

Synthesis Procedure for the Preparation of $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$

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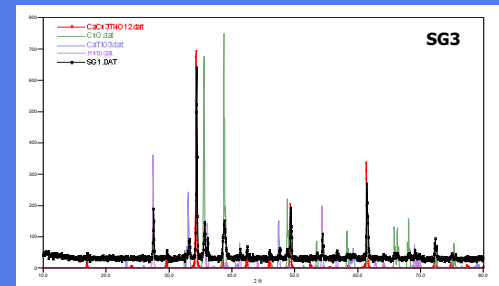
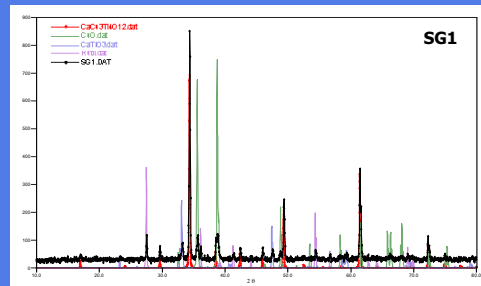
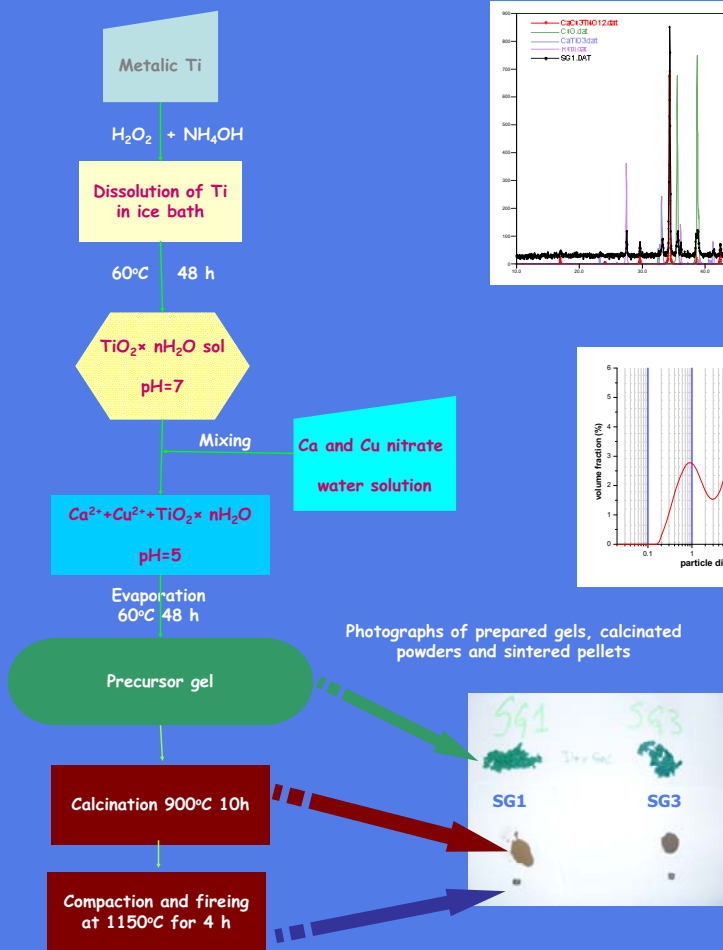
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Introduction

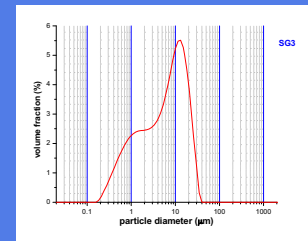
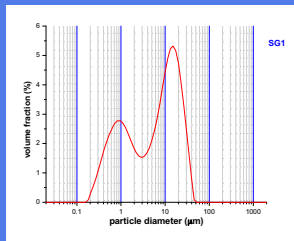
Type of bcc perovskite compounds with general formula $\text{AC}_3\text{B}_4\text{O}_{12}$ have been reported to have unusually high dielectric constant. In compound A can be Ca, Cd, Sr, Na or Th, B is Ti or $(\text{Ti}+\text{M}^{5+})$ in which M is Ta, Sb or Nb and C is Cu^{2+} or Mn^{3+} . Because its permittivity of 10^4 that is independent of temperature and frequency (from -170 to 100 °C up to 10 MHz) compound $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$ (CCT) has been recognized as promising material for capacitors in microelectronic devices and power electronics. Material has been synthesized mostly by solid state ceramic method, only a few soft chemistry methods have been used for preparation of this compound such as organic gel assisted citrate or oxalate process. In this work precursor is prepared by sol-gel method without addition of organic gelation agent, subsequent calcination of gel yielded CCT.

Experimental

Synthesis procedure is shown schematically on chart. After evaporation of water remained green gel. Calcination lead to formation of CCT, CuO, CaTiO_3 and TiO_2 which is shown on XRPD patterns for samples SG1 and SG3. Particle size distributions for these two powder samples are determined as well. Electrical measurements and SEM analysis were performed on sintered ceramics.

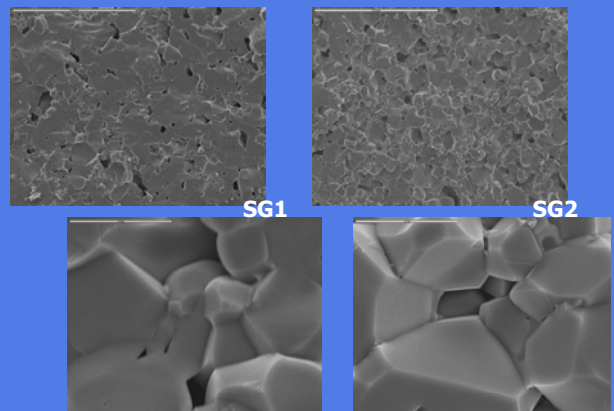


Size distributions of calcined powders



SEM

After electrical measurements, microstructure of CCT sintered pellets was characterized by SEM. The characterization was done on fractured surfaces.



Electrical Measurements

- CCT ceramics have been electrically studied. Samples for the electrical measurement were prepared from calcinated powders; powders were uniaxially pressed and sintered in air atmosphere on 1150 °C for 4 h.

- The electrical measurements were conducted on sintered pellets electroded with silver paste. The measurements were performed in air at ambient temperature using a Wayne Kerr Universal Bridge B224 (internal frequency 1000 Hz).

- The relative permittivity (ϵ_r) was calculated according to equation: $\epsilon_r = \frac{C \cdot h}{\epsilon_0 \cdot A}$

Sample	Relative permittivity
SG1	30600
SG3	12380

Conclusion

In this work it was shown that CCT based material with large dielectric constant can be successfully prepared by sol-gel method with subsequent calcination step. By controlling amount of Cu ions in gel is possible to tune electrical characteristics in sintered ceramics.