cbecimat26
26° Congresso Brasileiro de Engenharia e Ciência dos Materiais
22 a 26 de novembro de 2026
Costão do Santinho
Florianópolis-SC

Laser Texturing and Bioactive Glass Infiltration as Combined Surface Treatments on Zirconia-Based Ceramics: Effects on Morphology, Physicochemical Properties, and Cellular Viability

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Introduction

- **3Y-TZP** combines high strength (900–1200 MPa) with transformation toughening, but is prone to **low-temperature degradation** in humid physiological media.
- **$ZA_8Sr_8Ce_{11}$** (84 vol% Ce-TZP / 8 vol% Al_2O_3 / 8 vol% $SrAl_{12}O_{19}$) offers superior ageing resistance and damage tolerance [1].
- Both are **bioinert**: surface engineering is needed for cell attachment and osseointegration.
- **S53P4 bioactive glass** releases Si, Ca, Na and P ions that favour hydroxyapatite formation [2]; **picosecond Direct Laser Writing (DLW)** creates reproducible micro-grooves with minimal thermal damage [3].

Objectives

Evaluate the morphology, groove topography, crystalline phases, wettability and fibroblast viability of 3Y-TZP and $ZA_8Sr_8Ce_{11}$ after **individual and combined** sandblasting, S53P4 infiltration and ps-DLW, including the effect of **treatment sequence**.

Methodology

- Uniaxial pressing (250 MPa, 60 s); sintering 3Y-TZP 1500 °C/2 h and $ZA_8Sr_8Ce_{11}$ 1450 °C/1 h → discs Ø 8 × 2 mm.
- **7 surface conditions × 2 materials = 14 groups:**

As-Sinter	Sintered			
Blast	Sintered	› Blast		
BG	Sintered	› S53P4 BG		
Blast-BG	Sintered	› Blast	› S53P4 BG	
DLW	Sintered	› ps-DLW		
DLW-BG	Sintered	› ps-DLW	› S53P4 BG	
BG-DLW	Sintered	› S53P4 BG	› ps-DLW	

Blast: 4 bar, 30 s. BG: S53P4 slurry (50 µL), infiltrated at 1500 °C/2 h.

ps-DLW (Edgewave PX200, Nd:YVO_4)	3Y-TZP	$ZA_8Sr_8Ce_{11}$
λ / τ / f	532 nm / 10 ps / 10 kHz	532 nm / 10 ps / 10 kHz
Spot / line pitch	30 µm / 30 µm	30 µm / 30 µm
Fluence / scan speed	2.79 J/cm² / 1 mm/s	2.17 J/cm² / 2 mm/s

- **Characterization:** SEM/EDS; white-light interferometry (Zygo NewView 7300); XRD (Rigaku MiniFlex600); water contact angle (5 µL); MTS assay with L929 fibroblasts (24 h, OD 490 nm); ANOVA + Tukey (α = 0.05).

Results and Discussion

Surface morphology and chemistry (SEM/EDS)

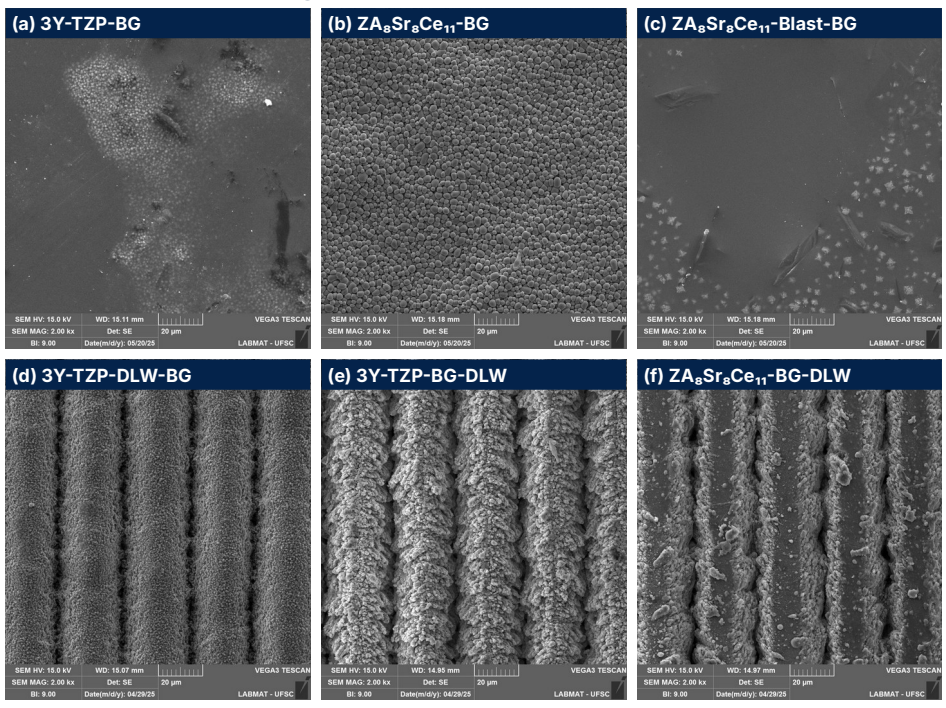


Fig. 1 – SEM micrographs (2.00 kx, scale bar 20 µm).

- **BG alone:** Si-rich glassy layer on 3Y-TZP (25.3 wt% Si); on $ZA_8Sr_8Ce_{11}$ the glass infiltrates the open porosity, unless blasting precedes it (residual film, 18.0 wt% Si).
- **Sequence matters:** DLW-BG coats ridges and grooves with sub-µm glass grains; in BG-DLW the laser partly removes the glass, leaving glass-rich crests (13.9 wt% Si).

Laser groove topography

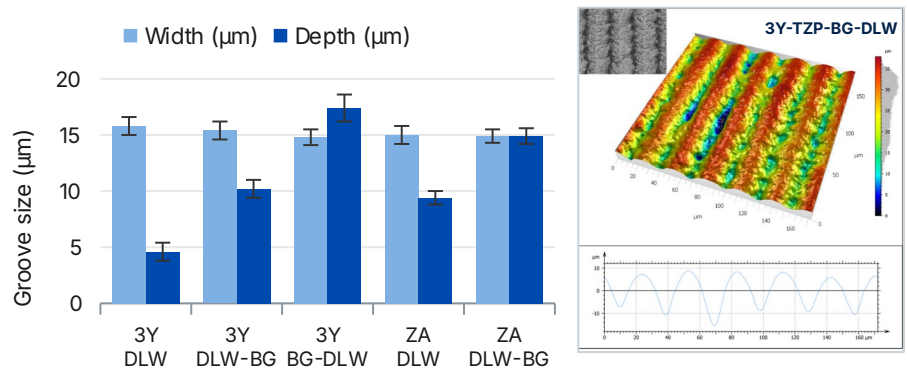


Fig. 2 – Groove width/depth (mean ± SD; 3Y = 3Y-TZP, ZA = $ZA_8Sr_8Ce_{11}$) and 3D topography of 3Y-TZP-BG-DLW (inset: SEM).

- **Width ≈ 15 µm** in all groups (set by the optics); **depth** depends on material and sequence: 4.6 → **17.4 µm** on 3Y-TZP when BG precedes DLW (silicate phase lowers the ablation threshold); $ZA_8Sr_8Ce_{11}$ ablates more readily.

Wettability

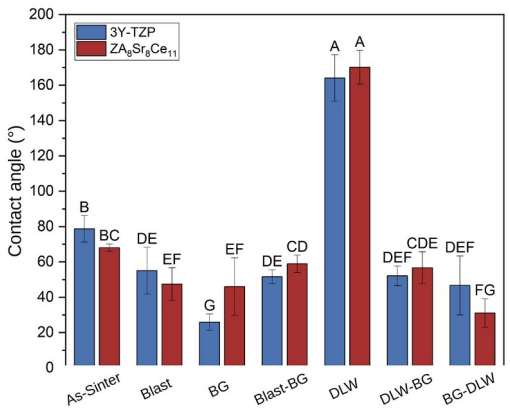


Fig. 3 – Water contact angle (letters: Tukey, p < 0.05).

- DLW alone: **superhydrophobic** (164° / 170°, Cassie–Baxter).
- Any S53P4 step: **hydrophilic** (θ < 60°; silanol groups, ion release).

Crystalline phases (XRD)

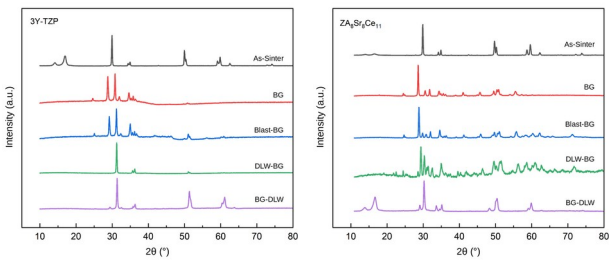


Fig. 4 – XRD: 3Y-TZP (left), $ZA_8Sr_8Ce_{11}$ (right).

- **Tetragonal phase preserved:** no t → m transformation after blasting or ps-DLW.
- Infiltrated groups: sodium calcium silicates (e.g. $Na_2CaSi_2O_6$) and an amorphous halo at 20–50°.

Cell viability (L929, MTS)

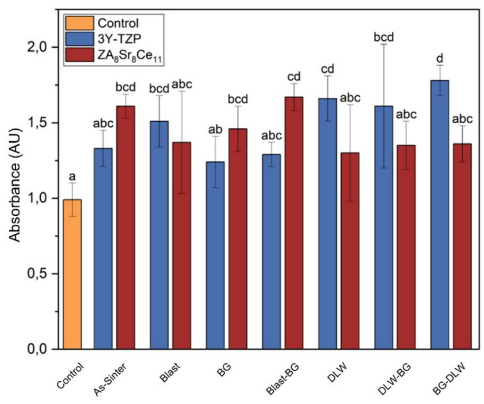


Fig. 5 – Metabolic activity after 24 h (p < 0.05).

- **All 14 surfaces non-cytotoxic** (absorbance ≥ control).
- 3Y-TZP: laser-treated groups highest, **BG-DLW ≈ 1.78**.
- Viability is not governed by wettability alone.

Conclusions

- ps-DLW produced reproducible ~15 µm-wide grooves (up to 17.4 µm deep) without destabilizing the tetragonal phase of 3Y-TZP.
- S53P4 infiltration turned superhydrophobic laser-textured surfaces (θ > 160°) into hydrophilic, silicate-rich surfaces (θ < 60°); the **treatment sequence** controls groove depth and glass distribution.
- All combinations were non-cytotoxic; laser-grooved, glass-infiltrated 3Y-TZP gave the highest fibroblast viability – a versatile route for tuning the tissue–implant interface of zirconia prostheses.

References and Acknowledgements

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