Work of indentation approach to load-penetration depth data in bulk BSCCO superconductor

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Indentations have been carried out on a bulk BSCCO superconductor by using a Vickers indenter. The load-penetration depth curves were analyzed by the work of indentation approach. Some specific material constant describing the properties of materials are calculated as total energy, plastic energy, elastic energy, elastic-total energy, plastic-total energy and elastic-plastic energy constants. The β -material for bulk BSCCO is also determined.

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1. Introduction

It is well known that the mechanical properties such as hardness, elastic modulus, and ductility as well as the critical temperature, critical current and critical magnetic field are important for various industry applications of the high-T_c superconductors (YBCO, BSCCO and MgB_2). Therefore, depth-sensing indentation tests are widely used for the mechanical characterization of the high-T_c superconductors (see for example [1-4]). This method is preferred because relatively small amount of test material are needed and there are no strict requirements for the shape of the sample. The measurements can also be performed without the destruction of the sample. The common feature of this test is that the applied load is registered as a function of indentation depth during both the loading and unloading period. Many methods are developed in the literature to analyze the loading and unloading curves [5-8]. However, these methods may not be reliable when pile up and sink in behaviors occur. The work of indentation approach and the displacement approach to indentation are both applicable to the cases of pile up and sink in and do not require imagining of the residual indents. The displacement approach to indentation is developed by Giannakopoulos and Suresh [9]. They show that two methods (i.e. the work of indentation approach and the displacement approach to indentation) are the equivalence to each other. The work of indentation approach is the first proposed by Stilwell and Tabor [10]. Then, Sakai [11] put forward a relationship between the energy of the hysteresis indentation loop and the hardness. Recently, Attaf [12] has also used the work of indentation approach to suggest some material constants. The β , a constant specific to the tested material, is calculated by using such constant [13].

In his work, we characterized the mechanical properties of BSCCO superconductor at room temperature by depth-sensing indentation tests. Some specific material constants describing the properties of materials are calculated. The β -material for bulk BSCCO is also determined.

2. Experimental

The bulk BSCCO superconductor was prepared by a standard solid-state-reaction method. X-ray diffraction data showed that the sample is mainly made of the high T_c phase 2223. Having obtained a good-quality sample, a Vickers indenter tests (Shimadzu, DUH-W201S) has been carried out. To avoid uncertainties arising from changes in the experimental procedure and to carry reliable comparisons of data for possible correlations, all tests were performed under the same operating conditions. To easily interpret the material behaviour at various depths, the maximum load was changed at regular intervals; 200, 400, 600, 800, 1000, 1200 and 1400 mN. The loading rate was 23. 5 mN/s.

3. Theoretical background

The typical indentation load-penetration depth (*P-h*) curves are depicted in Fig. 1. Considering the work of indentation approach, the known and commonly used energy terms are namely: (i) the total energy, W_T, located under the loading curve, (ii) the elastic energy, W_E, can be deduced from the area under the unloading curve, (iii) the plastic energy, corresponded to the enclosed area by the two curves, is the difference between these, $W_P=W_T-W_E$. In addition, the absolute energy, W_S, put forward by Attaf [12], has proportionately relationships with the other energies. These relations are given by the following formulas [12].

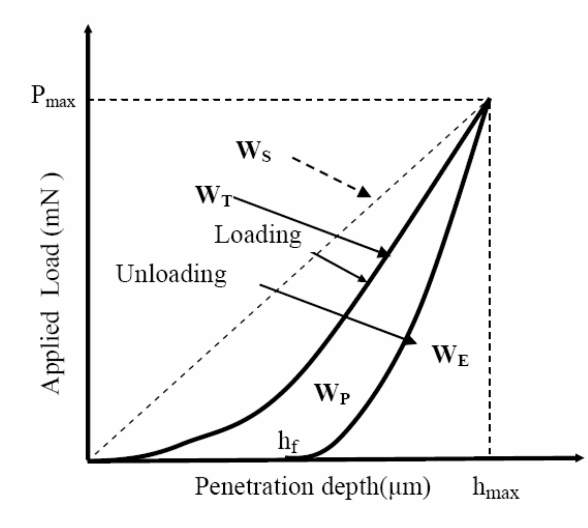

Fig. 1. Typical indentation cycle showing load-unload curves.

Total energy constant:

$$\upsilon_T = \frac{W_S}{W_T} \tag{1a}$$

Plastic energy constant:

$$\upsilon_P = \frac{W_S}{W_P} \tag{1b}$$

Elastic energy constant:

$$\upsilon_E = \frac{W_S}{W_E} \tag{1c}$$

Plastic-total energy constant:

$$\upsilon_{PT} = \frac{W_T}{W_P} \tag{2a}$$

Elastic-total energy constant:

$$\upsilon_{ET} = \frac{W_T}{W_E} \tag{2b}$$

Elastic-plastic energy constant:

$$\upsilon_{EP} = \frac{W_P}{W_E} \tag{2c}$$

These relations can play a crucial role in describing mechanical properties of materials. In addition, a comprehensive set of energy values with identified unique terms are proposed to facilitate further investigations and modeling of the indentation energy concept [13, 14].

4. Results and discussion

Fig. 2 shows a series of load against penetration depth plots recorded at different maximum loads for bulk BSCCO. It is clearly seen that the sample shows elastic-plastic deformations during Vickers indentation.

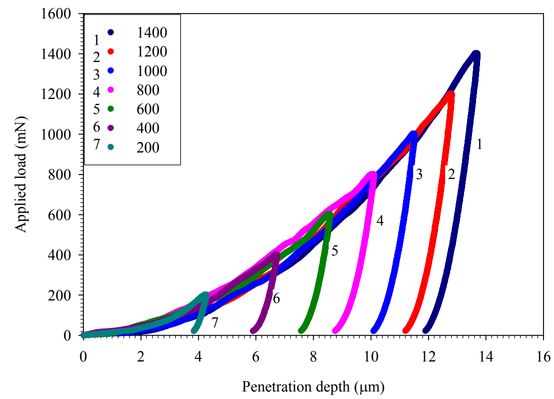

Fig. 2. Load–penetration depth behavior of BSCCO sample.

It is well known that the energy ratios can be used to determine mechanical behaviour and are worth to investigate. The slopes of plots in Fig.3 show the energy constant, ν_i (i=T, P, E, PT, ET and EP). The total energy constant, ν_T, is a proportionately constant specific to the total energy when compared to the absolute energy. This constant for bulk BSCCO is inherent as it does depend on the penetration depth. Other constants (the plastic (ν_P) and elastic (ν_E)) are related to the plastic and elastic character of bulk BSCCO sample, respectively. In addition, correlation between total, plastic and elastic energies are defined by three constants (plastic-total (ν_{PT}), elastic-total (ν_{ET}), elastic-plastic (ν_{EP})) calculated by the same data. All the energy constants are given in Table 1. To show the validation of the energy constant, we used the equations [15]:

$$\frac{\upsilon_P(\upsilon_E - \upsilon_T)}{\upsilon_E \upsilon_T} = 1 \text{ (Computed value: 1.018)} \tag{3a}$$

$$\frac{1}{\upsilon_{ET}} + \frac{1}{\upsilon_{PT}} = 1 \text{ (Computed value: 1.002)} \tag{3b}$$

Table 1. Energetic proportionality constant with corresponding statistical coefficients r^2

Sample	Constant	ν_T	ν_P	ν_E	ν_{PT}	ν_{ET}	ν_{EP}
	Definition	W_S/W_T	W_S/W_P	W_S/W_E	W_T/W_P	W_T/W_E	W_P/W_E
BSCCO	Value	1.490	1.769	10.540	1.167	6.873	5.874
	r^2	0.995	0.993	0.997	0.999	0.991	0.990

Those energy constants are similar to that of the YBCO and MgB_2 sample [1, 2]. The relationships given in Fig.3 show a linear character with regression coefficients (r^2) ranging from 0.990 to 0.999. The W_P/W_T is linked to that of h_f / h_{max}, relating to recovery behavior of material. Accordingly, the ratio of W_T / W_E is proportional to relationship between elastic modulus and hardness of materials *(E/H)*, which is an important parameter to determine the degree of elastic recovery and fracture toughness of materials .

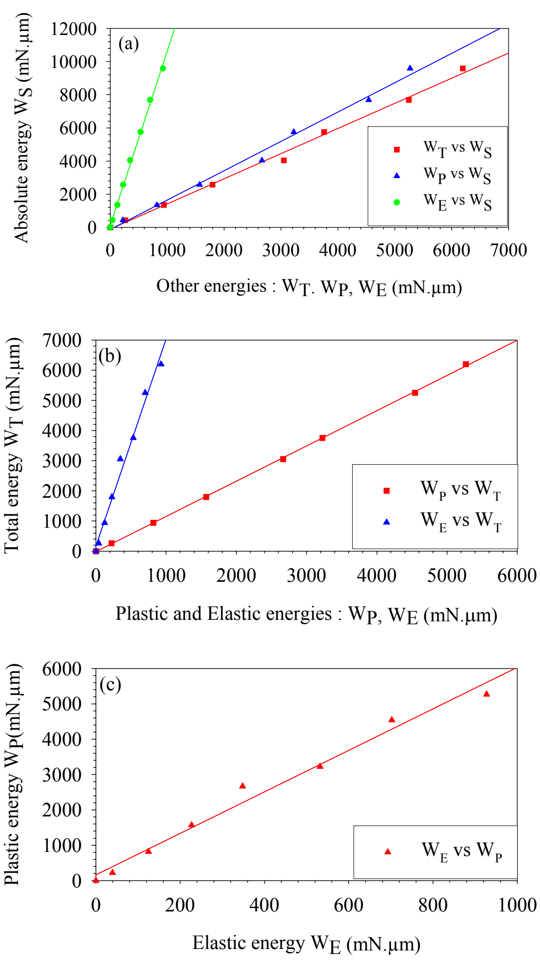

Fig. 3. Correlation between energetic quantities involved in BSCCO indentation with Vickers tip (a) Variation of absolute energy (W_S) versus the other energies. (b) Variation of total energy (W_T) versus elastic and plastic energies. (c) Variation of plastic energy (W_P) versus elastic energy (W_E).

On the other hand, the indentation-induced superelastic (SE) behavior may be characterized by the work recovery ratio measured from indentation load-penetration depth curves. The work recovery ratio, η_w, is defined as [16];

$$\eta_w = \frac{W_E}{W_T} \tag{4}$$

Fig. 4 shows the work recovery ratio of the Vickers indentation at various depths. It is clearly seen that the work recovery ratios are depth independent. These rations are similar to that of the MgB_2 sample [2].

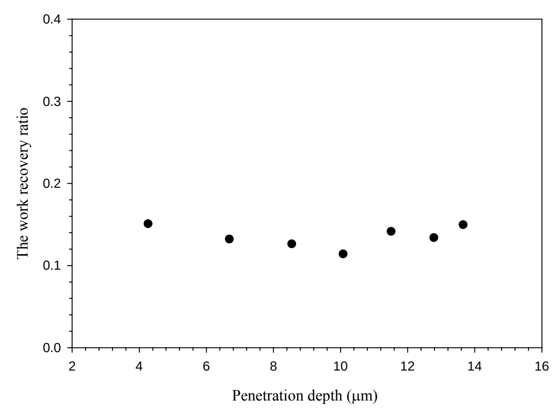

Fig. 4. Relationship between the work recovery ratio and penetration depth for BSCCO sample.

The mechanical behavior of indented materials is generally described by

$$P = Ch^m \tag{5}$$

where C and m are fitting constants that can be derived from the experimental data, with m ranging between 1.5 and 2 [17]. Attaf [13] proposed that β is a constant specific to the tested material. It was also proved that the β-material rules all the energy nanomechanical characterises [14, 15]. The β-material is calculated by:

$$\beta = \frac{1}{m} \tag{6}$$

To obtain C and m values, the load-penetration depth curves in Fig.2 were fitted using Eq (5). The best fit values of C and m are 7.806 and 1.982, respectively. Using the value of m, we obtain: β=0.5045. In order to show the validation of the β -value, we used by the equation [14]:

$$\frac{1}{\upsilon_T} = \sum_{i=1}^{n=\infty} \frac{[2(n-i)+1][i^{\frac{1}{\beta}} - (i-1)^{\frac{1}{\beta}}]}{n^{\frac{(1+\beta)}{\beta}}} \quad (7)$$

where n is a partition of the maximum depth of penetration ($h_m = n\Delta h$). The greater is n the more accurate is the computation of the ratio $1/\nu_T$. Usually n close to 100 suffices to ensure a primary convergence towards a characteristic curve, then a conveniently high value of i for the series terms defines a single point in that curve for a given material [15]. With n=90, we obtain: ν_T=1.4909. This value equals to ν_T in the Table 1.

5. Conclusions

A bulk BSCCO superconductor has been investigated by using a dynamic ultra-microindentation experimental technique. The P-h curves were analyzed by the work of indentation approach. In addition, some specific material constant describing the properties of materials are calculated as total energy, plastic energy, elastic energy, elastic-total energy, plastic-total energy and elastic-plastic energy constants. The β -material for bulk BSCCO is also calculated.

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