

## Sol-gel synthesis and thermal characterization of the batches of BSCCO system

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**Sol-gel synthesis and thermal characterization of the batches of BSCCO system:** A sol-gel method was used for the preparation of the superconducting Bi-2223 and Bi-2212 phases, using nitrate solutions. Citric acid was used as a precursor. The gels were converted to BSCCO phases by thermal treatment. The synthesized products were characterized by DTA and IR. It has been observed that the formation of the high  $T_c$  phase is remarkably enhanced at the temperature of the exothermic peak of the DTA curve.

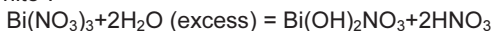
**Key words:** BSCCO, sol-gel method, superconductor.

### INTRODUCTION

Since the first report on new high  $T_c$  superconducting BiSrCaCuO ceramics by Maeda et al. [1], many attempts have been done on the effect of the doping or substitution by different rare earth elements on the electrical properties, magnetic properties and thermal characterizations for the high- $T_c$  superconductors [1–9]. Study of the thermal and structural properties [5–14] of BiSrCaCuO (BSCCO) glass ceramics is important to understand the nucleation and crystal growth mechanism which is essential to obtain high quality glasses for technological applications. Many works have reported the glass thermal properties studied by isothermal and non-isothermal methods [10–14]. The non-isothermal method offers some advantages when compared to isothermal method. One of them is that the non-isothermal experiments can be performed in shorter time period and in a wider temperature range. In addition, most phase transformations occur too rapidly to be measured under isothermal conditions because of the inherent transients associated with the experimental apparatus [15,16]. Glasses are useful model materials in which to study phase development and kinetics in variety of systems can be studied. Various mechanisms of superconducting phase development have been reported in the literature [17–21]. The crystallization kinetics of BSCCO systems have been investigated by using DTA, differential scanning calorimeter (DSC) and TG methods. [11–14,22–24]. The samples were fabricated by sol-gel method. In order to determine the crystallization kinetic of the investigated samples, DTA and TG analysis were employed.

### EXPERIMENTAL

The starting materials are  $\text{Bi}_2\text{O}_3$ ,  $\text{SrNO}_3$ ,  $\text{CaCO}_3$  and  $\text{CuO}$  powders which were used for production of the samples. Analytical grade of reagent powders were dissolved in distilled water to prepare nitrate aqueous solutions.  $\text{Bi}(\text{NO}_3)_3$  does not dissolve in water forming a white precipitate of basic nitrate classically referred to as the “Magistry of bismuth flake/pearl white”.



Therefore the addition of  $\text{HNO}_3$  is required to dissolve bismuth nitrate. For each batch, as sufficient amount of citric acid was added for requisite complexation. pH had to be raised by the addition of ammonia solution. A pH=6 was used to allow the citric acid to form organo-metallic complexes. These solutions then were mixed together by the mole ratio of Bi:Sr:Ca:Cu = 2:2:1:2 and Bi:Sr:Ca:Cu = 2:2:2:3. An appropriate amount of citric acid was added into the mixture to alter the citrate–nitrate ratio. Resultant solutions then were dried at 150 °C on the hot plate and then were placed into heating furnace for calcination at 400 and 850 °C for 2h each.

The investigation of complex and carbonates formation was carried out by IR spectrometer.

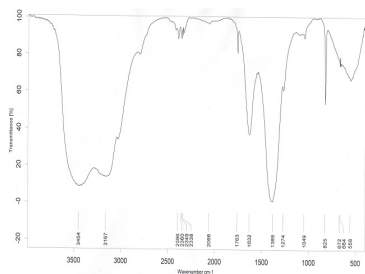


Fig. 1(a). IR spectra of Bi-2212/150 °C

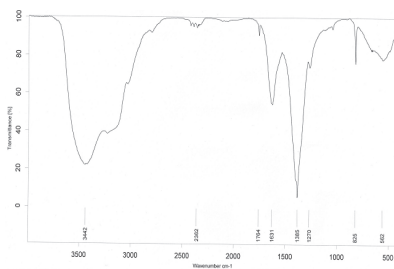


Fig. 1(b). IR spectra of Bi-2223/150 °C

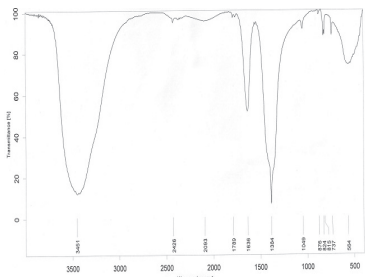


Fig. 1(c). IR spectra of Bi-2212/400 °C

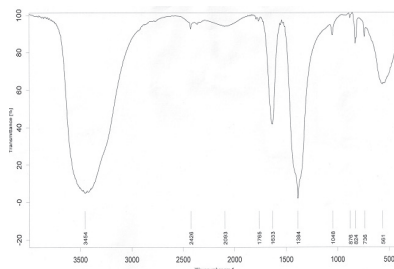


Fig. 1(d). IR spectra of Bi-2223/400 °C

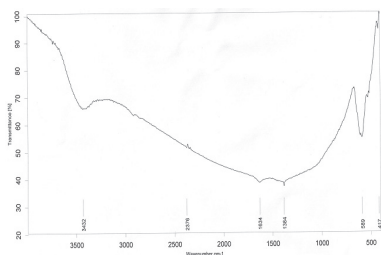


Fig. 1(e). IR spectra of Bi-2212/850 °C

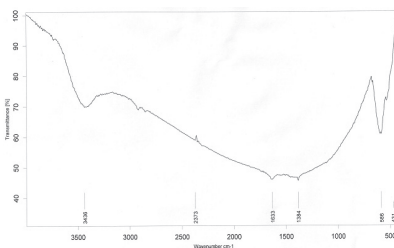


Fig. 1(f). IR spectra of Bi-2223/850 °C

It was observed that the decomposition process of the precursor was sharp and extremely exothermic. Therefore, to avoid vigorous heat evolution, burning and possible segregation, the precursor was heated in to stages at 400 and 850 °C. An IR spectrum of the gel heated first at 150, then 400 and 850 °C are shown in figs 1a-f. In fig 1 a-d the absorption peaks due to carboxylate ions were found at 664, 876 and 1632  $\text{cm}^{-1}$ . The presence of the 1631  $\text{cm}^{-1}$  band confirmed the complex formation in the BSCCO material. Only a few peaks due to  $\text{NO}_3^-$  ions were found at 1385, 825  $\text{cm}^{-1}$  (fig.1 a-d), which indicate that most of the ions were consumed to produce the precipitated  $\text{NH}_4\text{NO}_3$  in the precursor during the gel processing.

Fig. 2 a,b shows thermal analysis of Bi-Sr-Ca-Cu-O – 2212; Bi-Sr-Ca-Cu-O – 2223.

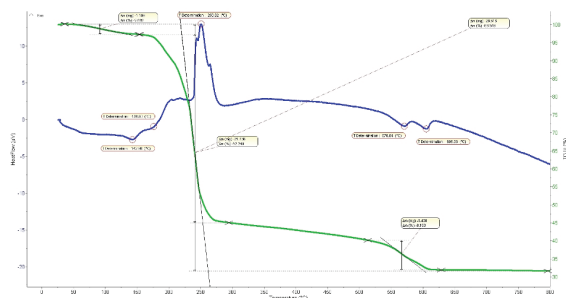


Fig. 2(a). DTA Bi-2212 sample

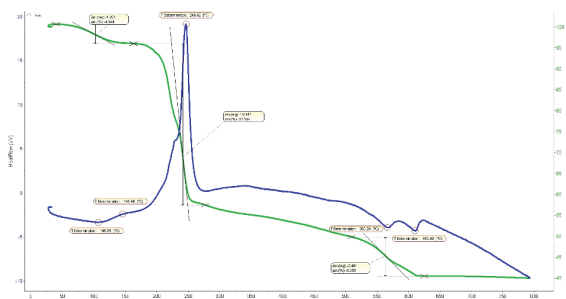


Fig. 2(b). DTA Bi-2223 sample

The thermal analysis indicated exothermic peak at 250.82 °C which shows decomposition of the citrate precursor. TG curve shows mass loss at 275 °C for both systems, Bi-2212 – 52,3%; Bi-2223 – 38,6%. From 275 to 510 °C the system become stable. Then at 510 to 620 °C was observed another mass loss: Bi-2212 – 8,2%; Bi-2223 – 9,4%.

## CONCLUSION

Powders of Bi-2212 and Bi-2223 were synthesized using starting materials – oxides. Sol – gel method is a successful technique to produce excellent ceramic materials. Gels were obtained by complexation of nitrates with citric acid and ammonia. The microstructure of the gels depends on the drying temperature.

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