



# Work of indentation approach for investigation of mechanical properties of YBCO bulk superconductor at cryogenic temperatures

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## Abstract

In this study, depth sensing Vickers microindentation test was carried out for YBCO polycrystalline superconductor with the temperature of ranging from 50 to 293 K at the load of 4.9 N. The applied load ( $P$ )-displacement ( $h$ ) curves and the energy data (the absolute, the total, the plastic, and the elastic energy) were analysed to evaluate the mechanical properties such as hardness and elastic modulus values. The values were calculated by the Oliver–Pharr method and the work of indentation approach. A comparison between the results obtained by the aforementioned two methods was made. The work of indentation approach performed well because of the fact that the error due to the pile-up was reduced. In addition, it was found that the hardness and elastic modulus increased with decreasing temperature. Some specific material constants describing the properties of materials were also suggested as total energy constant ( $v_T$ ), plastic energy constant ( $v_P$ ), elastic energy constant ( $v_E$ ), elastic-total energy constant ( $v_{ET}$ ), plastic-total energy constant ( $v_{PT}$ ), and elastic–plastic energy constant ( $v_{EP}$ ).

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## 1. Introduction

It is well known that high- $T_c$  bulk superconductors exhibit high critical current density and high-trapped magnetic field at cryogenic temperature. However, practical applications of these

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superconducting materials are often limited by their poor mechanical performance especially at cryogenic temperature. Hence, mechanical properties such as microhardness, elastic modulus, creep behavior, etc. are crucial for industrial application of high- $T_c$  oxide superconductors.

Depth sensing indentation, with its potential versatility, is a powerful experimental technique for determination of the mechanical properties of high-temperature oxide superconductors [1–4]. One of the most commonly used methods for estimating the mechanical properties of materials is Vickers microhardness test. Hardness is a mechanical parameter, which is strongly related to the structure and composition of solids. Therefore, hardness tests have become more and more important to characterize a material. Many researchers investigated deformation characteristics of different superconductor materials at room temperature (see, for example, [5–8]). Accordingly, the Vickers indentation experiment at cryogenic temperature would also provide valuable information to estimate low temperature deformation characteristic, which is important as considering practical application. However, there are limited investigations at cryogenic temperature compared with room temperature examinations [9,10].

Micromechanical properties are investigated by conventional formula such as the Oliver–Pharr method or the work of indentation approach. The Oliver–Pharr analysis is commonly used to determine hardness and elastic modulus due to easy application procedure. On the other hand, the work of indentation approach is also preferred in literature to reduce the errors due to pile up or sink in behavior of materials in instrumented indentation experiment [11,12]. The work of indentation approach was the first proposed by Stilwell and Tabor [13]. Then, Sakai [14] put forward a relationship between the energy of the hysteresis indentation loop and the hardness. Recently, Attaf [15] has also used the work of indentation approach to suggest some material constants.

In this work, the hardness and elastic modulus of superconducting materials (YBCO) at cryogenic temperatures (from 50 to 293 K) with 4.9 N were calculated by two different aforementioned meth-

ods. In addition, the temperature dependence of hardness and elastic modulus were discussed. Some specific material constant describing the properties of materials were also suggested.

## 2. Experimental

The YBCO sample was prepared by conventional solid-state reaction method. The powders of  $Y_2O_3$ ,  $BaCO_3$ , and  $CuO$  were thoroughly mixed in the appropriate amounts and calcined at 900 °C for 8 h. After calcination, the powders were mixed by a grinding machine for 4 h and then pressed into pellets of 13 mm in diameter and 2 mm in thickness at 375 MPa. These pellets were melted on an  $Al_2O_3$  substrate for a short time using LPG- $O_2$  flame. The molten materials were immediately poured onto a precooled copper plate and pressed with another plate to obtain quenched material. After quenching the YBCO material was crushed the powders and mixed using a mortar machine. Then, the powders were re-pressed into pellets and sintered at 945 °C for 24 h and cooled down to room temperature at the rate of 1 °C/min by oxidation at temperatures between 700 and 250 °C.

Vickers hardness tests were carried out using the test apparatus developed by Iwabuchi et al. [16]. Indentation was conducted on sample surface for three times at each temperature. In the measurements, applied load was 4.9 N at cryogenic temperatures. Indenter was kept 15 s after each loading. The mean loading rate was 6–8 N/s.

## 3. Theoretical consideration

The loading and unloading characteristic of  $P$ – $h$  curve is shown in Fig. 1. According to the work of indentation approach (the energetic aspect), the known and commonly used energy terms, based on the integral of the loading and unloading curves, are namely: (i) the area under the loading curve gives the total energy  $W_T$  done by the loading device during indentation, (ii) the elastic energy  $W_E$  can be deduced from the area under the unloading curve, and (iii) the plastic energy  $W_P$ ,

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