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Particle Size Determination And Morphological Study Of Nano Crystalline Ceramic Superconductor $La_{0.1}zry_{0.9}baca_2cu_3o_{4.5+X}$ At Three Different Temperatures

Physics

KEYWORDS : Lanthanum Zirconium Yttrium Barium Calcium Copper Oxide (La0.1ZrY0.9BaCa2Cu3O4.5+x), high temperature superconductors, XRD, SEM

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ABSTRACT

sToday we are familiar with a large series of "high temperature superconductors" based on the ceramic materials with layers of Copper-oxide spaced by layers containing Barium and other atoms. Most of these type super conductors containing yttrium compounds, it has a regular crystal structure while the Lanthanum version is classified as a solid solution. Here author calculated the particle size of the superconductor Lanthanum Zirconium Yttrium Barium Calcium Copper Oxide (La0.1ZrY0.9BaCa2Cu3O4.5+x) at different treating temperatures. It was prepared by the solid state reaction method via a high-energy ball milling process through mechanically assisted synthesis and calcined in a specially designed high temperature furnace. Here the authors characterized the sample by X-ray Diffraction (XRD), SEM and EDX. The structure of this material is found to be orthorhombic and is confirmed from the XRD results with JCPDS files and XPERT-PRO software, while a≠b≠c and α=β=γ=900. The particle size determination by the XRD analysis with Debye Scherrer's formula, SEM, instrumental broadening and Williamson-Hall Plot method confirmed that the sample's particle size is less than 100 nm.

INTRODUCTION

Ceramics can withstand very high temperatures such as temperatures that range from 800°C to 1,600°C.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. Also withstanding erosion from the acid and bases applied to it. At low temperatures, La_2O_3 has an A-M2O3 hexagonal crystal structure. The La_{3+} metal atoms are surrounded by a 7 co-ordinate group of O_2 atoms, the oxygen ions are in an octahedral shape around the metal atom and there is one oxygen ion above one of the octahedral faces Yttrium oxide (Y2O3) nanoparticle is an air-stable, solid substance. In this work the authors describe the preparation of $La_{0.1}ZrY_{0.9}BaCa_2Cu_3O_{4.5+x}$ ceramic material and characterized to show good quality, homogeneity and the desired non stoichiometry of the sample prepared. The results were analyzed by X-ray diffraction (XRD), SEM, EDX. The particle size was determined from XRD details by Debye Scherrer formula. The SEM studies revealed that its particle size is in hundred nanometer range. The EDX spectrum of $La_{0.1}ZrY_{0.9}BaCa_2Cu_3O_{4.5+x}$ gave the information on the elemental composition of the material. Instrumental Broadening and Williamson-Hall Plot method utilized to found the particle size and strain of the material.

2. Materials and Experimental Methods

$La_{0.1}ZrY_{0.9}BaCa_2Cu_3O_{4.5+x}$ has perovskite structure. The perovskite structure is adopted by many oxides. The representative structure of perovskite compounds is cubic, the compounds in this family may possess or undergo some distortion. The orthorhombic and tetragonal phases are most common variants. Figure.1 shows the structure of $La_{0.1}ZrY_{0.9}BaCa_2Cu_3O_{4.5+x}$ and T_c of some related high temperature superconductor materials.

Lanthanum-doped ceramics with the chemical formula $La_{0.1}ZrY_{0.9}BaCa_2Cu_3O_{4.5+x}$ was prepared by the solid state thermochemical reaction technique. For the Sample preparation reagent grade chemicals of high purity (99.99%) Lanthanum oxide, Zirconium oxide, Yittrium oxide, Barium carbonite, Calcium oxide and Copper oxide powders were used as the raw materials and weighed according to their molecular formula.

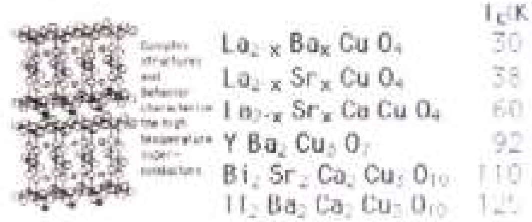

Figure.1 Structure of $La_{0.1}ZrY_{0.9}BaCa_2Cu_3O_{4.5+x}$

The powders of the required ceramics were mixed mechanically. Mechanical mixing is usually done by hand mixing in agate mortar for very long time. Then ball milled with suitable balls for long time to insure homogeneity and attrition milling. Then the material was calcined at different temperatures, 300°C and 950°C. After the furnace is off, on cooling the oxygen is allowed to flow into the furnace at intervals (Oxygen Annealing). A final furnace temperature of 950°C is maintained after the intermediate firings. Higher temperature than this will result in a material that is much harder to regrind. Temperatures above 1050°C may destroy the crystal structure. Then X-ray diffraction spectrum of these materials was taken. For experimental conformation calculated particle size value, Scanning Electron Microscopy (SEM) photograph was taken. The composition details of the prepared ceramics were determined from EDX.

2.1. XRD Analysis.

X-ray Diffraction pattern for the three different temperatures in steps for the sample $La_{0.1}ZrY_{0.9}BaCa_2Cu_3O_{4.5}$ was taken using Bruker AXS D8 advance diffractometer (figure 2). The diffractometer with radiations of wavelength 1.54184Å having Nickel filter, equipped with X-ray generator 1140/90/96 having X-ray source KRISTALLOFLXE 780,KE,4KE with wide angle goniometer PW1710/70 with single pen recorder pm 8203 and channel control PW1390 at 35kV, 10mA is used for the purpose. The scanning speed of the specimen is 2 degree/minute. From the XRD results, the obtained d values compared with the JCPDS (Joint Committee on Powder Diffraction Standards) file values[10,11] and XPERT PRO programme was applied. So it can be concluded that this crystal is found to be orthorhombic system:

a=15.3598, b=11.1644, c=3.3835; α=β=γ=90°

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RESEARCH ARTICLE

STUDIES ON THERMAL BEHAVIOUR OF NANO CRYSTALLINE CERAMIC LAZRYBACA2CU3O11

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ABSTRACT

Nano crystalline ceramic $LaZrYBaCa_2Cu_3O_{11}$ is prepared by the solid state reaction method. The process is mechanically done by high energy ball milling. The mixed powder is synthesised and calcined in a specially designed high temperature furnace. The pure material prepared is then calcined in ambient air and optimum temperature so that the desired homogeneity and phase formation is acquired. Using TGA, DTA and DSC thermal behaviour of the sample at a high temperature is studied. EDX analysis is done to find out the elemental composition.

INTRODUCTION

$LaZrYBaCa_2Cu_3O_{11}$ has perovskite structure. The most familiar ceramic superconducting materials have perovskite structure (Willander *et al.*, 2012). The perovskite structure is adopted by many oxides. The representative cubic structure of perovskite compounds undergo some distortion. The most common variants are orthorhombic and tetragonal phases (Anitha S. Nair *et al.*, 2014). The perovskite systems are considered as a potential candidate in ceramic industry. Ceramic materials are inorganic non metallic solid comprising metal, non metal or metalloid atoms primarily held in ionic and covalent bonds. They withstand chemical erosion that occurs in an acidic or caustic environment.

Ceramic materials are brittle, hard, Strong in compression, weak in shearing and tension. Conventional solid state reaction method is a common and effective way to fabricate modern ceramics (Leow Chun Yan *et al.*, 2011). Ceramics materials generally can withstand very high temperature such as 1000°C to 1600°C (1800°F to 3000°F). Conventional solid state reaction method is applied here to fabricate modern ceramics (Leow Chun Yan *et al.*, 2011). The Lanthanum Zirconium Yttrium Barium Calcium Copper Oxide ($LaZrYBaCa_2Cu_3O_{11}$) is a type of perovskite ceramic superconductor with high dielectric constant. Before final heating at 950°C, the material $LaZrYBaCa_2Cu_3O_{11}$ is treated at different temperatures, 30°C, 500°C and 800°C (Anusha Mony *et al.*, 2014). It is having a complex structure.

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Detailed understanding of this class of materials will help electronic industry in planning, design and processing of these materials. High dielectric constant (High-K) ceramic composites have become potential candidate materials for integration into high frequency electronics. Here $LaZrYBaCa_2Cu_3O_{11}$ nano crystalline ceramic high-TC superconductor material is fabricated by the solid state thermo chemical reaction technique and it was characterized to show good quality, homogeneity and the desired stochiometry of the sample prepared (Anusha Mony *et al.*, 2014). The results were analyzed by X-Ray Diffraction (XRD), SEM, and EDX. The thermal behaviour of lanthanum zirconium yttrium barium calcium copper oxide ($LaZrYBaCa_2Cu_3O_{11}$) calcined at 950^0C is presented here. TGA, DTA and DSC is used to analyze thermal behaviour of nanoparticles (Wendlandt *et al.*, 1986; Yao *et al.*, 1995 and Brown *et al.*, 1990), at a high temperature. Elemental composition is obtained using EDX.

Nanophase materials are in metastable state of thermal inequilibrium. Large amount of energy is stored in the grain boundaries and in other types of defects. By studying the transition from nanophase-state to thermal equilibrium state one can get information regarding the long-term thermal stability of such systems (Experimental Techniques). The phase transformation in nano materials due to temperature change is much different from that of bulk crystals. The free energy of nano particles are always higher than that of its conventional counterpart (Sankaranarayanan Potty, 2001). Phase transformations in nano structured materials are reported (Qin, Wu and Cheng, 1993).

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URBACH AND BANDGAP ENERGY ALONG WITH OPTICAL CONSTANTS ANALYSIS OF NANOCRYSTALLINE LZYBCCO CERAMICS

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Abstract

The crystalline ceramic lanthanum zirconium yttrium barium calcium copper oxide(LZYBCCO) was synthesized thermo chemically by solid state method at different treating temperatures. Depending on the surface chemistry and the synthesis conditions, the nano particles tend to form soft or hard agglomerates. Hard agglomerates consist of smaller particles which are connected to each other by sinter necks. They can be destroyed by high energy milling only. Soft agglomerates are accumulations of isolated particles that are connected to each other by attractive physical interactions like hydrogen bridge or van-der-Waals forces. They can be disrupted into smaller particles by shear forces generating mechanical stress gradients. A characteristic feature of all solid-state reactions is that they involve the formation of product phase(s) at the interfaces of the reactants. UV-VIS analysis of the sample was carried out. Tunable band gaps can be obtained by varying annealing temperatures. The optical constants of refractive index, extinction coefficient, normal-incidence reflectivity, and absorption coefficient showed systematic variation with temperature. The dispersion of refractive index was analyzed by the Wemple-DiDomenico single-oscillator model. Urbach and Band energy was also calculated.

Keywords

LZYBCCO, band gap energy, Dispersion, Wemple-DiDomenico model, refractive index, urbach energy.

1.Introduction

Nano science and nanotechnology is a vigorously developing field in research and technology for past few decades. Nano particles are particles with diameters below the micron dimension: generally, below 0.1µm. It can be also defined as particles with properties depending directly on their size, i.e., optical, electrical and magnetic properties. Nano materials are used in engineering, industrial, electronics and medical field. The perovskites are an extremely important class of ceramic nano materials. These compounds have a chemical formula ABO_3 where A is either a monovalent or divalent cation and B is either a pentavalent or tetravalent metal[1]. Many different optical properties of ceramic products are of concern in different applications. The optical properties of dielectric materials are generally of interest because of their good transmission in the optical part of the spectrum as compared with other classes of materials. At short wavelengths this good transmission is terminated at the ultraviolet absorption edge, which corresponds to radiation energies and frequencies ($E=h\nu=hc/\lambda$) where absorption of energy arises from electronic transitions between levels in the valence band to unfilled states in the conduction band[2]. Both the index of absorption and refractive index are necessary to describe the optical properties of a ceramic. The absorption index is a function of wavelength and is mostly related to the absorption coefficient $\beta=4\pi k/\lambda$. For a single-phase material, the fraction of light transmitted is given by the absorption coefficient and sample thickness; $dI/I0=-\beta dx$ and $T=I/I0=\exp(-\beta x)$, $\ln I/I0=-\beta x$ where I0 is the initial density, I is the transmitted intensity, x is the optical path length and T is the fraction transmitted. This overall transmission is given for normal incidence by $T^{1}=I_{tr}/I_{out}=(1-R)^2\exp(-\beta x)$ Where R is the reflectivity[3]. In solid state physics a band gap, is an energy range in an ideal solid where no electron states can exist. This is equivalent to the energy required to free an outer shell electron from its orbit about the nucleus to become a mobile charge carrier, able to move freely within the solid material [4]. In the present work the authors describes the optical behavior along with urbach and band gap energy of LZYBCCO nano crystalline superconductor material. The energy band gap values of the sample were analyzed for different temperatures and they are fundamentally important to the design of practical devices[5]. In the case of insulators the

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BANDGAP ANALYSIS OF NANO CRYSTALLINE L0.1ZY0.9BCCO CERAMICS

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ABSTRACT

Crystalline Ceramic Lanthanum Zirconium Yttrium barium Calcium Copper oxide($L_{0.1}ZY_{0.9}BCCO$) was prepared by a high-energy ball milling process through mechanically assisted synthesis at a high temperature to acquire the desired homogeneity and phase formation. Inorder to study the optical properties like reflectivity, absorptivity, refractive index, the UV-VIS analysis of the above nonstochiometric sample was carried out. The dispersion of refractive index was analyzed by the Wemple-DiDomenico single-oscillator model.The band gap energy of the sample was elucidated from the Tauc plot. The refractive index n was calculated and the results obtained are plotted with the wavelength.

Indexing terms/Keywords

L0.1ZY0.9BCCO; Dispersion; Wemple-DiDomenico model; band gap energy; refractive index; Urbach energy.

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Scientific Research

Studies on Nano Crystalline Ceramic Superconductor $LaZrYBaCa_2Cu_3O_{11}$ at Three Different Temperatures

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The high temperature superconductors are ceramic materials with layers of Copper-oxide spaced by layers containing Barium and other atoms. The Yttrium compound is somewhat unique in that it has a regular crystal structure while the Lanthanum version is classified as a solid solution. The Yttrium compound is often called the 1-2-3 superconductor because of the ratios of its constituents. Lanthanum Zirconium Yttrium Barium Calcium Copper Oxide (LaZrYBaCaCuO) was prepared by the usual solid state reaction method. In order to show the viability of the proposed method, super-conducting powder was prepared in special furnace. The sample was analyzed by X-ray Diffraction (XRD), Particle size determination, SEM and EDX. The comparison of XRD results with JCPDS files confirmed the orthorhombic structure of the sample with $a \neq b \neq c$ and $\alpha = \beta = \gamma = 90^\circ$. Scanning Electron Microscopy (SEM) studies revealed that its particle size is in the nanometer range. It also confirmed the calculated value of particle size from Debye Scherrer's formula. EDX spectrum shows the elements of the sample. X-ray instrumental peak broadening analysis was used to evaluate the size and lattice strain by the Williamson-Hall Plot method.

Lanthanum Zirconium Yttrium Barium Calcium Copper Oxide (LaZrYBaCaCuO), XRD, SEM, EDX, Debye Scherrer's Formula, Instrumental Broadening, Williamson-Hall Plot Method

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