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Fast one-step synthesis and sintering of materials promoted by electric fields

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[1] Subramanian, M. A. et al., High dielectric constant in ACu3Ti4O12 and ACu3Ti3FeO12 phases. J. Solid State Chem. 151, 323-325 (2000). [2] Cologna, M., Rashkova, B., RAJ, R., Flash sintering of nanograin zirconia in < 5 s at 850 °C. J. Am. Ceram. Soc. 93, 3556-3559 (2010). [3] Jesus, L. M. et al., Polymeric synthesis and conventional versus laser sintering of CaCu3Ti4O12 electroceramics: (micro)structures, phase development and dielectric properties. J. Alloys Compd., In press, DOI: 10.1016/j.jallcom.2015.09.027 (2015).

ELECTRIC FIELD-ASSISTED FLASH SINTERING OF FINE-GRAINED AND HIGH-PERMITTIVITY $CaCu_3Ti_4O_{12}$ ELECTROCERAMICS

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$CaCu_3Ti_4O_{12}$ (CCTO) has attracted great attention because of its potential application in microelectronic devices, as showing very high ε' values (~ 12,000) with good stability from room temperature to 300 °C [1]. This material is usually prepared through conventional synthesis (solid-state reaction) at 1000 °C followed by sintering at 1100 °C, for dwell times of several hours. These high annealing temperatures and times lead to ceramics with micro-sized grains, exceeding significantly 1.0 μm in most cases. Field-assisted flash sintering [2] was here considered for producing high-quality CCTO electroceramics, from a powder originally synthesized via a modified polymeric precursor method [3] and calcined at 800 ˚C for 2 h. The study includes analyzing the dynamics of material shrinkage and densification. With increasing electric field (E), three distinct regimes were distinguished (see Figure 1): a conventional-like sintering behavior for $E <$ 15 V/cm, followed by a region of accelerated (fast-dominated) sintering for $15 \lesssim E < 30$ V/cm, and then the flash-dominated regime, for $E \gtrsim 30$ V/cm, where sintering is not only accelerated but occurs suddenly. In consequence, under field action, sintering of the material was achieved at furnace temperatures sensibly lower, reaching a value as low as 750 ˚C for E = 60 V/cm versus 1050 ˚C in conventional processing. The physical mechanism behind each regime and the extent to which the rise in sample temperature induced by the applied field (Joule heating) is determinant are also discussed. Finally, a correlation was found between the microstructural characteristics achieved during sintering and the dielectric response measured.

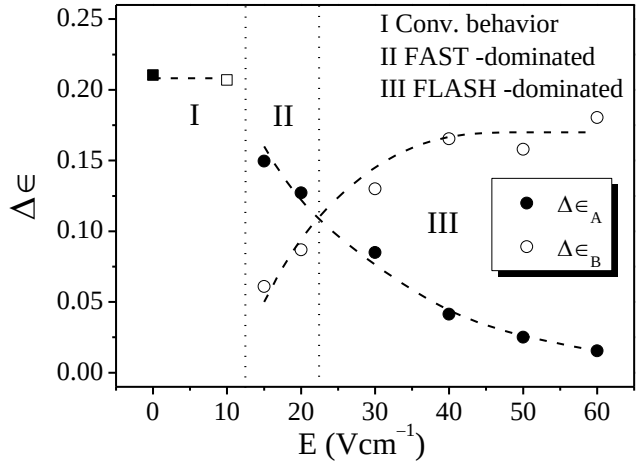

Figure 1 – Partitioning of shrinkage strain attributed to conventional-like behavior (I), FAST- (II) and FLASH-dominated regions (III).

References

[1] Subramanian, M. A. *et al*., High dielectric constant in $ACu_3Ti_4O_{12}$ and $ACu_3Ti_3FeO_{12}$ phases. *J. Solid State Chem.* **151,** 323-325 (2000).

[2] Cologna, M., Rashkova, B., RAJ, R., Flash sintering of nanograin zirconia in < 5 s at 850 °C. *J. Am. Ceram. Soc.* **93,** 3556-3559 (2010).

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