Investigation of physical and mechanical properties of (BaSnO₃)ₓ(Bi,Pb)-2223 composite

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Abstract: The effect of BaSnO₃ nanoparticles addition on the structural and mechanical properties of (Bi,Pb)-2223 superconducting phase by means of X-rays diffraction analysis (XRD), scanning electron microscope (SEM), electrical resistance and Vickers microhardness measurement was studied. BaSnO₃ nanomaterial and (BaSnO₃)ₓ(Bi,Pb)-2223 superconducting samples were prepared using co-precipitation method and standard solid-state reaction techniques, respectively. From XRD data, the addition of BaSnO₃ into (Bi,Pb)-2223 phase does not affect the tetragonal structure and the lattice parameters. SEM images indicate that the microstructure of (Bi,Pb)-2223 was enhanced by adding BaSnO₃ nanoparticles by filling its pores and voids. The superconducting transition temperature Tc as well as the critical transport current density Jc, estimated from electrical resistivity measurements, are increased up to x = 0.5 wt%, then decreased with further increase in x. Vickers microhardness measurements were carried out at room temperature as a function of applied.

The experimental results were analysed in view of Meyer’s law, Hays and Kendall (HK) approach, elastic/plastic deformation (EPD) and proportional specimen resistance (PSR). All samples exhibit normal indentation size effect (ISE), in addition to that, the analysis shows that the Hays and Kendall model is the most suitable one to describe the load independent microhardness for (BaSnO₃)ₓ(Bi,Pb)-2223 superconducting samples.

1. Introduction

(Bi,Pb)-2223 based compound appears to be the most effective candidate for power and technological applications because of its high superconducting transition temperature (Tc ≈ 110 K), atmospheric stability and its high current and magnetic carrying capacity [1,3]. On the other hand, the major limitations of (Bi,Pb)-2223 phase are related to its ceramic nature, quite brittle, anisotropic and difficult to produce as a single phase [1]. The addition of various nanoparticles into (Bi,Pb)-2223 phase improved its physical properties such as microhardness, elastic modulus, yield strength and fracture toughness [4-6].

In this work, the effect of (BaSnO₃) nanoparticles addition with different weight percentages on the structural and mechanical properties of (Bi,Pb)-2223 is investigated.

2. Experimental Techniques

Co-precipitation method was used to synthesis BaSnO₃ nanoparticles. Stoichiometric amounts of BaCl₂•2H₂O and SnCl₄•5H₂O were dissolved in 50 mL of distilled water. Simultaneously, the solution of BaCl₂•2H₂O was heated and stirred till the temperature reached 75°C. A solution of NaOH is added to get pH=13, followed by the addition of SnCl₄•5H₂O. The mixture was then carried by a simultaneous heating and stirring until the temperature reached 95°C for 1h. The solution was washed with distilled water, after aged overnight. The resultant filtrate of BaSnO₃ was dried at 100°C for 24 h. The substrate was finally calcinated at 900°C for 2 h. Superconducting samples of (BaSnO₃)ₓ(Bi₁.6Pb₀.4)Sr₂Ca₂Cu₃O₁₀₋δ, 0.0% ≤ x ≤ 1.5%, were prepared by solid-state reaction technique as reported by Rahal et al. [5]. Resistivity-temperature and transport current density data were obtained by using four probe DC method. Vickers
microhardness $H_v$ is defined as the ratio of an applied load $F$ in (N) to the pyramidal contact area $d^2$ in ($\mu m^2$) of indentation defined by:

$$H_v = 1854.4 \frac{F}{d^2} \quad (1)$$

Vickers microhardness of the samples were measured in atmospheric air at room temperature using a digital microhardness tester (MHVD-1000 IS) with a pyramidal indenter and loads ranging between 0.49N and 9.8 N.

3. Results and Discussion:

XRD patterns for (BaSnO$_3$)$_x$(Bi,Pb)-2223 superconducting samples indicate that all the major peaks belong to the tetragonal indices of (Bi,Pb)-2223 with a space group $P_{4/mmm}$ where the minor peaks correspond to (Bi,Pb)-2212 and Ca$_2$PbO$_4$ phase.

The calculated lattice parameters, $a = 5.394 \pm 0.002 \ A$ and $c = 37.14 \pm 0.04 \ A$, do not show a significant change by adding BaSnO$_3$ nanoparticles. Moreover, the volume fraction of (Bi,Pb)-2223 is increased with increasing BaSnO$_3$ addition from 93.42% to 95.01% for $x=0.5$ wt%. SEM micrographs showed an enhancement in connection between the superconducting grains in the form of platelets which are also found by adding BaSnO$_3$ nanoparticles, indicating that nanoparticles do not participate in the crystal structure, they are sited between the superconducting grains [6-8].

The temperature dependence of the electrical resistivity for (BaSnO$_3$)$_x$(Bi,Pb)-2223 samples is shown in Figure 1a. The dc electrical resistivity measurements show a well-defined metallic behavior and superconducting transitions for all samples. The superconducting transition temperature is enhanced by increasing the addition of BaSnO$_3$ nanoparticles up to 0.5 wt% as well as the critical current density in Figure 1(b). This result is consistent with the behavior of the volume fraction and grain connectivity versus $x$.

![Figure 1](image_url)

**Figure 1.** a. Variation of electrical resistivity versus temperature. b. Variation of critical current density $J_c$ versus the $x$ addition of (BaSnO$_3$) nanoparticles for (BaSnO$_3$)$_x$(Bi,Pb)-2223 samples.

The room temperature Vickers microhardness $H_v$ are calculated according to Equation (1) and plotted as function of the applied load in Figure 2. It is obvious that $H_v$ is decreased non-linearly as the applied load is increased up to 3 N, beyond which the curves tend to saturate. This non-linear behavior, knowing as indentation size effect (ISE) can be explained qualitatively on the basis of penetration depth of the sample [5,9,10]. It is also observed that $H_v$ increases with increasing BaSnO$_3$ nanoparticles addition from 0.0 to 0.05 wt%. This improvement can be attributed to the reduction of porosity, enhancement of resistance to crack propagation among the grains and the increase in grain connectivity.

The normal ISE behaviour could be explained according to Meyer’s law which represents the relation between the load ($F$) and the indentation diagonal length ($d$):

$$F = Ad^n \quad (2)$$
The exponent $n$ is the Meyer’s index which is less than 2 for normal ISE and greater than 2 for reverse ISE [9,10]. It is noted that the values of $n$ (1.1 ≤ $n$ ≤ 1.4) confirm the normal ISE behavior for all prepared samples. Several models were applied in order to estimate the true microhardness value. Hays and Kendall model revealed the existence of a minimum applied test load $F_{HK}$ to initiate plastic deformation from which Hays and Kendall load-independent microhardness $R_H$ can be estimated [11]:

$$F = W_{HK} + A_1 d^2$$

$$R_H = 1854.4 \times \frac{F - W_{HK}}{d^2}$$

(3)

The Elastic/Plastic model assumed that the indentation contains an elastic portion, so to estimate the independent elastic/plastic microhardness $R_{EPD}$, an elastic component ($d_0$) should be added to the measured semi-diagonal plastic indentation [10-12]:

$$F = A_2 (d + d_0)^2$$

$$R_{EPD} = 1854.4 \times \frac{F}{(d + d_0)^2}$$

(4)

Moreover, the proportional specimen resistance model assumed that during an indentation process, the external work applied by the indenter is changed into surface energy component ($\alpha_s$) and strain energy component ($\beta$). Thus, PSR load independent microhardness $R_{PSR}$ can be estimated [13]:

$$\frac{F}{d} = \alpha_s + \beta d$$

$$R_{PSR} = 1854.4 \times \frac{\alpha_s d + \beta d^2}{d^2}$$

(5)

Figures 4(a-c) display the variation of load variation with the indentation according to various models. The fitting parameters $A_1, W_{HK}, A_2, d_0, \alpha_s$ and $\beta$ are listed in Table 1 versus $x$. It is clear that $W_{HK}$ is positive indicating that the applied load is sufficient to create both elastic and plastic deformation. Also, the positive values of $d_0$ also indicate that the elastic deformation is observed along with the plastic deformation so elastic relaxation is present for all the samples. However, the increase in the value of surface energy $\alpha_s$ with increasing $x$ up to 0.05% is due to dissipation of the energy from cracks at the

**Table 1. Fitting parameters of HK, EPR and PSR for (BaSnO₃)$_x$(Bi,Pb)-2223 samples.**

| $x$ (wt%) | $H_v$ (GPa) | $A_1 \times 10^{-4}$ (GPa) | $W_{HK}$ (GPa) | $A_2 \times 10^{-4}$ (GPa) | $d_0$ (µm) | $H_{EPD}$ (GPa) | $\alpha_s \times 10^{-2}$ | $\beta \times 10^{-4}$ | $H_{PSR}$ (GPa) |
|-----------|-------------|---------------------|--------------|---------------------|----------|----------------|-----------------|-----------------|-------------|
| 0.00      | 0.277       | 1.61                | 0.987        | 0.257               | 1.19     | 52.3           | 0.219           | 1.94            | 0.91         |
| 0.05      | 0.894       | 4.38                | 0.745        | 0.891               | 3.39     | 24.78          | 0.629           | 2.43            | 3.1          |
| 0.25      | 0.146       | 0.797               | 1.09         | 0.146               | 0.578    | 80.67          | 0.106           | 1.59            | 0.42         |
| 0.75      | 0.085       | 0.391               | 1.17         | 0.081               | 0.25     | 141.1          | 0.046           | 1.35            | 0.19         |
| 1.50      | 0.054       | 0.271               | 0.872        | 0.052               | 0.185    | 137.9          | 0.034           | 0.96            | 0.12         |
interfaces [14]. By comparing the data, the experimental microhardness values are very close to those obtained from HK model with deviation of about 7% which is the best fitting model. On the other hand, the values of $r_3$ obtained from EPD and PSR models are much lower than the experimental values with deviations 21% and 40%, respectively.

4. Conclusion:
Series of (BaSnO$_3$)$_x$(Bi,Pb)-2223 samples were successfully prepared via conventional solid-state reaction technique. XRD shows that the tetragonal structure and the lattice parameters of (Bi,Pb)-2223 were not affected by the addition of nanosized (BaSnO$_3$). The grain connectivity was enhanced and the voids were filled up with (BaSnO$_3$) as the micrographs of SEM indicate. Vickers microhardness was measured and analysed using Meyer’s law, HK approach, EPD and PSR models. The calculated values of $H_P$ by the HK approach were more suitable than the other models, where $H_P$ increased up to $x = 0.05\%$, and then it decreased for further increase in $x$.

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