UNDERSTANDING FLASH SINTERING OF SEMICONDUCTOR OXIDE MATERIALS AT THE NANO-ATOMIC SCALE

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As the demand for energy-efficient technologies increases, it is critical to develop processes that produce materials at a lower cost. In this context, flash sintering (FS) is a viable alternative to conventional sintering, leading to a reduction of the processing times from hours to seconds, and a decrease in operating temperatures. Recently, room temperature FS was reported in the literature for materials such as ZnO or YSZ, however, the relationship between the micro/nanoscale properties and the physical mechanisms operating during FS is not fully understood [1], [2]. Also, the investigation of these mechanisms on semiconductor oxides is scarce. This is important as this class of materials has a wide range of applications due to their properties such as superconductivity, ferroelectricity, and dielectric assets [3]. Previous work demonstrates that the electrical response of piezoelectric ceramics is related to the defect formation and kinetics during the flash event [4], [5].

In this work, ex-situ FS studies of dense $La_{0.75}Sr_{0.25}CrO_3$ bulk samples were performed at room temperatures. FS processing parameters were correlated to the nano/atomic structure, such as average grain size, grain size distribution, and composition distribution, obtained through TEM/STEM coupled with EDS. An investigation of the electrochemical reduction of species after FS, likely due to thermal runaway and defect migration, was conducted with aberration-corrected TEM/STEM coupled with Mono EELS. In this way, both the valence state of chromium and the distribution of oxygen were mapped before and after FS. Additionally, the grain boundaries were characterized by aberration corrected TEM/STEM to identify the presence of amorphous phases resulting from local melting, which is also associated with the thermal runaway mechanism. Finally, an experimental setup for in-situ TEM flash sintering analysis was developed, where changes in the structure of electron transparent samples could be detected and monitored during the application of an electric field to simulate FS at room temperature, (Figure 1).

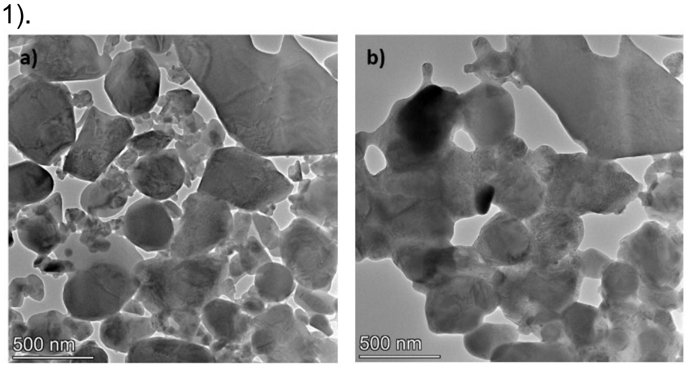

Figure 1 - TEM images of $La_{0.75}Sr_{0.25}CrO_3$ a) before and b) after flash sintering.

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[1] N. Yan et al., “Ethanol-Induced Flash Sintering of ZnO Ceramics at Room Temperature,” Materials (Basel), vol. 15, no. 3, pp. 1–9, 2022. [2] Y. Zhu et al., “Gas-discharge induced flash sintering of YSZ ceramics at room temperature,” J. Adv. Ceram., vol. 11, no. 4, pp. 603–614, 2022. [3] R. Catlow and A. Walsh, “Semiconducting oxides,” J. Phys. Condens. Matter, vol. 23, no. 33, pp. 2010–2011, 2011. [4] R. Serrazina, A. Tkach, L. Pereira, A. M. O. R. Senos, and P. M. Vilarinho, “Flash Sintered Potassium Sodium Niobate: High-Performance Piezoelectric Ceramics at Low Thermal Budget Processing,” Materials (Basel), vol. 15, no. 19, 2022. [5] R. Serrazina, J. S. Dean, I. M. Reaney, L. Pereira, P. M. Vilarinho, and A. M. O. R. Senos, “Mechanism of densification in low-temperature FLASH sintered lead free potassium sodium niobate (KNN) piezoelectrics,” J. Mater. Chem. C, vol. 7, no. 45, pp. 14334–14341, 2019.