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Investigation of the microwave sintering of carbonated hydroxyapatite

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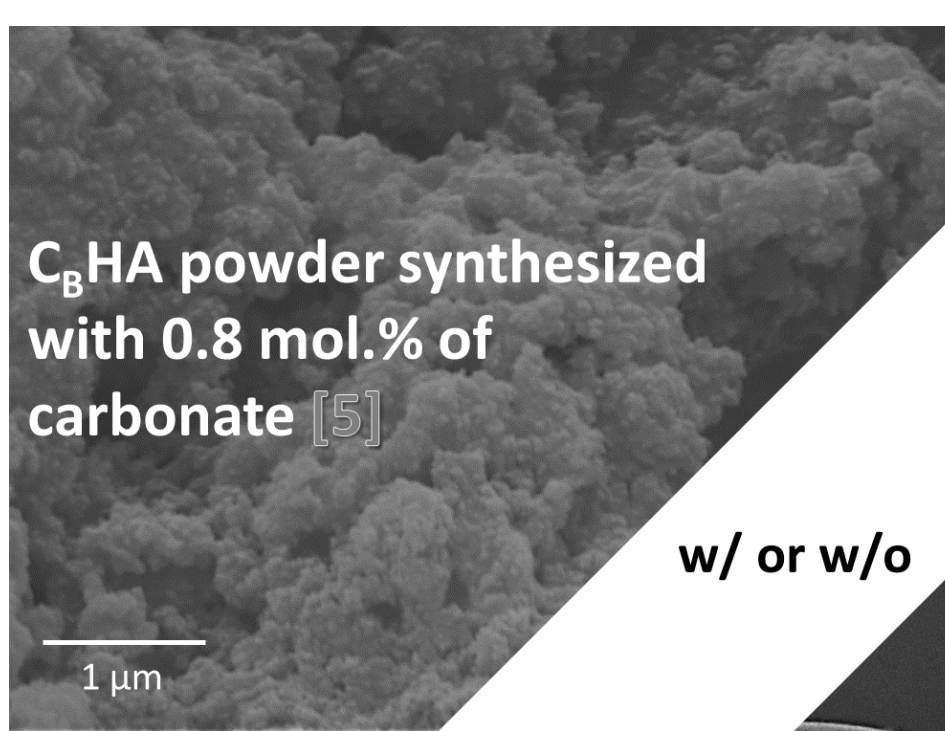


>>> Context

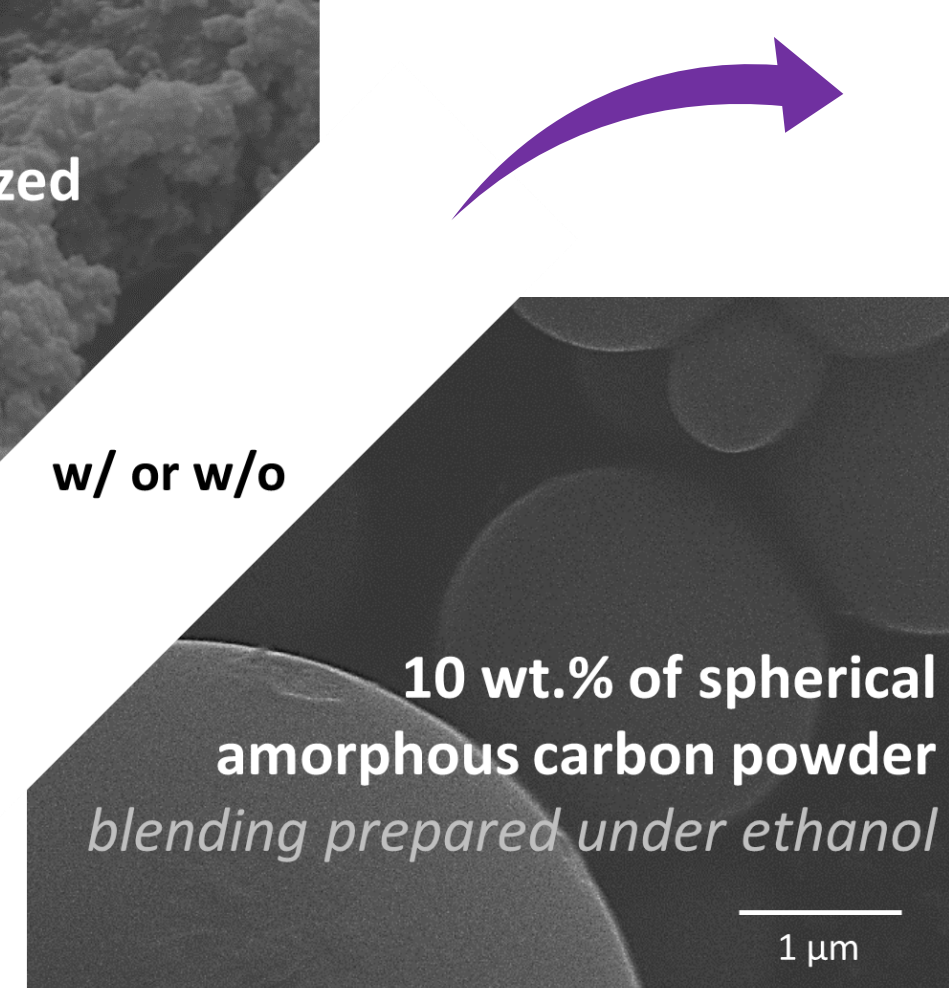
- Carbonated hydroxyapatite (CHA) are biodegradable ceramics suitable for the treatment of bone defect [1]. With a biodegradation rate related to their carbonate ion content [2], it is possible to consider patient-specific implants. Carbonate ions can be located at both the hydroxyl (A-site) and phosphate (B-site) structural sites of hydroxyapatite (HA, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$). B-site CHAs ($\text{C}_\text{B}\text{HA}$) powders are obtained through aqueous precipitation while A-site CHAs ($\text{C}_\text{A}\text{HA}$) result from thermally activated reaction between the apatite and a CO_2 -rich atmosphere [3].
- The resistive sintering of CHA required CO_2 to prevent thermal decomposition, leading to an enrichment of CO_3^{2-} ions in the A-site. Since microwave (MW) sintering is a volumetric heating related to the dielectric properties of the heated material, it allows faster processing times than resistive one [4]. As such, new chemistries as well as finer and thus biologically more reactive microstructures could be expected thanks to MW sintering.
- This study aims at investigating the MW sintering under air of compact $\text{C}_\text{B}\text{HA}$ powders mixed or not with carbon powder acting as a precursor of CO_2 .

>>> Materiel & Methods

1 Powder preparation



2 Uniaxial & isostatic compaction

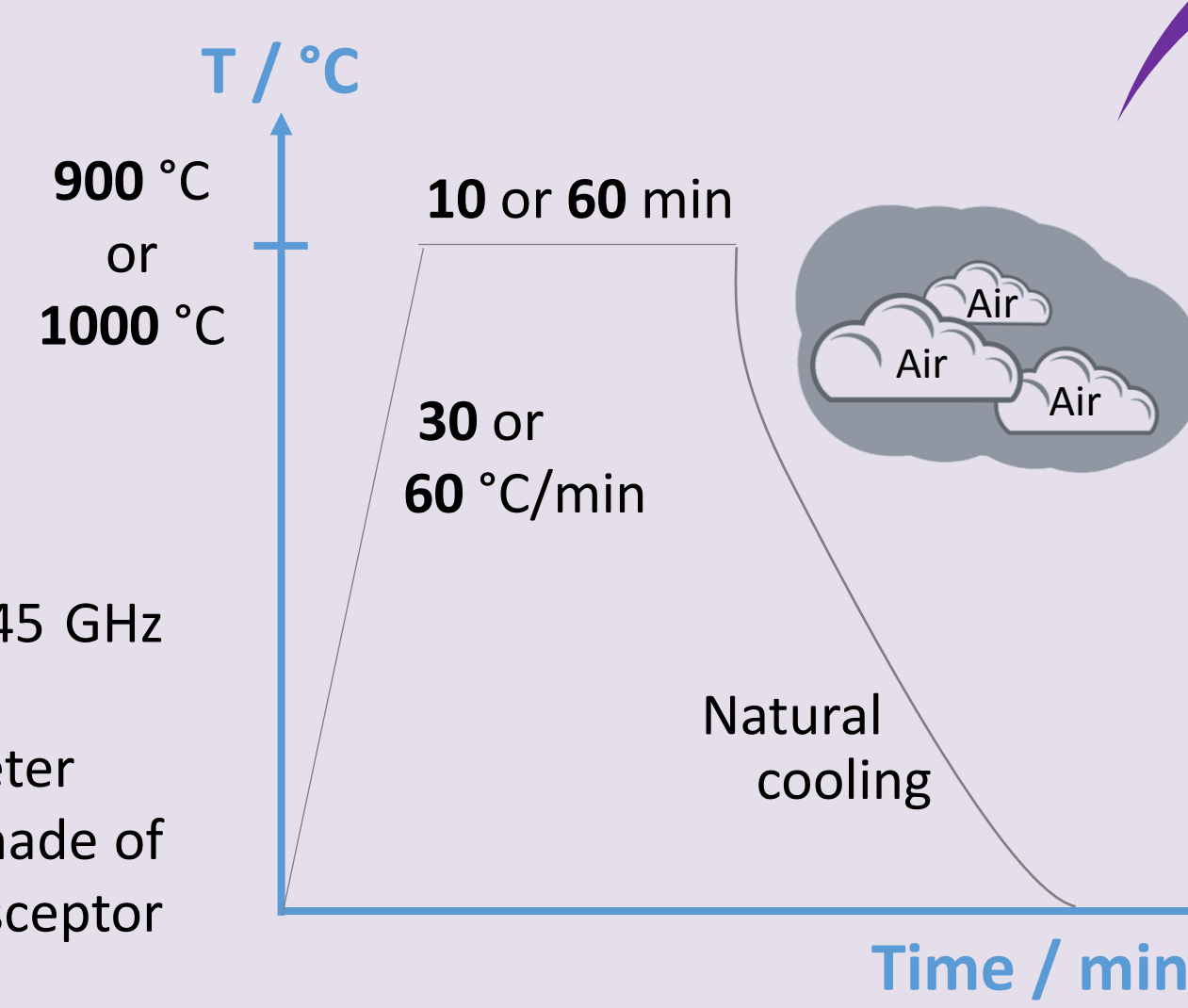


3 MW sintering [6]

Various thermal treatments performed in a multimode cavity under air



- Multimode cavity equipped with a 2,45 GHz MW generator,
- Temperature measurement by pyrometer
- Sample placed inside a sintering cell made of refractory material and a SiC susceptor (hybrid heating)

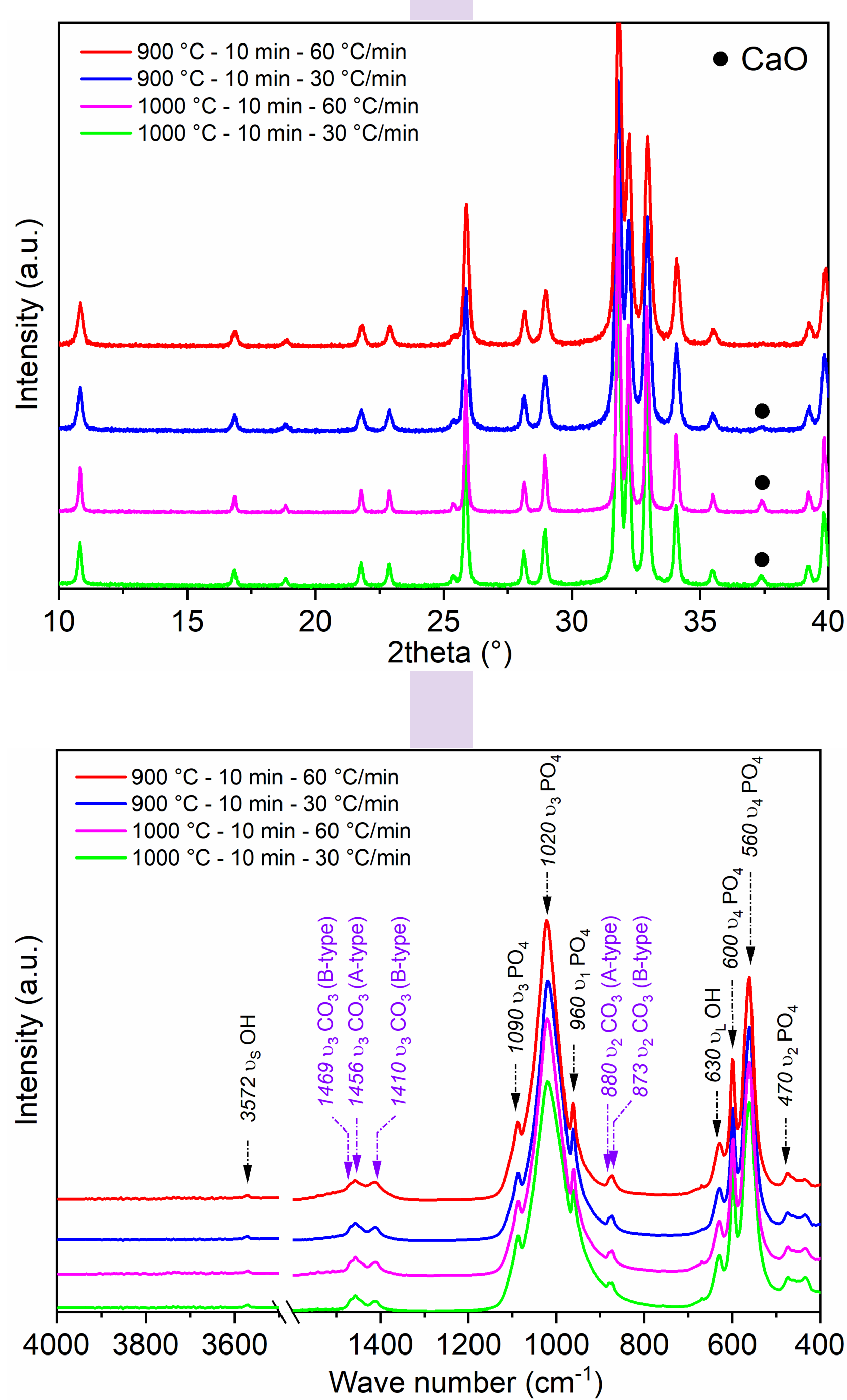


4 Physico-chemical & morphological characterization

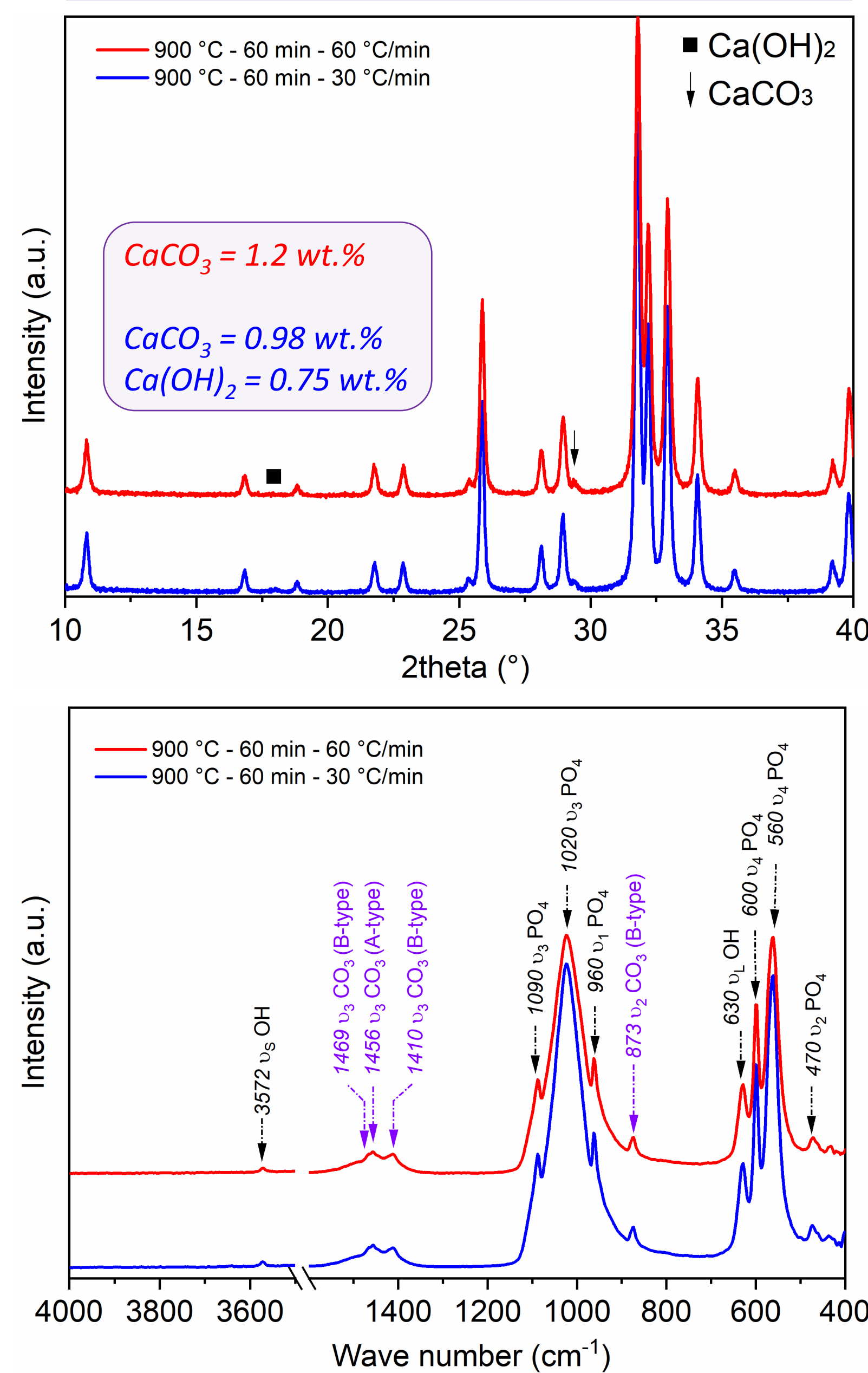
- X-ray diffraction for phase analysis
D8-A25 θ/θ X-ray diffractometer, Bruker, Germany
- Fourier Transformed Infrared spectroscopy
MIR VERTEX 70 Spectrometer, Bruker, Germany
- Scanning Electron Microscopy
JSM 6500F, Jeol, Japan
- Density measurement with the Archimedes method
Kit YDK03, Sartorius, Germany
Theoretical density (TD) set at $3 \text{ g}\cdot\text{cm}^{-3}$ [5]

>>> Results

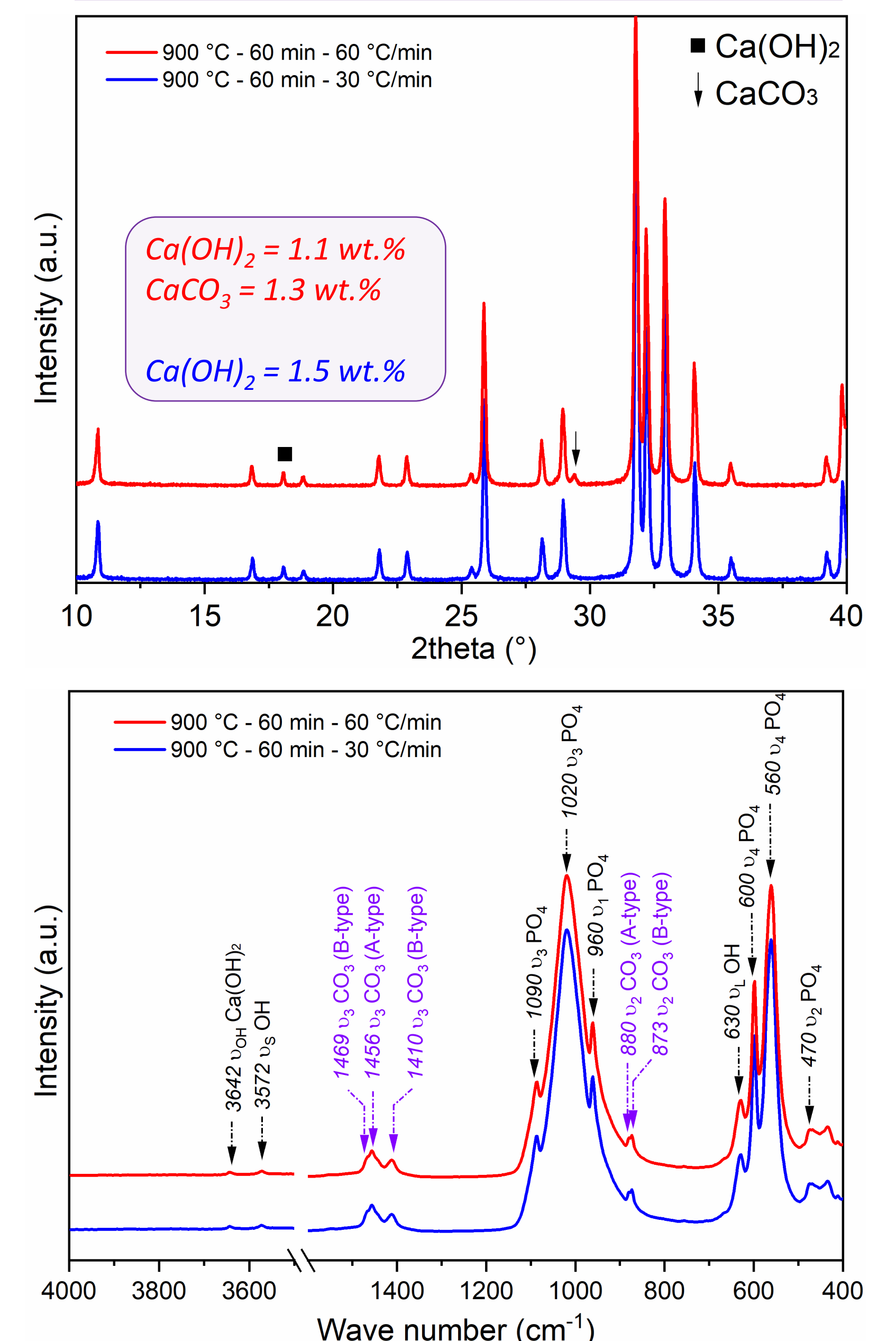
Influence of the temperature



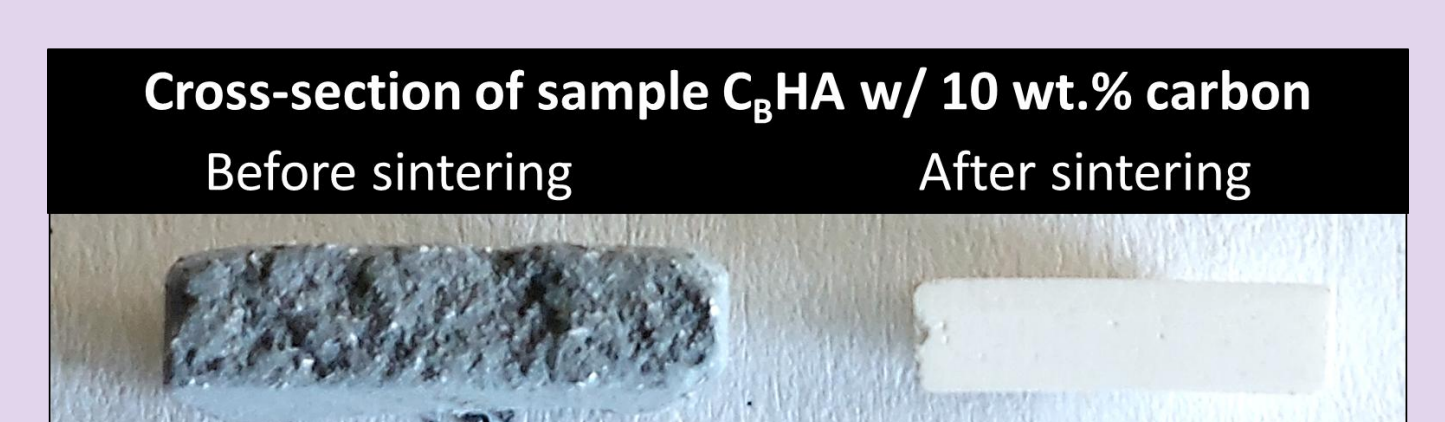
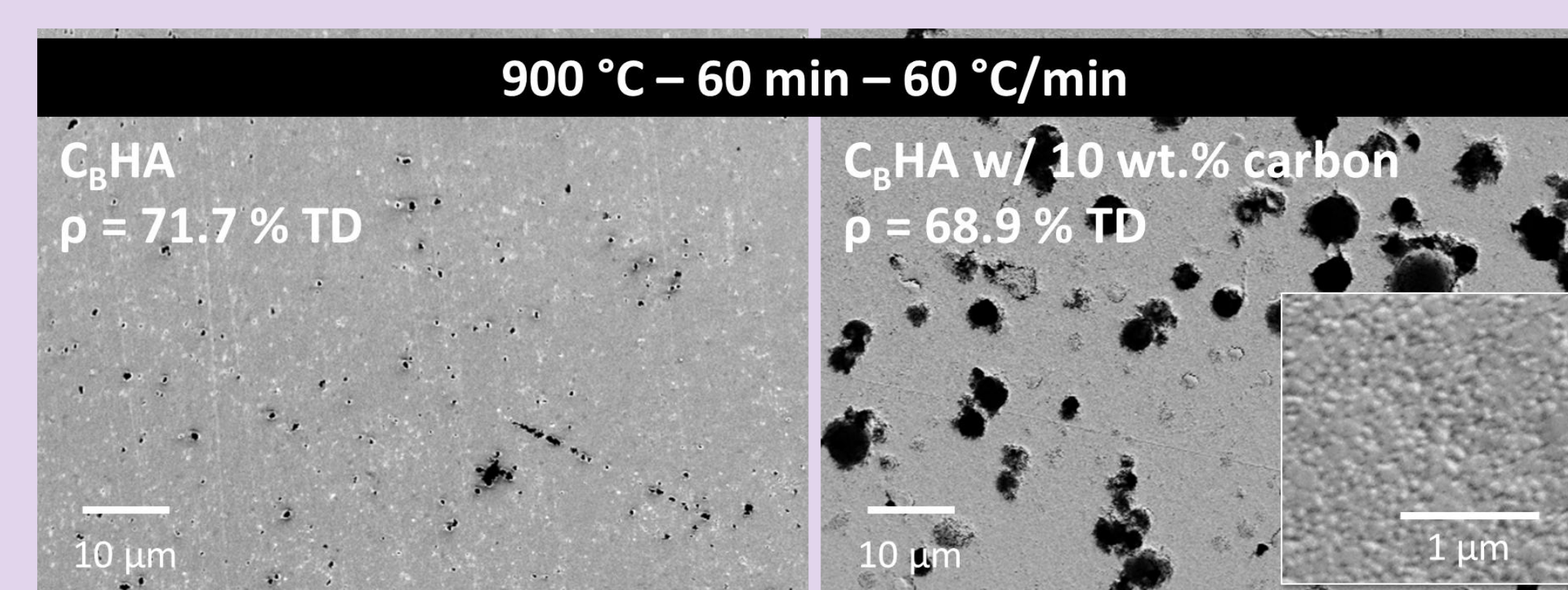
Influence of the dwell time



Influence of adding carbon



- Only the shortest sintering cycle (i.e., 900 °C, 10 min, 60 °C/min) leads to an apparent monophasic ceramics with a final density of 71.3% TD.
- Small amounts of A-type carbonate appear in all samples; probably due to the CO_2 generated during the decomposition of $\text{C}_\text{B}\text{HA}$.



- The carbon powder is completely decomposed during sintering and acts as a porogen that generate a new level of porosity

- At 900 °C, increasing the dwell time leads $\text{C}_\text{B}\text{HA}$ decomposition.
- The presence of higher amount of A-type carbonate in $\text{C}_\text{B}\text{HA}$ sintered w/ 10 wt.% carbon reveals an effective *in situ* generation of CO_2 .
- However, this CO_2 atmosphere is insufficient to avoid $\text{C}_\text{B}\text{HA}$ decomposition at 900 °C.

>>> Conclusions & Prospects

- It is not possible to sinter a pure $\text{C}_\text{B}\text{HA}$ without controlling the MW cavity atmosphere.
- The addition of carbon generates a CO_2 rich atmosphere responsible for the substitution of A-type carbonate but insufficient to avoid the decomposition of $\text{C}_\text{B}\text{HA}$.
- Further studies are needed to optimize the sintering conditions (e.g. thermal cycles, control of the atmosphere inside the cavity) and the preparation of the powder mixtures.

>>> Références

- [1] Paré et al., ACS Biomater Sci Eng, 2020
- [2] Barralet et al., J Biomed Mater Res, 2000
- [3] Lafon et al., J Eur Ceram Soc, 2008
- [4] Oghbaei et al. J. All.