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Development of bismuth-based electronic materials from Indian red mud

R.N.P. Choudhary[a], C. Behera[a,*], Piyush R. Das[a], R.R. Das[b]

[a]Multifunctional Material Research Laboratory, ITER, Siksha 'O' Anusandhan University, Bhubaneswar-751030, India
[b]R&D, GIET, Gunupur, Odisha, India

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Abstract

This paper reports the synthesis and characterization of bismuth-based new electronic materials fabricated from silicon-free Indian red mud (RM). The materials were prepared using a solid-state reaction technique. Preliminary X-ray (XRD) structural analysis exhibits the formation of compounds closely related to $BiFeO_3$ (with the presence of some additional impurity phases). The surface morphology recorded by scanning electron microscopy (SEM) reveals a polycrystalline nature of texture with uniform distribution of grains. Some dielectric parameters (relative permittivity (ε_r) and tangent loss (tanδ)) of the compounds are almost independent on frequency in the low-temperature range. In the high-temperature region, relative permittivity of the compounds is found to be higher with low tangent loss. Detailed studies of impedance and related parameters exhibit that the parameters are strongly dependent on temperature, and show a good correlation with their microstructures. The bulk resistance, evaluated from complex impedance spectra, is found to be decreasing with rise in temperature. The nature of impedance spectra exhibits a typical negative-temperature-co-efficient of resistance (NTCR) behavior similar to the semiconductors. Studies of electric modulus show the presence of hopping conduction mechanism in the system with non-exponential type of conduction relaxation. The low-leakage current and NTCR behavior of the materials are found to be consistent with their *J–E* characteristics. The ac conductivity spectra of the materials show a typical-signature of an ionic conducting system, and are found to obey Jonscher's universal power law.
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Keywords: Red mud; XRD; Bismuth ferrite; Impedance spectroscopy

1. Introduction

Because of the huge storage/dumping problems and creation of environmental pollutions, proper utilization of red mud (RM) has been a subject of major scientific research in the world. During last few decades, several efforts have been made to solve the problems of the red mud. Some encouraging and interesting results were obtained on utilization of this waste material for commercial uses. Some of them are; production of iron, steel, titanium, alumina, minor constituents elements, building materials (i.e., bricks, light weight aggregates, roofing and flooring tiles, etc), catalysis, ceramics (pottery, sanitary ware, special tiles and glasses, glazes, ferrites), filler, fertilizer [1–3]. Unfortunately, economics of the successful recovery of major constituents of RM is still not favorable; hence, persistence of disposal problem of huge amount of RM remains. For example, the suggestion on cost effective recovery of major constituent of RM such as iron [4–7], titanium dioxide, vanadium, rare earths [8–11] is still a big challenge for researchers. However, recent efforts for production and utilization of building materials using red mud (such as cement, bricks, roofing tiles and glass–ceramics) have been found very successful and rewarding. The utilization of RM for the above applications has three-fold benefits; (i) the bulk production of building materials could eliminate the disposal problem, (ii) utilization of waste material (red mud) as a low-cost raw material for various industrial products and (iii) easy to implement developed methods for production of industrial products in most of the countries of the world [12–14]. Detailed literature survey clearly reveals that attempts have been made to utilize the red mud only for large size and scale industrial products. The national aluminium company (NALCO) of this region produces a huge amount of red mud. The compositional analysis of the red mud of this company shows that it contains

*Corresponding author. Tel.: +91 9438651271.
E-mail addresses: cdbehera1986@gmail.com, chakradharbehera@soauniversity.ac.in (C. Behera).

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various oxides with different percentages (shown in parenthesis) such as Al_2O_3 (14.14), SiO_2 (11.53), Fe_2O_3 (48.50), TiO_2 (5.42), CaO (3.96), Na_2O (7.50), P_2O_5 (0.297), V_2O_5 (0.116), K_2O (0.058), MnO (0.17), MgO (0.049) and others (8.26). In order to get silicon-free red mud with the maximum percentage of Fe_2O_3, it was further purified for the present research work. Hence; it was proposed to use red mud for the fabrication of bismuth-based ferrite or multiferroic by combining it with bismuth oxide (Bi_2O_3). Recently, we have developed bismuth-based bismuth ferrite using silicon-containing red mud [15].

Detailed literature survey reveals that no attempt has been made to develop multiferroics (electronic materials) using industrial waste material (red mud) for dielectric, ferroelectric, piezoelectric, ferromagnetic and multiferroic devices. In view of the above, we have fabricated and characterized red mud based-bismuth ferrite ($BiFeO_3$) using standard experimental techniques in different experimental conditions (temperature, atmosphere, frequency, ac and dc signal). In this paper, we report synthesis and studies of structural and electrical properties of bismuth ferrite for its possible multifunctional applications [16,17].

2. Materials

2.1. Materials preparation

The polycrystalline samples of red mud based bismuth ferrites were prepared using a standard solid-state reaction or mixed-oxide technique. Bismuth oxide (Bi_2O_3) (99.9%, M/s LOBA Chemie Pvt. Ltd., India) was mixed with silicon-free red mud (RM) in different percentages (by weight). Three different types of samples were prepared such as (i) S1 (RM-8 gm + Bi_2O_3-2 gm), (ii) S2 (RM-6 gm + Bi_2O_3-4 gm), and (iii) S3 (RM-5 gm + Bi_2O_3-5 gm) with red mud (S4). These ingredients were initially mixed mechanically in an agate-mortar for 2 h followed by wet grinding (in methanol medium) for another hour to get homogeneous mixture of the constituents. Subsequently, it was calcined at 950 °C for 4 h in air. The formation of the desired compounds was checked by preliminary X-ray (XRD) structural analysis. The calcined powder so obtained was cold pressed into cylindrical pellets (10 mm diameter and 1–2 mm thickness) using polyvinyl alcohol (PVA as the binder) using a hydraulic press at an iso-static pressure of 4×10^6 N/m^2. The pellets were then sintered at an optimized temperature 1000 °C for 4 h in air atmosphere. The sintered pellets were then polished with fine emery paper to make their faces smooth and parallel. The pellets were finally coated with conductive silver paint, and dried at 150 °C for 8 h before carrying out electrical measurements.

2.2. Material characterization

X-ray diffraction (XRD) data of the materials was obtained in the wide range of Bragg angle (2θ) ($20 \leq 2\theta \leq 70$) at scan speed 2°/min by an X-ray powder diffractometer (Rigaku Miniflex, Japan) using CuK_αradiation (λ=1.5405 Å) at room temperature. Scanning electron micrographs of the materials were taken with scanning electronic microscope (SEM-JEOL-JSM, model 6510, USA) to study the surface morphology/microstructure of the sintered sample pellets. The impedance and related parameters were obtained in the temperature range of 30–500 °C using a computer-controlled phase sensitive meter (PSM/LCR N4L, Model: 1735, UK) in the frequency range of 100 Hz–1 MHz. An input ac signal/voltage of small amplitude was applied across the sample cell followed by the thermal stabilization for 2 h prior to the measurements. The *J–E* characteristics of the pellet samples were obtained in the temperature range of 25–550 °C using a high-resistance electrometer (KEITHLEY INSTRUMENTS INC. 6517B). A constant voltage was applied across the samples to measure their dc conductivity.

2.2.1. Structural and micro-structural properties

Fig. 1 shows the room-temperature XRD pattern of four samples S1, S2, S3 and S4 (red mud) The patterns consist of a number of sharp peaks which are different from those of the ingredients confirm the formation of new polycrystalline compounds. Most of the peaks of S1, S2, S3 and S4 are closely related to BFO. Some additional small peaks may be attributed to some unknown phases of raw red mud. Using 2θ value of diffraction peaks, prominent peaks of the patterns were indexed in the cubic or rhombohedral crystal system of BFO unit cell using computer software POWD [18]. A good agreement between observed (obs.) and calculated (cal.) inter-planar spacing d (i.e., $\sum \Delta d = d_{obs} - d_{cal} =$ minimum) was found in the rhombohedral system for S1.

Fig. 2 compares the room temperature scanning electron micrographs of the samples. On increasing the Bi_2O_3 concentration, the grain size goes on increasing. The regular distribution of grains (similar in nature and size) shows that the materials are densely packed with small number of voids.

2.2.2. Dielectric properties

Fig. 3 shows that the relative dielectric constant/permittivity (ε_r) and tangent loss ($\tan\delta$) parameters of S1, S2 and S3 are

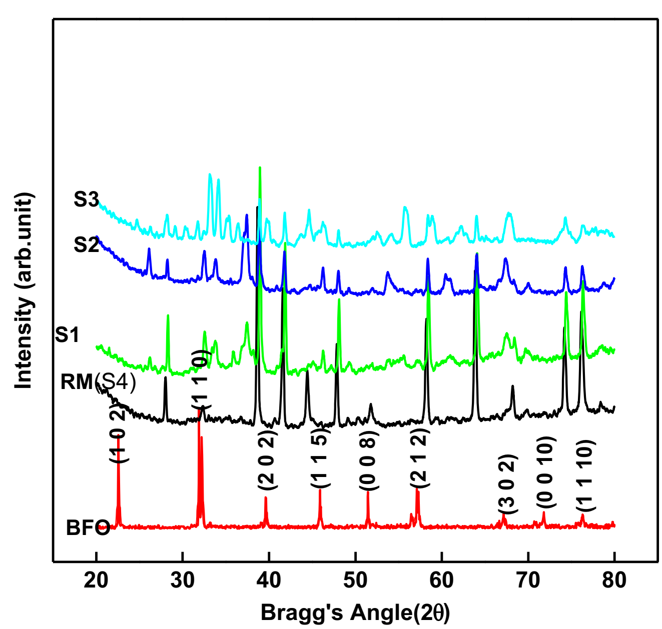

Fig. 1. Room temperature XRD pattern BFO, S4(RM), S1(RM-80%), S2(RM-60%), S3(RM-50%).

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