Nanoindentation study of bulk MgB_2 crystal

N. GÜÇLÜ*, O. UZUN, U. KÖLEMEN
Department of Physics, Faculty of Science and Arts, Gaziosmanpaşa University, 60240, Tokat, Turkey

Nanoindentation tests were carried out on a bulk MgB_2 superconductor with the temperature of ranging from 297 K and 673 K at 2000mN. The load-penetration depth curve was analyzed by Oliver and Pharr method to calculate hardness. It was found that the hardness increases with decreasing temperature. The activation energy and the energy barrier at 0 K are also calculated.

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1. Introduction

Since the discovery of superconductivity in MgB_2 [1], there has been considerable interest in the characterization and optimization of superconducting properties (see, for example [2-4]). These studies showed relatively high transition temperature, high transport critical current density, simple crystal structure and large coherence length for MgB_2. On the other hand, the mechanical properties such as hardness, elastic modulus, fracture toughness, the activation energy, and the energy barrier at 0 K etc. are as important as superconducting properties for industrial applications of superconducting materials. The MgB_2 has higher mechanical strength than the other HTc ceramic superconductor materials [5]. Thus, different sample forms such as bulk [6] and wires [7] are characterized by using indentation methods. We obtained that, hardness and elastic modulus are higher than that of the other superconductor materials. It can also be seen that there is a positive relationship between hardness and the critical current density of MgB_2 wires [7]. In addition, some specific material constant describing the properties of materials were also calculated as total energy, plastic energy, elastic energy, elastic-total energy, plastic-total energy and elastic-plastic energy constants [8]. Besides, the β -material for bulk MgB_2 is calculated [9].

Vickers microhardness test is an important tool to investigate the mechanical properties of materials such as elastic modulus and hardness. This method is preferred because relatively small amounts of testing material are needed and there are no strict requirements for the shape of the sample. Moreover, the measurements can be performed without the destruction of the sample. The common feature of these tests is that the applied load is registered as a function of indentation depth during both the loading and the unloading period. Material hardness is obtained by dividing the maximum applied load by the residual contact area. Classical hardness experiments required the imaging of the indents to measure the contact area. The two methods were developed in the literature to calculate the contact area directly from the load-displacement curves [10, 11]. The most commonly used method, developed by Oliver and Pharr [11], calculates the contact area from the contact depth, determined from the load-displacement curves.

In this paper, Nanoindentation tests were carried out on a bulk MgB_2 superconductor with the temperature of ranging from 297 K and 673 K at the load of 2000mN. The load-penetration depth curves were analyzed by Oliver and Pharr method to obtain the hardness-temperature curves. The activation energy and the energy barrier at 0 K are also calculated.

2. Experimental

The sample was prepared from the MgB_2 powder (Alfa Aesar Co., 99 % purity). A 1GPa pressure is applied to prepare test specimen. Then, pellets are placed in an α-alumina boat and sintered at 950 ^oC for 2h under Ar atmosphere. In order to compensate evaporation of Mg from the sample, extra pure Mg powder of 5 mg is used [6]. The test specimen is mechanically polished by alumina powders (0.1 and 0.05 μm) and cleaned with acetone. A Vickers indenter was used in a UMT high temperature microhardness tester (CETR). Indentation was carried out on a bulk MgB_2 superconductor with the temperature of ranging from 297 K and 673 K at 2000mN.

3. Theoretical background

The typical indentation load-penetration depth (P-h) curves are depicted in Fig. 1.

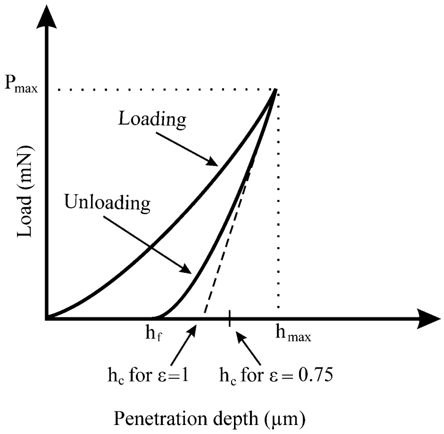

Fig. 1. Typical indentation cycles showing load-unload curves.

The important quantities are the peak load (P_{max}), the maximum depth (h_{max}), the final or residual depth after unloading (h_f) and, the contact depth (h_c). The contact depth is determined by the geometry of indenter. h_c is calculated by the following equation;

$$h_C = h_{\max} - \varepsilon(h_{\max} - h_f) \tag{1}$$

where ε is 0.75 for the Vickers indenter [12].

The hardness is obtained from following equations [11]:

$$H = \frac{P_{\max}}{26.43 h_c^2} \tag{2}$$

4. Results and discussion

Fig. 2 exhibits load-penetration depths curve of MgB_2 sample at various temperatures. It is clearly seen that the penetration depth increases with increasing temperature for the same applied load. This observation leads to hardness decreasing with increasing temperature.

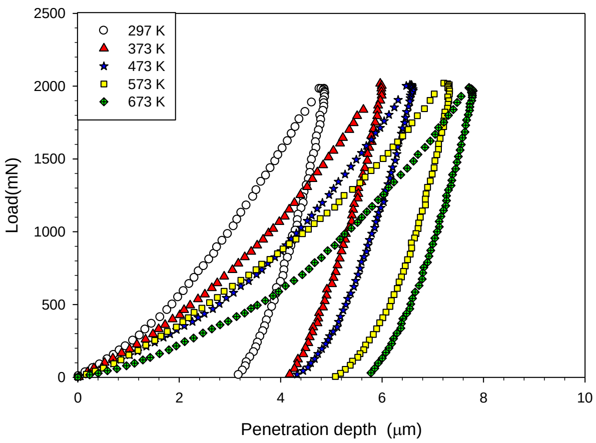

Fig. 2. Load-penetration depth behavior of MgB_2 sample on which 2000 mN load applied at various temperature.

Fig. 3 shows hardness by calculated Oliver and Pharr method (Esq.(2))-temperature curves. The hardness increases with decreasing temperature. Since the hardness is regarded as the plastic deformation resistance, the increase in hardness with decreasing temperature means the increase in the plastic deformation resistance. If such plastic deformation is dominated by the dislocation movement, the inverse of the hardness could be considered as the index representing the mobility of the dislocation or velocity of it.

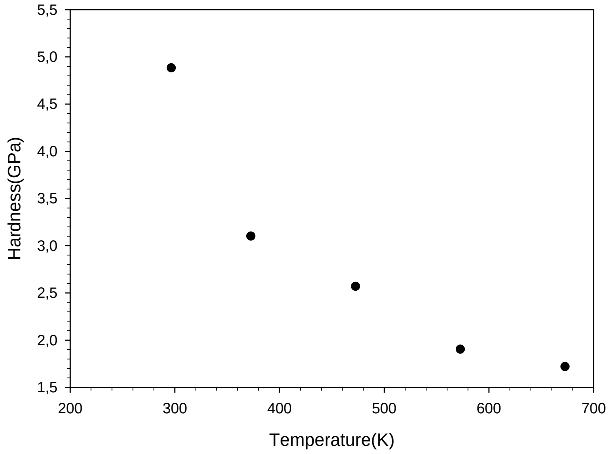

Fig. 3. Temperature dependence of hardness.

Therefore, the hardness could be attributed to thermal activation process in the Arrhenius equation as follows [13]

$$H = H_0 \exp(-Q / RT) \tag{3}$$

where H_0, Q, R, T is the hardness at 0 K or Arrhenius constant , activation energy, Boltzmann's constant, temperature, respectively. Fig. 4 showed the relationship between log H and 1/T for MgB_2 sample. By using plot of log H vs. 1/T, thermal activation energy is found to be 0.0473 eV**.**

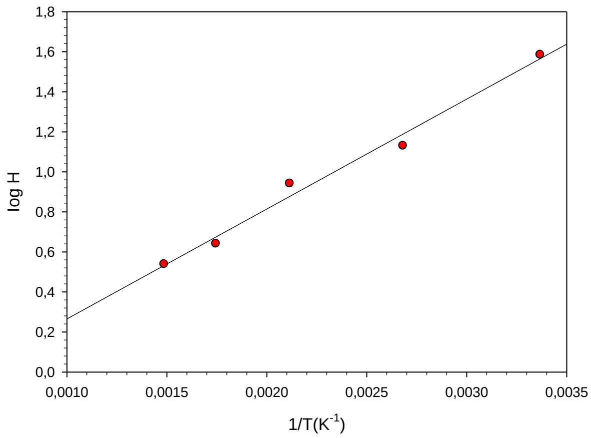

Fig. 4. The relationships between log H and 1/T for MgB_2 sample.

Another interesting parameter that can be obtained form these measurements is the energy barrier for plastic flow at 0 K. Gilman [14] proposed

$$\frac{H}{H_0} = 1 - \left[\frac{2R\theta}{U} \left[\coth\left(\frac{\theta}{T}\right) - 1 \right] \right] \tag{4}$$

where U, θ is the energy barrier and the Debye temperature, respectively. Fig. 5 exhibited the relationship between H and $\coth(\theta/T)$ for MgB_2 sample. By using plot of H v.s $\coth(\theta/T)$ and θ=884 for MgB_2 sample [15], the energy barrier at 0 K is 0.0047 eV.

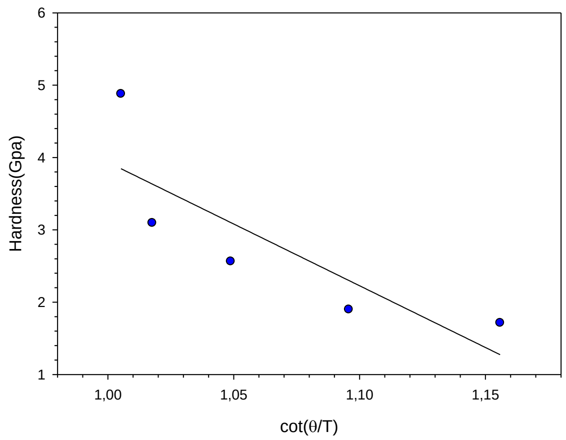

Fig. 5. The relationship between H and $\coth(\theta/T)$ *for* MgB_2 *sample.*

5. Conclusions

Nanoindentation tests were carried out on a bulk MgB_2 superconductor with the temperature of ranging from 297 K and 673 K at the load of 2000mN. The load-penetration depth curve was analyzed by Oliver and Pharr method. It was found that hardness decreases with increasing temperature. The thermal activation energy and energy barrier at 0 K are calculated.

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*Corresponding author: guclu06@hotmail.com