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MATERIAL CHARACTERIZATION OF SUPERCONDUCTING NANO CRYSTALLINE CERAMIC YSrBiCuO BY THERMAL TECHNIQUES

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ABSTRACT

The nano-sized ceramic superconducting material YSBCO having perovskite structure was prepared according to the chemical formula by the thermo chemical solid state reaction technique using a high-energy ball milling process through mechanically assisted synthesis. It was characterized by XRD, SEM and EDX. Here the authours studied the thermal characterization using TGA, DTA and DSC.

Indexing terms/Keywords

YSrBiCuO; TGA; DSC; EDX.

Academic Discipline And Sub-Disciplines

Faculty of Science, Physics

SUBJECT CLASSIFICATION

Physics; Condensed Matter.

TYPE (METHOD/APPROACH)

Full Research article - Experimental along with theory and analysis

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1.INTRODUCTION

There is a complete expulsion of magnetic flux, that superconductors are perfectly diamagnetic [1] [2]. High-voltage generator can be developed by using HTS inductor and electronic RCL series resonant circuit [3] [4]. The most familiar ceramic superconducting materials have perovskite structure[5]. Ceramics generally can withstand very high temperatures such as temperatures that range from 1000˚C to 1600˚C (1800˚F to 3000˚F). Conventional solid state reaction method is a common and effective way to fabricate modern ceramics [6]. The Yttrium Strontium Bismuth Copper Oxide (YSrBiCuO) is a type of perovskite ceramic superconductor with high dielectric constant. High dielectric constant (High-K) ceramic composites have become potential candidate materials for integration into high frequency electronics. Detailed understanding of this class of materials will help electronic industry in planning, design and processing of these materials.

The authors fabricated YSrBiCuO nano crystalline ceramic type II high-TC superconductor material by the solid state thermo chemical reaction technique and it was characterized to show good quality, homogeneity and the desired stoichiometry of the sample prepared [7]. The results were analyzed by X-Ray Diffraction (XRD), SEM, and EDX.

In this work the authors are presenting the material characterization of Ytttrium strontium bismuth copper oxide (YSrBiCuO / YSBCO) calcined at 950^0C by thermal behavior to evaluate the chemical and physical properties. TGA, DTA and DSC are used to analyze thermal behaviour of nanoparticles [8-10] at a high temperature. Using EDX elemental composition is obtained. The phase transformations in nano materials due to temperature change is much different from that of bulk crystals. Phase transformations in nano structured materials reported [11]. The free energy of nano particles are always higher than that of its conventional counterpart[12].

2.EXPERIMENTAL

2.1. Preparation of the Sample

The reagent grade chemicals of high purity yttrium oxide, strontium carbonate, bismuth oxide, cupric oxide powders were used as the raw materials and weighed according to their molecular formula for the preparation of the sample YSBCO. The sample was ball milled for three weeks with suitable zirconium balls to insure homogeneity and quality. Then it was attrition milled for five hours. Then the material calcined at different temperatures, finally at 950˚C. After the furnace is off, on cooling the oxygen is allowed to flow into the furnace at intervals (oxygen annealing). A temperature much higher than this will result in a material that is much harder to regrind, information from the thermal studies of this material. Control of temperature is often necessary to ensure that the desired crystalline phase is formed with optimum particle size [13]. For thermal studies TGA, DTA and DSC data were analyzed. From EDX, the composition details of the prepared ceramics were determined.

2.2. TGA –Analysis

TGA measures a sample's weight as it is heated or cooled in a furnace. Thermogravimetric Analysis is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature program in a controlled atmosphere. Factors such as sample mass, volume and physical form, the shape and nature of the sample holder, the nature and pressure of the atmosphere in the sample chamber, and the scanning rate have significant influences on the characteristics of the recorded TG curve. Because most events that occur in a TGA are kinetic in nature, any experimental parameter that can affect the reaction rate. The reaction is characterized by two temperatures, Ti and Tf , which are called the procedural decomposition temperature and the final temperature[14]. Figure 1 shows a schematic representation of thermal balance instrument.

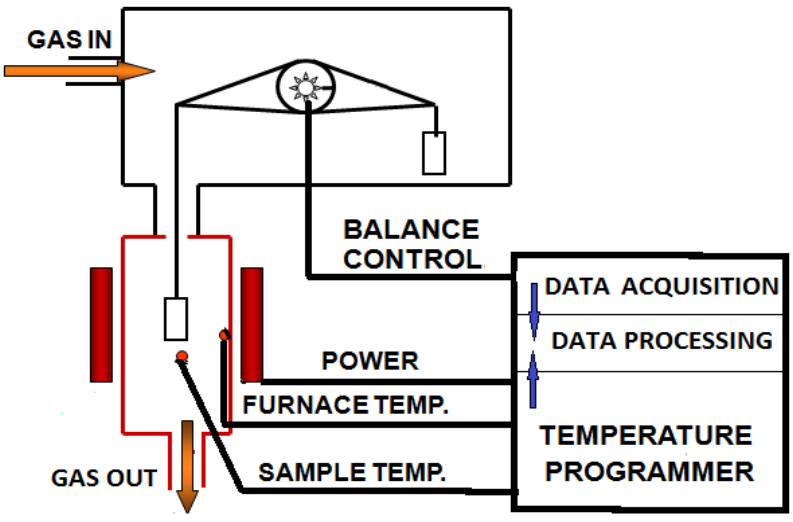

Figure 1. A schematic thermobalance instrumentation

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Thermogravimetric Analysis (TG) determines the weight changes of a sample, whereas Differential Thermal Analysis (DTA) measures changes in temperature between a sample and a reference, as a function of temperature or time.TG and DTA curves of the sample were recorded using Perkin Elmer, Diamond TG/DTA with Flexible axial and radial view instrument, with high concentration capabilities. TGA is plotted in figure 2 (a) & (b). DTA/DTG is plotted in figure 3.

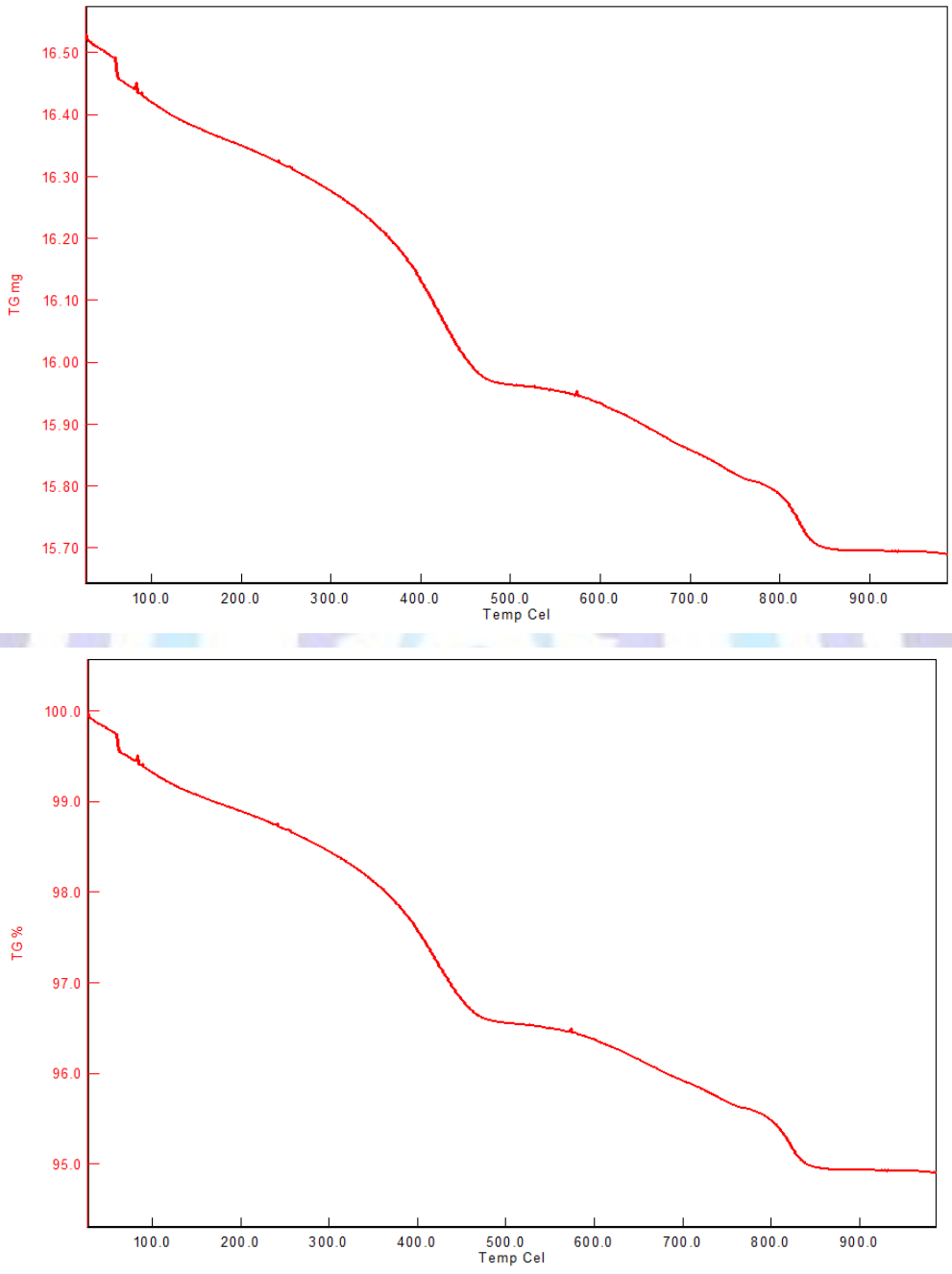

Fig.2 (a) & (b) TGA curve of YSBCO (in weight and in percentage vs. Temp.)

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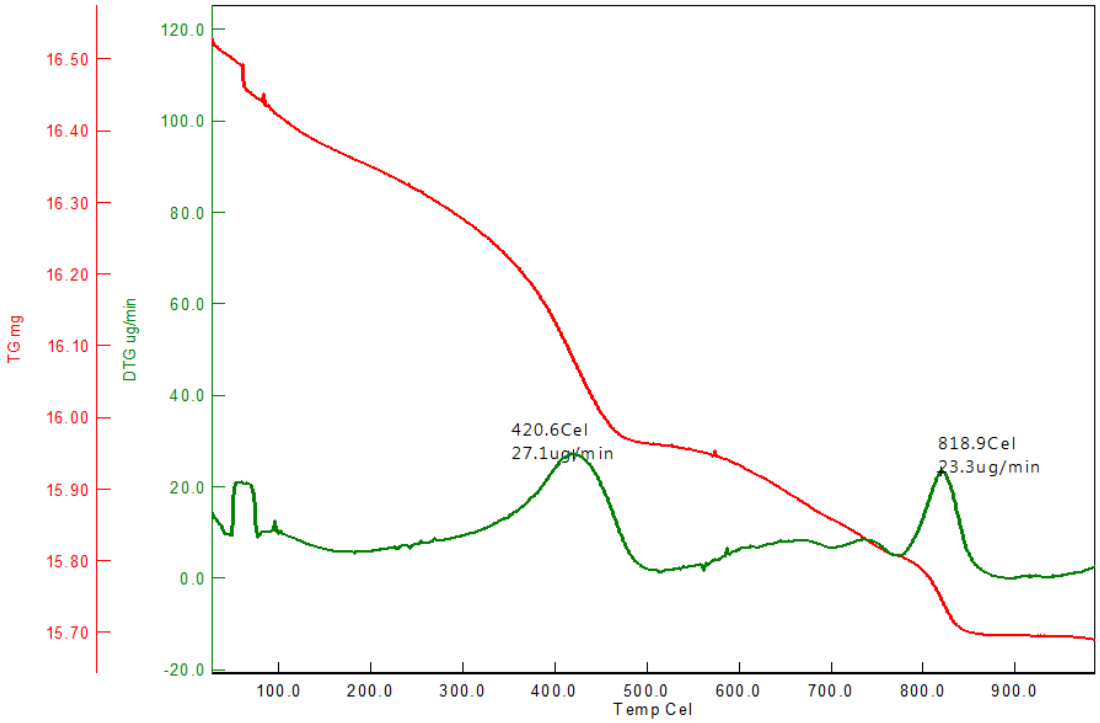

Fig.3. TG/DTA /DTG curve of the sample YSBCO

2.3. DSC Analysis

Differential scanning calorimetry (DSC) is an inexpensive and rapid method to measure heat capacities of condensed phases. From these measuremenmst, enthalpy changes for phase transitions can easily be determined. The applications of DSC are numerous, either for routine quality control measurements or in research, where high sensitivity and flexibility are important aspects. DSC curve was plotted using Mettler Toledo DSC 822e which is shown in figure 4.

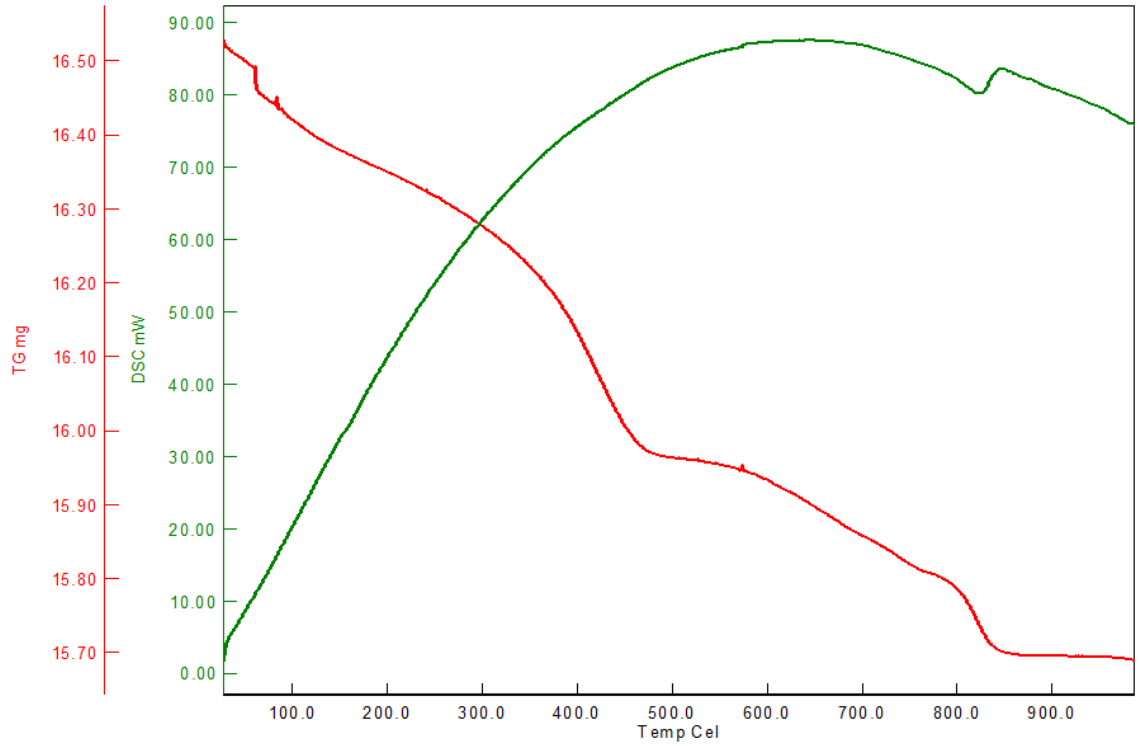

Fig. 4 DSC curve ofYSBCO

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Differential scanning calorimetry (DSC) monitors heat effects associated with phase transitions and chemical reactions as a function of temperature. In a DSC the difference in heat flow to the sample and a reference at the same temperature, is recorded as a function of temperature. The reference is an inert material such as alumina. The temperature of both the sample and reference are increased at a constant rate. Since the DSC is at constant pressure, heat flow is equivalent to enthalpy changes. The heat flow difference between sample and reference can be either positive or negative. In an endothermic process, such as most phase transitions, heat is absorbed and, therefore, heat flow to the sample is higher than that to the reference. Hence dH/dt is positive. In an exothermic process, such as crystallization, dH/dt is negative.

2.4. EDX Analysis

The composition details (EDX) of the prepared nanocrystalline ceramic powder YSBCO at 950^{o}C [7] is plotted in figure 5 using ISIS Link Oxford Instrument UK. In this technique an electron beam of 10-20 KeV strikes the surface of a sample which causes X-ray to be emitted from point of incidence. When an X-ray strikes the detector, it will generate a photoelectron which in turn generates electron-hole pairs. The energy of the X-ray emitted depends on material under examination. The energy of the characteristic X-ray emitted from the different elements is different and thus it gives the unavoidable signature of the particular element. A strong electric field attracts the electrons and holes towards the opposite ends of the detector. The size of the pulse thus generated depends on the number electron-hole pairs created, which in turn depends on the energy of the incoming X-ray. Table 1 indicates the contents of the material.

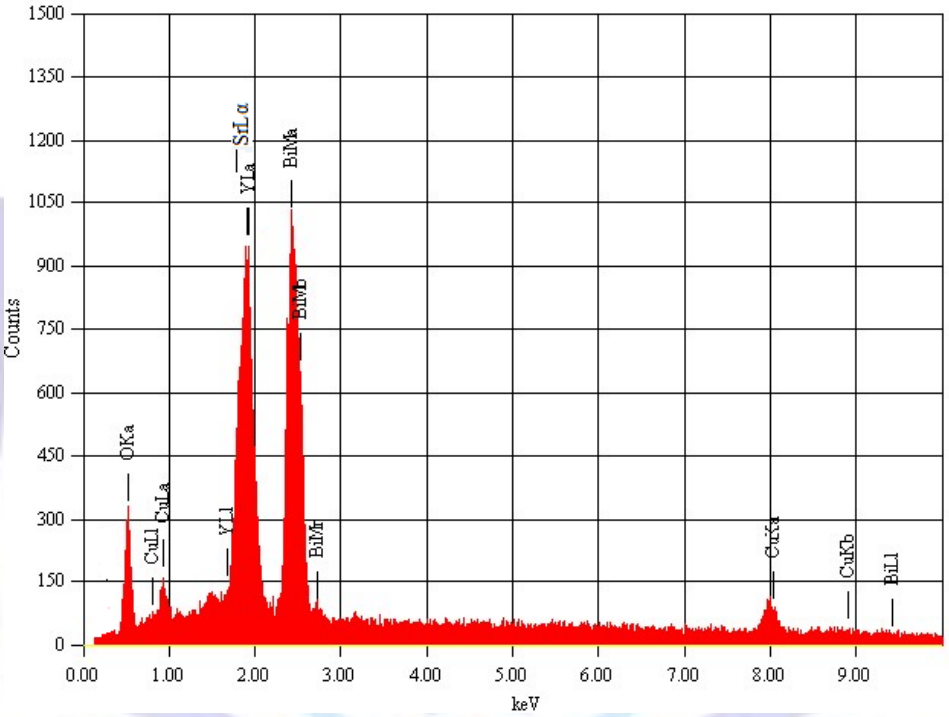

Fig.5 EDAX spectrum of YSBCO

Table 1 shows the Percentage composition of prepared sample YSrBiCuO

Element	(keV)	Mass%
O K	0.525	3.57
Cu K	8.04	3.81
Y L	1.922	15.35
Sr L	1.806	15.75
Bi M	2.419	61.52
Total		100

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3. RESULTS AND DISCUSSION

From the XRD profile of the sample (YSBCO) it is understood that the intensities of the peaks decreases on increase of temperature[7]. This intensity anomaly can be explained as the rigorous thermal agitations of the lattice planes resulting in the formation of new phase at the high temperature. When a material heated from room temperature to high temperature, the amplitude of the thermal vibrations increases. That means, as the atomic vibration amplitude increases, the intensity of the diffracted beam also decreases because it has the effect of smearing out lattice planes. The intensity of a diffracted beam decreases as the temperature is raised[15]. XRD spectrum for the different temperatures gave a clear idea about the maximum intensity peak shifting corresponds to the different treating temperatures. And also get the maximum intensity peak difference [2]. The peak broadening in the XRD patterns clearly indicated the nature of the very small nanocrystals [7]. Heat treatment causes the particles to anneal and form larger grains, which of course indicates that the particles become larger. Hence, the large particle size of sample at 950^{o}C is expected / confirmed. This also agrees with the higher crystallinity, as having larger grains means more long-range order, and hence more crystallinity[16, 17].

Material Characterization by thermal technique is employed to evaluate the Chemical and Physical Properties of Ceramic Materials. From the TGA / DTA curves it is considered to be having mainly three stages. The material YSBCO which initially has a weight of 16.531mg attains 16.491 mg and a fall in weight to 16.006 at 450^{0}C with a loss of 2.95%. This may be due to distortion in lattice. Then there is a reduction in weight and it becomes 15.714 at 832.0419°C.Weight loss is 1.5%. From there onwards weight is almost constant. These can be seen from the TG curve given above. Moisture content is not appreciably observed. Loss on ignition is in total 5% upto 1000^{0}C. In these temperatures since the sample is a good ceramic material much loss is not expected.

The thermal study of the sample clearly shows that the phase transition is taking at a very high temperature. The DTA curves are in confirmity with these observations. The free energy inside the interface regions of the nano materials affects the phase transitions. Changes in lattice imperfections also arise due to the miniature size of the particles. Hence nano materials have a different or modified behaviour than that of the bulk materials. Polymorphism in crystalline structure also can be observed. Thus the thermal stability of the sample can be confirmed from the TGA, DTA & DSC analysis. From the DSC curve of the sample it is evident that the process is endothermic.

From the EDAX spectrum of YSBCO (Fig. 5), the information on the elemental composition of the material can be elucidated. The dominant peak positions at 1.922keV (Y Lα), 1.806keV (Sr Lα), 2.419keV (Bi M), 0.930, 8.040keV (Cu Lα, Kα), 0.525keV (O Kα) correspond quite well to the energy pattern of the corresponding materials (Y, Sr, Bi, Cu and O) reported in the EDAX international chart. Percentage composition according to EDAX analysis is shown in table 1.

4. CONCLUSION

YSBCO ceramics were prepared successfully and characterized by XRD, SEM, and particle size measurement. XRD data confirmed the formation of the perovskite phase structure [7]. In this work TGA, DTA and DSC analysis was carried out and confirmed that much loss on ignition is not observed, characteristic of a good ceramic material. The EDX analysis indicates the presence and percentage of the elements existing in the sample and it agree with the stoichiometric relations of the prepared compound. Each of these thermal techniques provides unique information that can be used to evaluate the thermal and mechanical properties of the end product.

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