ISSN 2321-807X

THERMOGRAVIMETRIC ANALYSIS OF NANO CRYSTALLINE CERAMIC PbSrCaCuO

Reenu Jacob[1], Jayakumari Isac[1*]

[1,1*]Centre for Condensed Matter, Department of Physics, CMS College, Kottayam, India

Email: reenujacob12@gmail.com, drjayacmscollege@gmail.com

ABSTRACT

Nano crystalline ceramic PbSrCaCuO was prepared by the solid state technique via a high-energy ball milling process through mechanically assisted synthesis.The prepared sample was then subjected to calcinations to acquire the desired homogeneity and phase formation. To establish the elemental composition EDX analysis was done.TGA and DTA were used to study the behaviour of the sample upon heating to a high temperature.

Indexing terms/Keywords

PbSrCaCuO; Thermo gravimetric analysis(TGA); Differential Thermal Analysis(DTA).

Academic Discipline And Sub-Disciplines

Faculty Of Science,Physics

SUBJECT CLASSIFICATION

Condensed Matter Physics, Nanoceramics Composite

TYPE (METHOD/APPROACH)

Full Research Article, Experiment Along With Theory And Analysis.

Council for Innovative Research

Peer Review Research Publishing System

Journal: Journal of Advances in Chemistry

Vol. 10, No. 7

editorjaconline@gmail.com

www.cirjac.com

ISSN 2321-807X

INTRODUCTION

Conventional solid state reaction method is a common and effective way to fabricate modern ceramics [1]. The perovskite systems are considered as a potential candidate in ceramic industry. Ceramic materials are brittle, hard, strong in compression, weak in shearing and tension. They withstand chemical erosion that occurs in an acidic or caustic environment. In many cases withstanding erosion from the acid and bases applied to it. The Lead Strontium Calcium Copper Oxide (PbSrCaCuO) is a type of cuprate ceramic superconductor. All superconducting cuprates are layered materials having a complex structure described as a superlattice of superconducting CuO2 layers separated by spacer layers. Pb substitute oxides all have layered perovskite like crystal structure, and manifest superconductivity above 77 K [2].

The phase transformations in nano materials due to temperature change is much different from that of bulk crystals. Phase transformations in nano structured articles are reported by Chang.et.al[3]. The free energy of nano particles are always higher than that of its conventional counterpart [4].

In this work the authors gives information about the thermal behaviour of PbSrCaCuO ceramic material. Thermo Gravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) can be used to study the thermodynamical behaviour of nanoparticles [5-7]. Large amount of energy is stored in the grain boundaries and in other types of defects. Nanophase materials are in metastable state of thermal inequilibrium. Hence one can get information regarding the long-term thermal stability of such systems by studying the transition from nanophase-state to thermal equilibrium state[8].The EDX spectrum of ceramic PbSrCaCuO gave the information on the elemental composition of the material.

2.EXPERIMENTAL.

2.1. Preparation of The Sample:

Ceramics with the chemical formula **PbSrCaCuO** were prepared by the solid state reaction technique according to their molecular formula using a high-energy ball milling process through mechanically assisted synthesis. For preparing sample, the reagent grade chemicals of high purity Lead dioxide, Strontium Carbonate, Calcium Oxide and Cuprous oxide powders were used as the raw materials and weighed according to their molecular formula. The required powders were mixed mechanically , ball milled for three weeks with suitable zirconium balls to insure homogeneity and milling. After milling the material was calcined to a temperature 950˚C in a special furnace with oxygen flow arrangements.

2.2. TGA –Analysis.

Thermo Gravimetry (TG) is the branch of thermal analysis which examines the mass change of a sample as a function of temperature in the scanning mode or as a function of time in the isothermal mode.

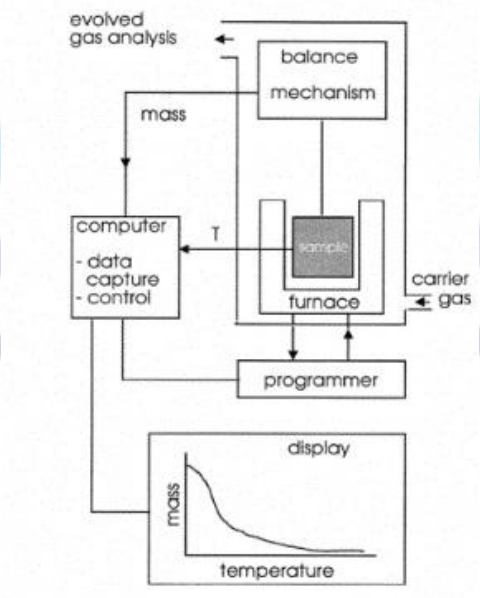

Figure 1. TGA process

Thermo gravimetric curves are characteristic for a given polymer or compound because of the unique sequence of the physiochemical reaction that occurs over specific temperature ranges and heating rates and are function of the molecular structure.

ISSN 2321-807X

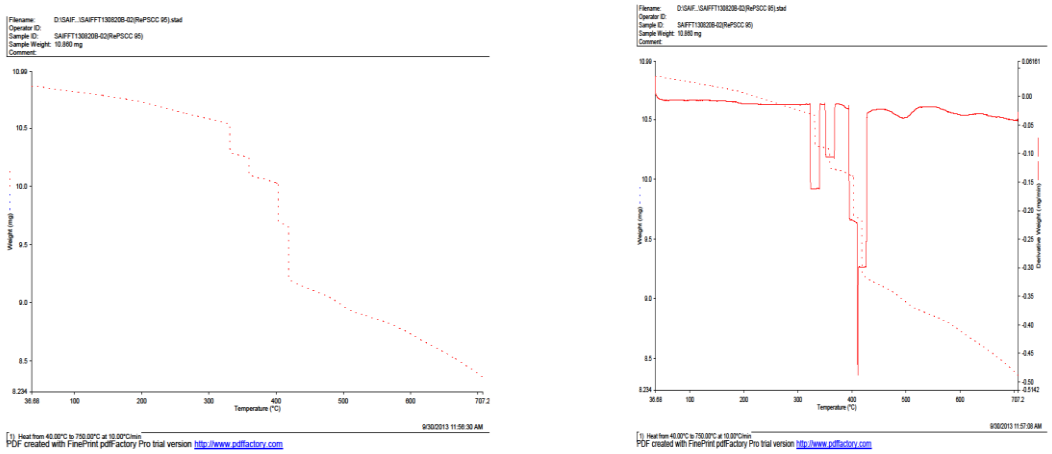

Figure 2. TG and the derivative curve of PbSrCaCuO

The mass change characteristics of a material are strongly dependent on the experimental conditions employed. 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 (meaning they are dependent on absolute temperature and time spent at that temperature), 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, respectively[9].TGA curve and the DTG curve of the sample are plotted in Fig 2.

2.3.Differential Thermal Analysis

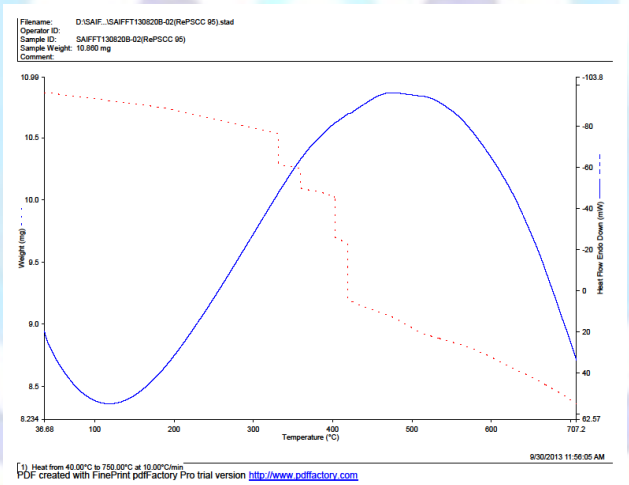

Figure 3. Differential Thermal heat flow curve of PbSrCaCuO

The sample PbSrCaCuO with a weight of 10.866mg: initially at temperature 30◦C reduces to 10.4mg at temperature of 310◦C.The weight or mass percentage continuously reduces and the percentage of loss is 3.7%.

At 320◦C the weight again reduces to 10.2mg with a mass loss of 5.55%. The graph shows a steep reduction in mass at 390◦C and again at 410◦C. Above 707◦C the weight reduces and reaches almost a constant value of 8.4mg with a loss percentage of 22%. These stages are evident from the Derivative (DTA) graph of the sample PbSrCaCuO as plotted in fig 3.

2.4. EDX Analysis

To establish the elemental in corporation in the sample PbSrCaCuO EDX had been taken (figure 4). The data relating to the integrated counts of X-ray photoelectrons taken for a definite time interval had been helpful in getting the quantitative analysis of the sample PbSrCaCuO**.** From the EDX spectrum, the five dominant peak positions at 8.04 KeV, 3.69 KeV, 2.34 KeV, 1.80 KeV, 0.525KeV correspond quite well to the energy pattern of the corresponding materials (Cu, Ca, Pb, Sr and O) reported in the EDAX international chart, giving the evidence that Pb and Sr are dominant in PbSrCaCuO sample. **Table 2**,shows the percentage of the elements in the prepared PbSrCaCuO sample.

ISSN 2321-807X

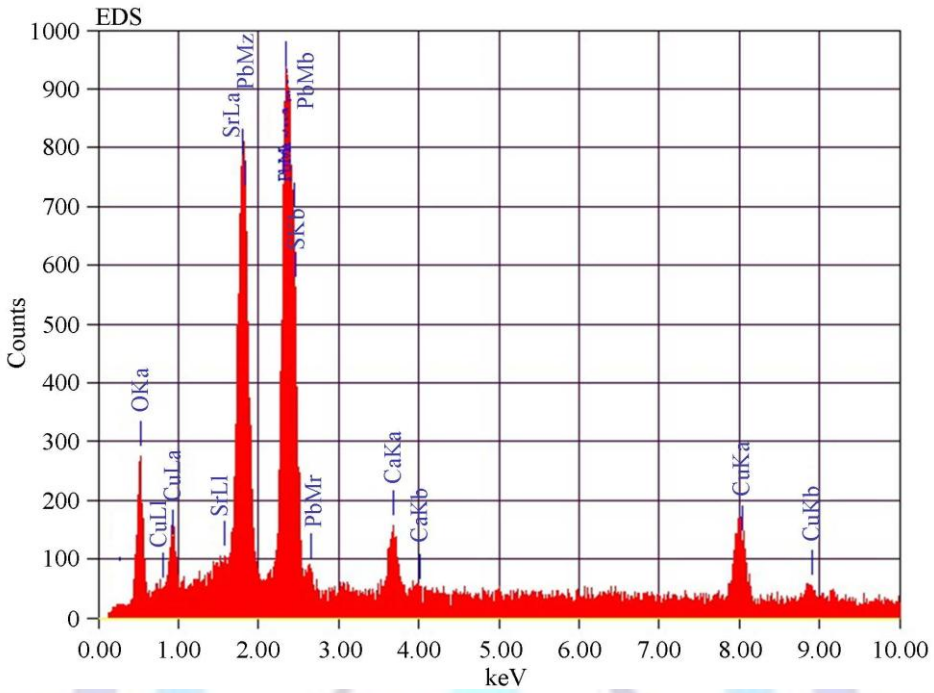

Figure 4. EDX of PbSrCaCuO

Table 2. Material Content (EDX).

Material Content	%
Pb	62.93
Sr	20.8
Cu	10.33
Ca	2.27
O	3.58

3. Results and Discussion:

The XRD analysis of this material is already reported[2]. From the XRD profile it is understood that the intensities of the peaks decreases on increase of temperature. 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. Thus the reinforcement of waves scattered at the Bragg angle by various parallel planes is not as perfect as it is for a crystal with fixed atoms. This reinforcement requires that the path difference, which is a function of the plane spacing *d*, between waves scattered by adjacent planes be an integral number of wavelengths. The thickness of the planes is 2*u*, where *u* is the average displacement of atom from its mean position. Under these conditions reinforcement is no longer perfect, and it becomes more imperfect as the ratio *u*/*d* increases, *i.e*., as the temperature increases since that increases *u*, or as *θ* increases,since high-*θ* reflections involve planes of low *d* value. Thus the intensity of a diffracted beam decreases as the temperature is raised. [10].

EDX data supported the existence of the materials in the composite prepared. From the EDX spectrum, the five dominant peak positions at 8.04 KeV 3.69 KeV, 2.34KeV, 1.80 KeV, 0.525KeV correspond quite well to the energy pattern of the corresponding materials (Cu, Ca, Pb, Sr and O) reported in the EDAX international chart, giving the evidence that Pb and Sr are dominant in PbSrCaCuO sample. **Table 2**,shows the percentage of the elements in the prepared PbSrCaCuO sample.

Thermal analysis of the sample PbSrCaCuO was successfully carried out by the TGA & DTA results. TGA/DTA curves exhibits two important stages- first part shows a weight loss corresponding to an endothermic process reported between 120◦C to 200◦C which corresponds to the loss of free bound molecules. Second part shows the exothermic curve or decomposition of the metal with the liberation of gases. No significant weight loss occurs further shows the beginning of crystallisation of the perovskite phase. The curves showed the temperatures at which the sample causes dehydration and

the intermediate stages the sample crossed before forming the final product. The loss of mass percentage was also calculated. The DTA curves are in confirmity with these observations.

The study of the sample clearly shows that the phase transition is taking at a very high temperature. 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.[4].Thus the thermal stability of the sample can be confirmed from the TGA/DTA analysis.

Acknowledgments

The authors are thankful to SAIF, Kochi for providing the data analysis and to the Principal, CMS College, Kottayam, Kerala for providing the facilities.

References

1. Leow Chun Yan, Jumian Hassan, Mansor Hashim, Wong Swee Yin, Tan Foo Khoon and Wong Yick Jeng. Effect of Sintering Temperatures on the Microstructure and Dielectric Properties of SrTiO3 World Applied Sciences Journal 14 (7): 1091-1094, 2011 ISSN 1818-4952 © IDOSI Publications, 2011
2. Vinila, V. S., Reenu Jacob, Anusha Mony, Harikrishnan G. Nair, Sheelakumari Issac, Sam Rajan, Anitha S.Nair, Jayakumari Isac. 2014. Xrd Studies On Nano Crystalline Ceramic Superconductor Pbsrcacuo At Different Treating Temperatures, Crystal Structure Theory And Applications, 2014, 3, 1-9 Published Online March 2014 In Scires, Http://Dx.Doi.Org/10.4236/Csta.2014.31001.
3. X Y Qin,X J Wu,and L F Cheng:Nanostructured materials2,99(1993)
4. Sankaranarayanan Potty.S,2001.Synthesis of nanoparticles of certain materials and their Characterisation.
5. W. W. Wendlandt, Thennal Analysis, 3'* Edition, John Wiley & Sons, New York (1986)
6. Y. D. Yao, Y. Y. Chen, *(2.* M. Hsu, H. M. Lin, C. Y. Tung, M. F. Tai, D. H. Wang, K. T. Wu, C. T. Suo, Nanostructured Materials ***6*** (1995) 933
7. M. E. Brown, Themla1 Analysis, Techniques and Applications, Academic Press, New York (1990).
8. EXPERIMENTAL TECHNIQUES - shodhganga.inflibnet.ac.in/bitstream/10603/468/8/08_chapter2.pd a
9. PST 522E – Synthesis and Characterization of Macromolecules –Ch12 Thermogravimetric Analysis.
10. Cullity, B.D. (1978) Elements of X-Ray Diffraction. 2nd Edition, Addison-Wesley Publishing Company.Inc., Phillippines.