



Apatite layer formation on PLD bioactive glass thin films

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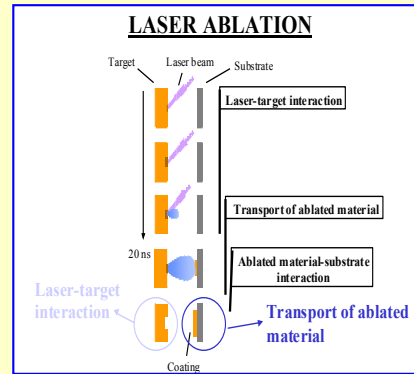
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1.- Production of bioactive glass thin films by Pulsed Laser Ablation

Experimental Conditions:

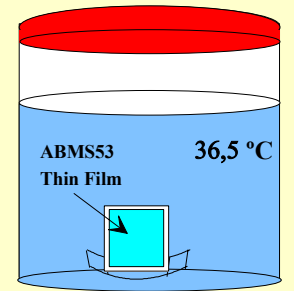
- * **Target composition:** 53% SiO₂, 20% CaO, 23% Na₂O, 4% P₂O₅
- * **Film thickness:** 5 μm
- * **ArF excimer laser:** 193 nm, 175 mJ, 10 Hz.
- * **Substrate temperature:** 25 °C



2.- In vitro study of the different stages of the bioactivity for bioactive glass thin films after immersion in simulated body fluid (SBF).

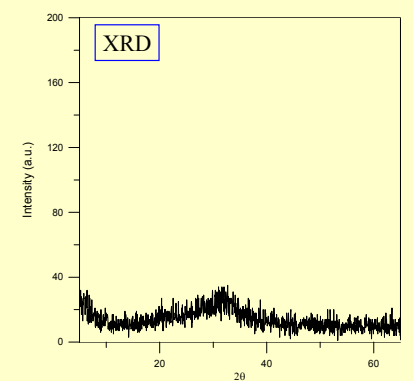
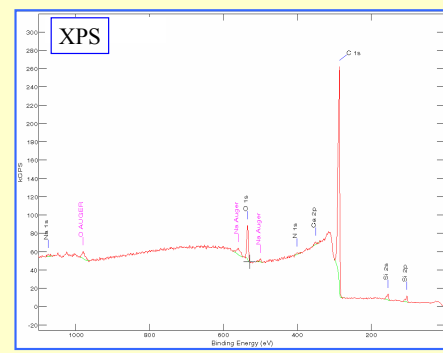
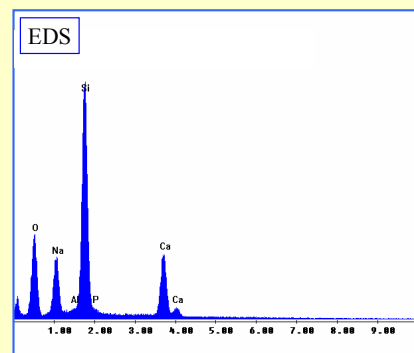
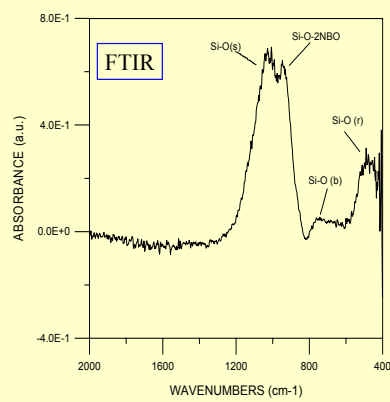
Experimental conditions:

- * 50 ml SBF
- * Sterilised polystyrene bottle
- * Incubator at 36,5°C
- * Immersion times: 12, 24 and 62 hours



RESULTS

ORIGINAL BIOACTIVE GLASS THIN FILM: Vitreous silica network with alkali ions (Ca²⁺, Na⁺)



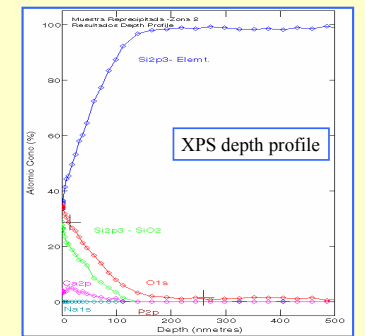
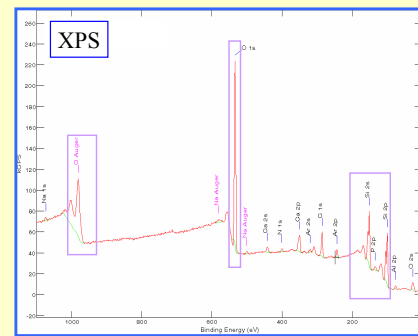
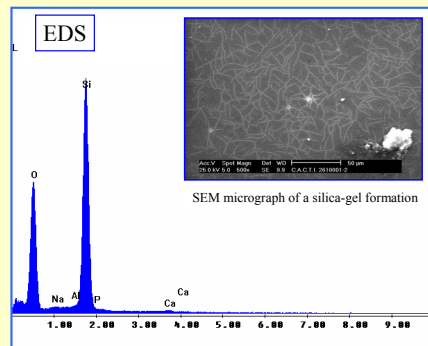
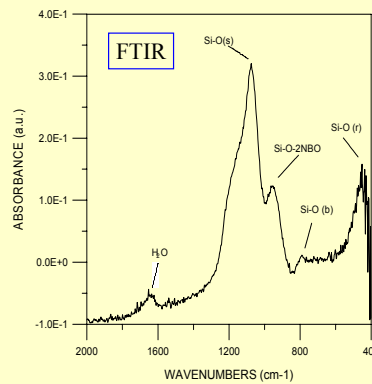
Main IR bands assigned to a silica glass material:

- * Si-O-Si stretching (s), bending (b) and rocking (r) vibration
- * Si-O-2NBO (non-bridging oxygen bond) originated by the addition of the alkali ions

EDS and XPS measurements show the presence of Ca, Na and P ions in a silica (Si, O) network.

The original bioactive glass films are amorphous

AFTER 12 HOURS SOAKING IN SBF: Partial dissolution of the glass surface occurs, leading to the formation of a silica-rich gel layer

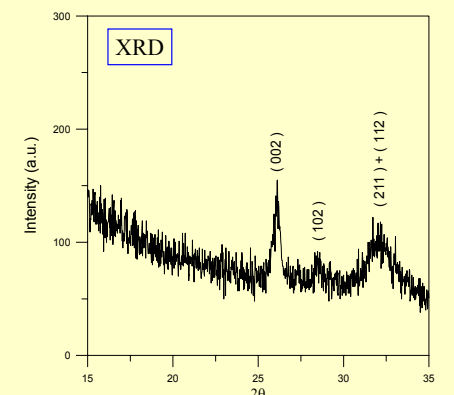
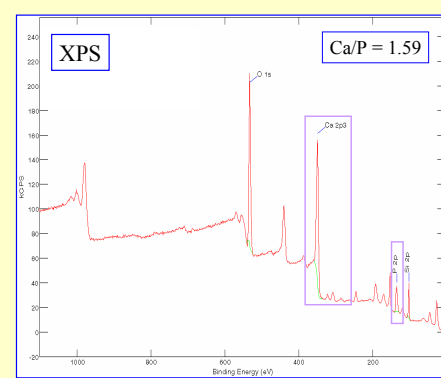
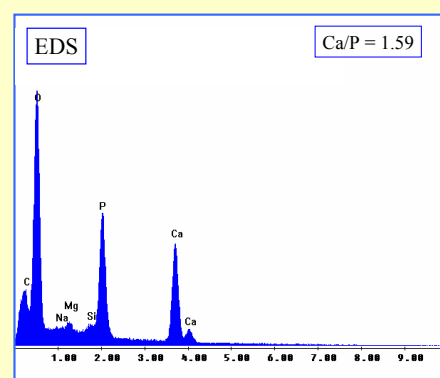
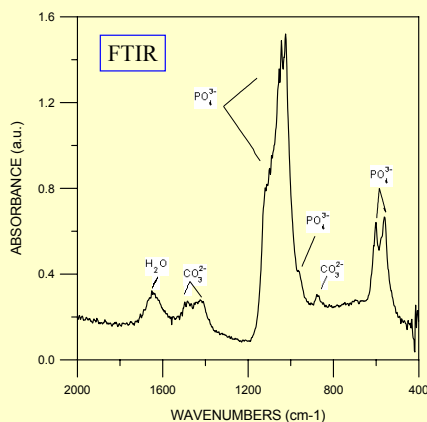


Shift of the Si-O-Si band towards values associated to stoichiometric silica, and diminution of the intensity corresponding to the Si-O-2NBO.

EDS and XPS analysis confirm the reduction of the Ca and Na concentration and an increase of the Si and O concentration.

XPS depth profiling shows the formation of a silica-rich gel layer.

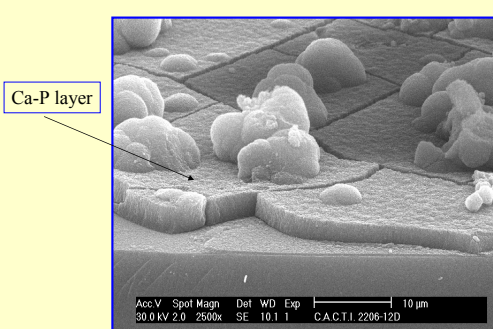
AFTER 62 HOURS SOAKING IN SBF: Precipitation of a calcium phosphate layer



Typical IR absorption bands of apatite film assigned to PO₄³⁻ and CO₃²⁻ bond vibrations. H₂O and OH groups are incorporated.

EDS and XPS measurements and the quantitative analysis confirm the apatite formation with a relation Ca/P=1.59.

XRD spectrum presents the typical diffraction peaks (002, 102, 211, 112) for apatite material.



SEM micrograph of a calcium phosphate layer after soaking in SBF for 62 hours

CONCLUSIONS

- * Bioactive glass thin films have been deposited by pulsed laser ablation.
- * It has been demonstrated that the PLD thin films present, when immersed in SBF, a similar bioactivity behaviour than the bulk glass material.
- * The different stages of the release precipitation process have been identified by FTIR, XRD, EDS and XPS analysis.
- * After soaking in SBF for 62 hours a calcium phosphate layer is formed with a Ca/P relation close to the apatite value.