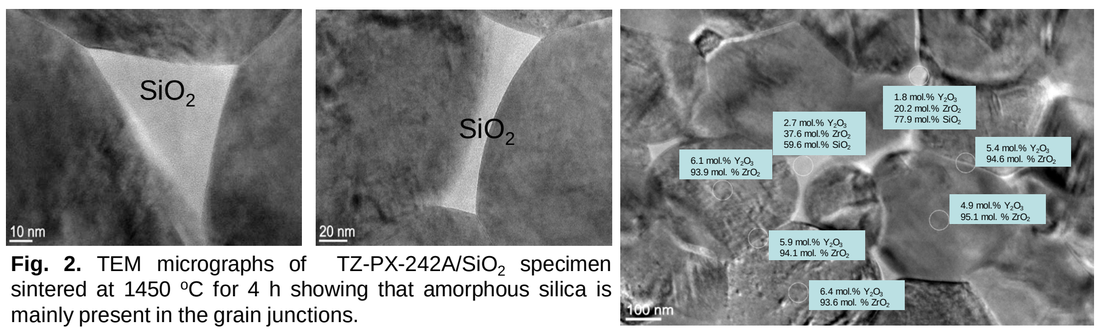


Fig. 2. TEM micrographs of TZ-PX-242A/SiO_2 specimen sintered at 1450 °C for 4 h showing that amorphous silica is mainly present in the grain junctions.

Mechanical properties

Sample	Flexural strength, MPa	Hardness, H_v (GPa)	Fracture toughness, K_{IC} (MPa·$m^{1/2}$)
TZ-PX-242A	1072 ± 48	15.2 ± 0.5	4.5 ± 0.7
TZ-PX-242A/SiO_2	1150 ± 150	14.1 ± 0.4	4.2 ± 0.3
TZ-3YB	1051 ± 136	14.5 ± 0.5	4.4 ± 0.3
TZ-3YB/SiO_2	1076 ± 114	13.9 ± 0.3	4.3 ± 0.1

In-vitro ageing behavior

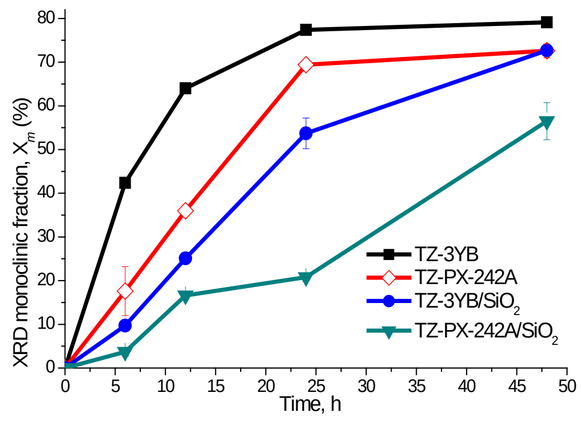


Fig. 3. Calculated monoclinic fraction versus in-vitro ageing time for monolithic and silica doped TZ-PX-242A and TZ-3YB materials.

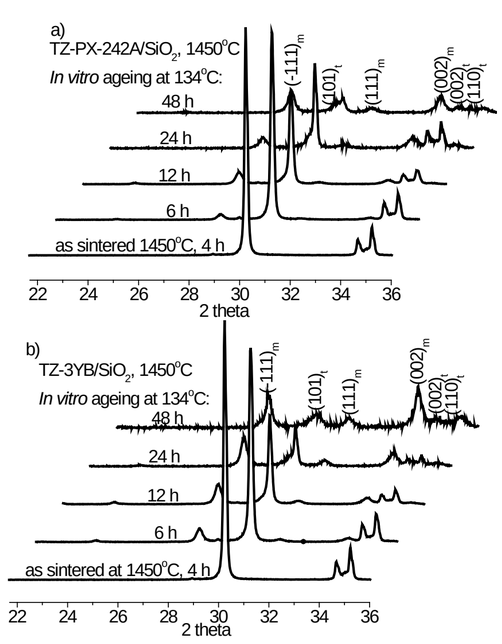


Fig. 4. XRD patterns obtained from a) SiO_2-TZ-PX-242A and b) SiO_2-TZ-3YB ceramic surfaces, sintered for 4 h at 1450 °C and aged in water at 134 °C for 6, 12, 24 and 48 h.

Conclusions

- Silica doped 3Y-TZP ceramics with the same grain size were prepeared by the pressureless infiltration of pre-sintered specimens with silica sol synthesized *in situ* by the sol-gel method
- The results of TEM analysis revealed that silica was mainly present as an amorphous phase concentrated at grain junctions
- The presence of silica substantially improves the LTD resistance without decreasing mechanical properties.