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[1] Zhi Fu, Paula M. Vilarinho, Aiyong Wu, Angus I. Kingon, *Advanced Functional Materials*, 19, 1–11, 2009. (<http://dx.doi.org/10.1002/adfm.200801000>). [2] Luis Amaral, Christine Jamin, Ana Senos, Paula M. Vilarinho, Olivier Guillon, *J. Amer. Ceram. Soc.* 12, 3781-3787, 2012. (DOI: 10.1111/j.1551-2916.2012.05466.x). [3] Luis Amaral, Christine Jamin, Ana Senos, Paula M. Vilarinho, Olivier Guillon, *J. Europ. Ceram. Soc.*, 33, 1801-08, 2013. <http://dx.doi.org/10.1016/j.jeurceramsoc.2013.01.031>

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## Abnormal grain growth in pressure assisted sintering of BaLa<sub>4</sub>Ti<sub>4</sub>O<sub>15</sub>

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The application of compressive stresses during sintering is known to be an efficient way to eliminate porosity at lower temperatures by the direct positive effect on the sintering driving force. Therefore, it is the basis of some special sintering techniques, such as: Hot pressing (HP), Hot isostatic pressing (HIP), Hot forging (HF) and, together with the electric field, in the Electric field assisted sintering (EFAS). Despite the fact that the role of applied stress on the densification is well established, its effect on the movement of grain boundaries (GBs) for grain growth in 2D/3D ceramics has not been sufficiently addressed in sintering models. We have been investigating this topic in functional titanate materials, namely in BaLa<sub>4</sub>Ti<sub>4</sub>O<sub>15</sub> (BLT), a key material in microwave applications due to its high permittivity, high quality factor Q (low dielectric losses) and low temperature coefficient of resonant frequency. The microstructure developed during sintering, in special the anisotropic grain growth, was found to alter the electrical properties of ceramic samples and films [1]. We have reported for the first time that the GB mobility was shown to be dependent on the external stresses created or applied during sintering and that stress assisted sintered samples present enhanced anisotropic grain growth [2,3].

In this work, pressure assisted sintering was applied to BLT and particular attention was given to the microstructure with statistical analysis of the grain size distributions to clarify the role of stresses on the grain growth phenomena. HIP with 65 MPa, HP with 60 MPa and conventional sintering, without any external pressure (CS), for comparison, were used to design microstructures with different grain size distributions, keeping the same thermal history for all the samples. Based on cross section and top view Scanning Electron Microscopy (SEM) micrographs, the grain size was evaluated by measuring the area of the elongated grains. Samples were thinned down to 20 μm using precision ion polishing system for High Resolution Transmission Electron Microscopy (HRTEM) studies.

Via the stereological analysis of the grain size distribution, which was separated into two populations, i.e., normal (NGs) and abnormal grains (AGs), we verified that the main effect of external pressure lies on triggering AG growth, increasing both the number and size of AGs and leading to a more anisotropic grain growth (more elongated and oriented grains), whereas not a significant change was found for the grain growth of the NGs population. Complementarily, the HRTEM analysis showed that the structure of grains and GBs is quite different in stress assisted samples, presenting a larger density of crystal imperfections and thicker GB disorder regions than in CS samples.

### References:

- [1] Zhi Fu, Paula M. Vilarinho, Aiyong Wu, Angus I. Kingon, *Advanced Functional Materials*, 19, 1–11, 2009. (<http://dx.doi.org/10.1002/adfm.200801000>).
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- [3] Luis Amaral, Christine Jamin, Ana Senos, Paula M. Vilarinho, Olivier Guillon, *J. Europ. Ceram. Soc.*, 33, 1801-08, 2013. <http://dx.doi.org/10.1016/j.jeurceramsoc.2013.01.031>