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ORIGINAL PAPER

Mechanical Properties of BSCCO Superconductor by Oliver–Pharr Method and Work of Indentation Approach

**Uğur Kölemen · Orhan Uzun · Cem Emeksiz ·
Fikret Yılmaz · Atilla Coşkun · Ahmet Ekicibil ·
Bekir Özçelik**

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Abstract Polycrystalline superconducting Bi–Pb–Gd–Sr–Ca–Cu–O bulk samples with nominal composition $Bi_{1.7}Pb_{0.3-x}Gd_xSr_2Ca_3Cu_4O_{12+y}$ ($x = 0.00, 0.01, 0.05$) were produced by the melt quenching method. The mechanical properties of the samples were characterized by depth sensing indentation technique under different peak loads ranging from 200 to 1800 mN. The experimental data were comparatively analyzed by the Oliver–Pharr method and a work of indentation approach. It was found that the work of indentation approach gave more reliable results because of the reducing pile-up effect. The results implied that both microhardness and reduced elastic modulus increased with increasing Gd substitution.

U. Kölemen · F. Yılmaz
Department of Physics, Arts and Sciences Faculty,
Gaziosmanpaşa University, Tokat, Turkey

O. Uzun
Deparment of Metallurgical Engineering, Faculty of Engineering,
Bülent Ecevit University, 67100, Zonguldak, Turkey

C. Emeksiz
Department of Technical Programmes, Turhal Vocational College,
Gaziosmanpaşa University, Günesli District, Turhal Campus,
60300, Tokat, Turkey

A. Ekicibil · B. Özçelik (✉)
Department of Physics, Arts and Sciences Faculty, Çukurova
University, Adana, Turkey
e-mail: ozcelik@cu.edu.tr

A. Coşkun
Department of Physics, Faculty of Sciences and Letters, Mugla
University, Mugla, Turkey

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1 Introduction

In current research on superconducting ceramics, the Bi–Pb–Sr–Ca–Cu–O (BSCCO) superconducting compound has received much attention because of its high critical temperature, lower hardness value compared to YBaCuO and MgB_2 superconductors, and there is a lesser tendency to react with water or carbon dioxide and no need for special annealing in oxygen [1, 2]. In addition, BSCCO superconductor is suitable than the other oxide superconductor (YBaCuO and MgB_2) manufacturing of wire and tape [3]. Superconducting tape is a new promising product for high current applications such as power transmission cables, high-efficiency generators, transformers and motors, current fault limiters and superconducting magnetic technology, e.g. MRI scanners, levitated train and superconducting magnetic energy storage systems (SMES) [3]. For most applications, superconducting materials have to be machined in the shape of wires and tapes, and subjected to large mechanical stresses in making coils and Lorentz force due to high magnetic fields. Under high stresses, the generation of small cracks at high currents will cause serious damage or destruction of the coil. Besides, magneto elastic effects associated with flux pinning induce internal macrostresses. These effects often cause fatal cracking of Melt-Grown (MG) bulks as they become magnetized [4]. Therefore, the mechanical properties such as strength, fracture toughness, hardness, ductility, elastic modulus, and creep as well as superconducting properties, are very important for industrial application of high temperature oxide superconductors.

In the open literature, studies on investigation of the mechanical properties of the superconducting materials have been carried out by using Conventional (or static) Microindentation (CMI) technique so far [5]. While the mechanical properties by this technique have been investigated extensively, there are limited studies on the mechanical characterization of the superconductors by using Depth Sensing Indentation (DSI) technique [6]. Due to some technical disadvantages reported in many studies, the CMI has not been most commonly used. The determination of conventional microhardness value is the residual impression left behind upon unloading. Accordingly, an insignificant measurement error (especially in small indentation imprints) can lead to higher relative error. Contrary to the CMI, in recent decades the development of depth sensing indentation devices has enabled researchers to determine the most common mechanical parameters namely hardness and elastic modulus easier and more reliable. The DSI offers great advantages over the CMI in two aspects. (i) Apart from microhardness (or micro strength), it can also provide well-defined mechanical parameters such as reduced elastic modulus of interfacial zone. (ii) As load and penetration depth of an indentation are continuously monitored, optical observation and measurement of diagonal length of the indent/impression, which can be difficult and subjected to inaccuracy, is no longer required. Therefore, the mechanical characterization of materials using the depth sensing indentation technique has gained rapid acceptance in recent years.

In the present investigation, the melt-quenching technique has been adopted to synthesize BSCCO superconductors. The aim of this work is to investigate the effect of Gd substitution on mechanical properties of the bulk BSCCO ceramic materials.

2 Experimental

Bi–Pb–Gd–Sr–Ca–Cu–O bulk samples with nominal composition $Bi_{1.7}Pb_{0.3-x}Gd_xSr_2Ca_3Cu_4O_{12+y}$ ($x = 0.00, 0.01, 0.05$) were prepared by the melt-quenching method, the samples are named as X_0, X_1, and X_2, respectively. The appropriate amounts of powdered Bi_2O_3, PbO, $SrCO_3$, CaO, and CuO in the stoichiometric ratios of $Bi_{1.7}Pb_{0.3-x}Gd_xSr_2Ca_3Cu_4O_{12+y}$ were well mixed by milling and calcined at 840 °C for 10 h in air. The calcined powders were held in platinum crucibles at 1200 °C until the samples were completely melted. The melts were poured onto a pre-cooled copper plate and pressed quickly by another cooper plate to obtain plate like Gd free reference specimen. Gd_2O_3 powder was later added to the reference material and the mixture was ground for about one hour; the resulting powder was then pressed into pellets of 10 mm diameter by applying a pressure of 5 tons. Finally, the precursor materials produced by melt-quenching were annealed at 192 h in air to achieve good superconductivity with relatively high T_c [7].

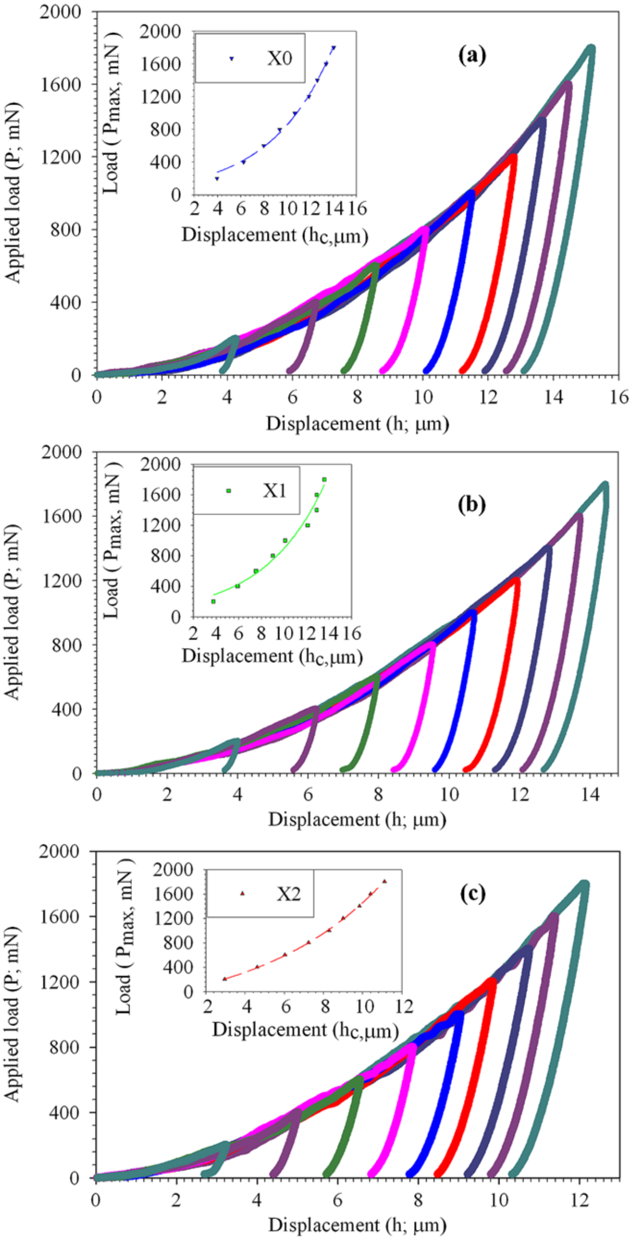

Fig. 1 (**a**), (**b**), and (**c**) illustrate our load-penetration depth measurements for samples X_0, X_1, and X_2 obtained by microindentation for applied ranging from 200 to 1800 mN, respectively. The *inset* depicts variation of penetration depth as a function of load room temperature

The samples were cold mounted and prepared in the normal manner, ground on 2400 grit and then polished on 0.5 and 0.1 µm alumina lap wheels, respectively. A Vickers pyramidal indenter was used in a dynamic ultra microhardness tester (Shimadzu, DUH-W201S). Depth sensing indentation experiments were performed in the loads ranging from 200 to 1800 mN. Load-penetration depth curves contained nine loading-unloading cycles. The loading rate was 23.53 mN/s. Three indentation tests were conducted on sample surface to increase reliability of experimental results.

3 Results and Discussion

The loading-unloading curves of the BSCCO specimens (X_0, X_1, and X_2) obtained by increasing loads are given in Fig. 1a, b, and c. It is clearly seen from the figures that the samples show elasto-plastic behavior at room temperature. However, this characteristic cannot entirely clarify that of whether the specimens have brittle or ductile properties. On the other hand, loading curves under different peak loads can be accurately fitted by one curve due to their overlapping characters. The unloading curves show similar behavior if they are shifted according to their final depths. Besides, the inset plots in Fig. 1 illustrate that the contact depths (h_c) increase with increasing applied load (P_{max}). Therefore, it can be suggested that the BSCCO superconductors have similar elastic and plastic deformation mechanism for our experimental load range.

In addition, the ratio between final penetration depth, h_f (the depth of indentation after unloading) and the depth of indentation at maximum load, h_{max}, is another interesting experimentally measurable parameters that can be used to identify the expected indentation behavior of a given material [8]. Because the conical or pyramidal indenters have a self-similar geometry, h_f/h_m, which naturally ranges from 0 to 1, does not depend on the depth of indentation. The lower (0) and upper (1) limits signify fully elastic and rigid-plastic behavior of materials, respectively. Bolshakov et al. [8] reported that the h_f/h_m parameter is a useful indicator of pile-up or sink-in deformation characteristics around the indenter. Pile-up is significant only when h_f/h_m is higher than the critical value (0.70). On the other hand, sink-in behavior is dominant factor when the h_f/h_m is less than the above mentioned critical value, and material shows work hardening deformation behavior. The average h_f/h_m values were found to be 0.87, 0.88 and 0.86 for the samples X_0, X_1, and X_2, respectively, which are higher than the reported critical value 0.70. These results imply the existence of pile-up behavior around indenter.

P–h curves were analyzed by using Oliver–Pharr and Work of Indentation Approach in order to calculate the hardness and elastic modulus of the samples. Although the Oliver–Pharr method is the most adopted procedure because of its easy application [9], the work of indentation approach is considered as the most effective model to reduce the errors in many cases where pile-up or sink-in is dominant [10]. The calculated dynamic microhardness (H_{OP}) and reduced modulus values (Er_{OP}) at different applied loads by Oliver–Pharr model are given in Fig. 2 for each sample. The curves in the graph show that H_{OP} and Er_{OP} values decreased non-linearly as the applied load increased until, at about 1200 mN, a threshold above which H_{OP} and Er_{OP} values tend to attain saturation. This type of non-linear is called indentation size effect (ISE) and has been reported different of material and in the literature [11]. It is obvious from Fig. 2 that H_{OP} and Er_{OP} values increase with increasing Gd amount in the composition. Hardness and reduced modulus are a property that can be described with regard to resistance against to dislocation motion and bonding force between the atoms. The general contribution to the resistance is mainly of two types: (i) the intrinsic resistance and (ii) the resistance due to imperfections. Changes in the hardness and reduced modulus of the BSCCO ceramic material depending on doping or substitution concentration have been reported in the literature [12]. In our case, increase in the hardness and reduced modulus of the BSCOO samples with Gd substitution may be due to both pinning of dislocations at the impurity sites and other defects caused by the presence of impurity atoms in the crystal, and also to variation in the magnitude of the bond forces in the crystal containing impurities.

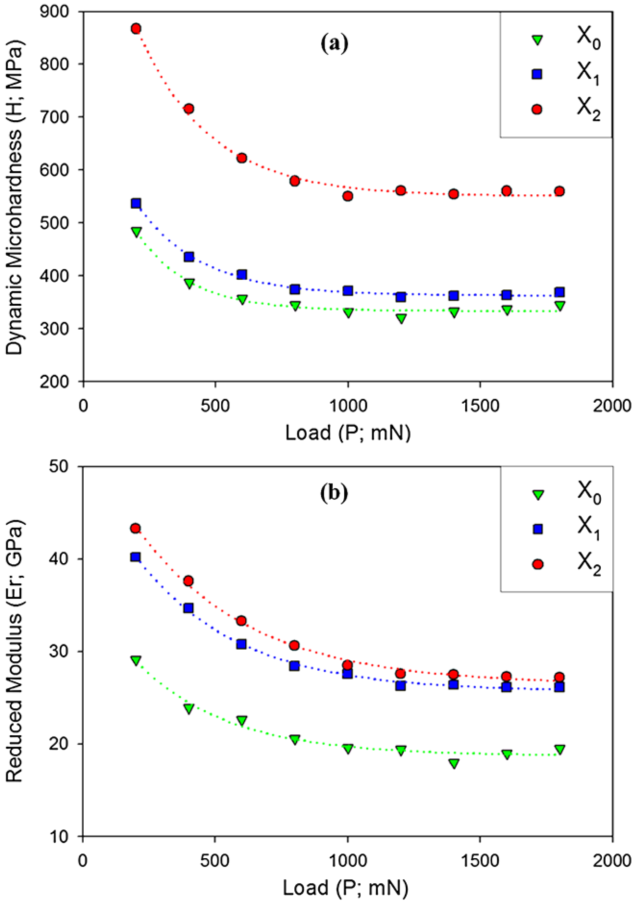

Fig. 2 (**a**) Dynamic microindentation variation as a function of maximum applied test load obtained by Oliver–Pharr model for samples X_0, X_1, and X_2. (**b**) The variation of the reduced modulus with the peak load for X_0, X_1, and X_2 samples

Figure 3 shows the results of Oliver and Pharr calculations in the form of dynamic microhardness, H_{OP}, versus the

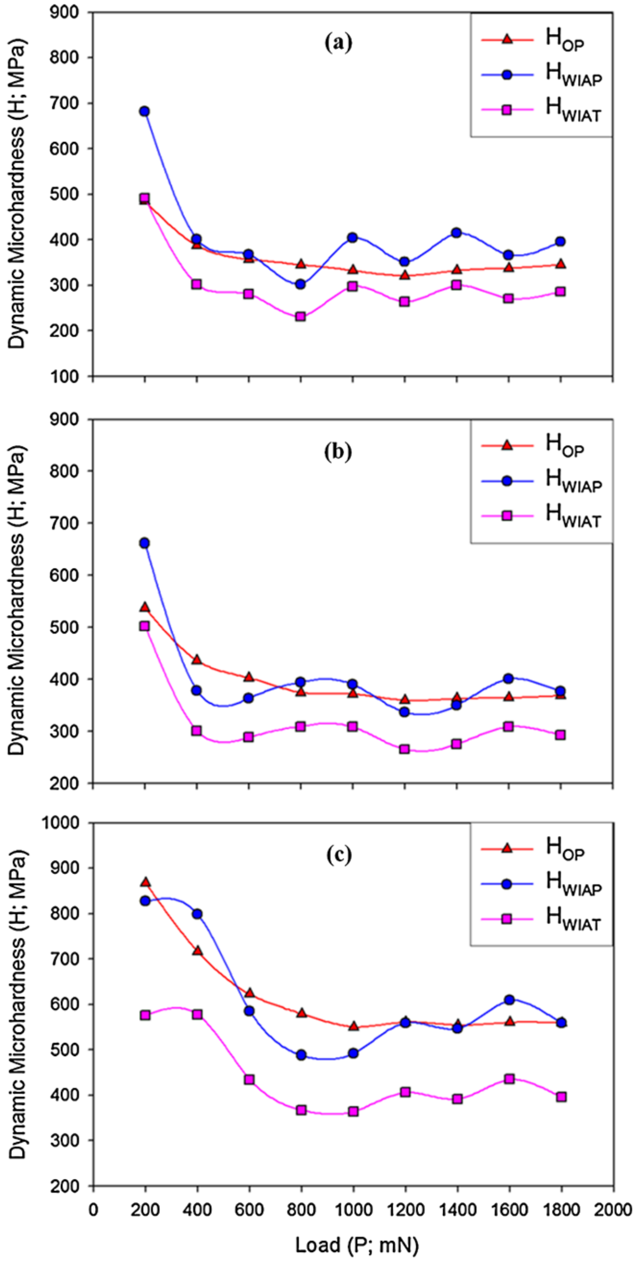

Fig. 3 Dynamic microhardness values obtained by Oliver–Pharr and Work of Indentation Approach for the samples (**a**) X_0 (**b**) X_1, and (**c**) X_2

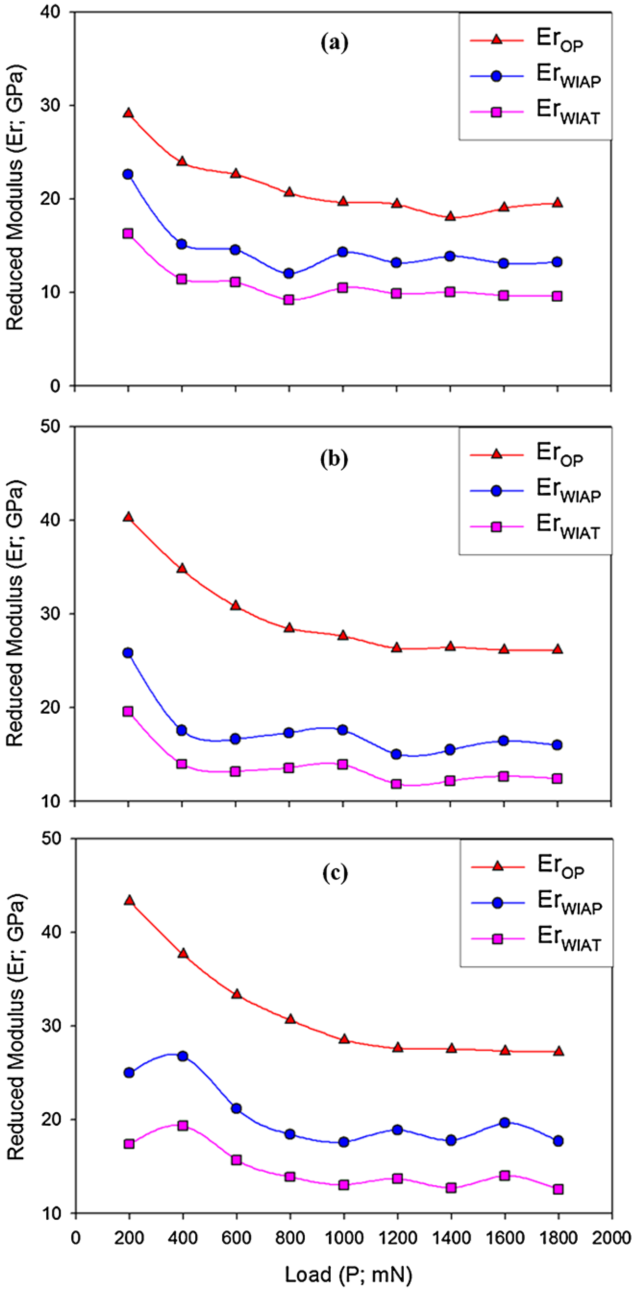

Fig. 4 Reduced modulus values obtained by Oliver–Pharr and Work of Indentation Approach for the samples (**a**) X_0 (**b**) X_1, and (**c**) X_2

applied load, P. Alongside these values, H_{WIAP} and H_{WIAT} calculated from the plastic and total work, respectively, are also included in Fig 3. It is clear that the total work definition underestimates the hardness. This effect is likely to be caused by large elastic contribution to the total work of indentation. The hardness calculated from the plastic work seems to correlate very well with the Oliver and Pharr method [13]. However, recent works [14] suggest that harder materials do not experience considerable elastic deformation, while pile-up is seen to cause a problem. Due to the pile-up, the plastic contact depth is greatly underestimated in the Oliver–Pharr method, therefore producing overestimated hardness values. The work of indentation approach is not affected by the pile-up, as calculations are performed directly from the analysis of energy, without the need for the estimation of penetration depths, diagonals areas or volumes.

Figure 4 shows E_{ROP}, E_{RWIAP}, and E_{RWIAT} values determined by two models plotted versus the applied loads. The E_{RWIAP} and E_{RWIAT} values obtained by work of indentation approach are lower than E_{ROP} values. Inversely, the values E_{ROP} calculated by Oliver–Pharr method are the highest due to underestimating of the true contact area. As a result, it may be suggested that estimating of reduced elastic modulus by plastic work is more convenient.

4 Conclusions

Based on the results of mechanical analysis of BSCCO ceramic superconductors, the following conclusions can be drawn:

1. Microhardness and reduced elastic modulus values of BSCOO increased with Gd substitution.
2. The obtained microhardness and reduced elastic modulus by using total work were found to be the lowest owing to the elastic contribution.
3. The microhardness and reduced elastic modulus calculated by the work of indentation approach are more accurate than that calculated by the Oliver–Pharr method due to reducing of pile-up error.
4. The calculated $h_\mathrm{f}/h_\mathrm{max}$ values suggested that the pile-up effect is dominant in our samples.

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