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Mechanical characterization of Ti-added MgB_2/Cu superconducting wires using Vickers hardness test

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Abstract

The Ti-added (0%, 5%, and 10%) MgB_2/Cu superconducting wires were prepared by the powder-in-tube (PIT) method. Mechanical properties of the samples were then characterized by using a dynamic ultra-microindentation experimental technique. We observed that the sink-in effect is significant in our samples. So, the loading–unloading (*P*–*h*) curves were analyzed by the displacement approach to indentation. It was found that hardness (*H*) and the effective elastic modulus (*E*) values increased with added Ti. In addition, these values showed peak load dependence (i.e. indentation size effect (ISE)).

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Keywords: MgB_2 wire; Ti-added; Hardness; Elastic modulus; Depth-sensing indentation (DSI)

1. Introduction

Since the discovery of superconductivity in MgB_2 at 39 K [1], many efforts have been devoted to improve its superconducting properties [2–5] by doping and/or adding other elements into MgB_2. Due to its relatively high transition temperature, high transport critical current density, large coherence length, simple crystal structure and low cost of materials, the compound has been attractive for various applications. In view of this, the studies on MgB_2 superconductor still continue to be of considerable research interest. Soon after its discovery, several groups have fabricated MgB_2 wires or tapes with high critical current density using the powder-in-tube (PIT) method [6,7]. The powder-in-tube method [8] appears to be the most practical and advantageous technique, but, requires a suitable, non-reactive sheath material, such as Ag [6], Fe [4], and Cu [9]. Copper is one of the most suitable sheath materials for the fabrication of MgB_2 composite wires due to its low cost and high ductility. On the other hand, the grain size of MgB_2 is an important factor for grain connectivity in these superconducting wires. Poor connectivity between grains and lack of pinning centres are the cause of low critical current densities in these wires. Many elements inside MgB_2 have been used to improve the critical current density J_c in the wires [8–10]. As an adding material, Ti has a hexagonal crystal structure similar to MgB_2. It can fill the voids and connect the grain boundaries since Ti has less molecular volume than MgB_2. Ti is a good electrical conductor and anticipated to improve connectivity between grains at the boundaries. So, the maximum improvement of J_c in MgB_2 has been observed with Ti doping [11]. It is also expected that the mechanical properties (i.e., hardness, elastic modulus) can be improved with addition of Ti. To the best of our knowledge, no investigation on the mechanical behaviours of Ti-added MgB_2 composite wires has been carried out.

Depth-sensing indentation (Vickers, Berkovich, and Conical), as a convenient, non-destructive, and low-cost test method, has received great attention in recent years with the need of measuring the mechanical properties from a small volume of materials. The most important quantities given by a depth-sensing indentation test are the indentation load *P* and penetration depth *h*. Therefore, the *P*–*h* relation is essential to determine the mechanical properties such as hardness and effective elastic modulus. Many methods are developed in the literature to analyze the *P*–*h* curves [12,13]. However, these methods may not be reliable when pile-up or sink-in behaviour occurs. The work of indentation approach of Stillwell and Tabor [14], and

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the displacement approach to indentation of Giannakopoulos and Suresh [15], are both applicable to the cases of pile-up and sink-in and do not require imaging of the residual indents.

In this paper, we characterized the mechanical properties of Ti-added MgB_2 superconducting wires at room temperature by using a dynamic ultra-microindentation experimental technique. To calculate the hardness and effective elastic modulus values, the loading–unloading (P–h) curves were analyzed by the displacement approach to indentation of Giannakopoulos and Suresh [15]. We noted that these values increased with added Ti.

2. Experimental

The Ti-added (0%, 5%, and 10%) MgB_2/Cu superconducting wires were prepared by the powder-in-tube method. The preparation details, structure, and superconductivity properties can be found in our previously published work [16]. A Vickers indenter was used in a dynamic ultra-microhardness tester (Shimadzu, DUH-W201S). The experiments were carried out on the samples annealed at 800 °C for 1 h. All tests were performed under the same operating conditions to avoid uncertainties arising from changes in the experimental procedure. To easily interpret the material behaviour at various depths, the maximum load was changed at regular intervals: 100 mN, 200 mN, 300 mN, 400 mN, 500 mN. The loading rate and hold time were 14.12 mN s^{-1} and 10 s, respectively.

3. Theoretical consideration

A typical load-penetration depth curve is shown in Fig. 1. During indenter loading, the material undergoes both elastic and plastic deformation. This loading part of the curve, with the subtraction of the elastic deformation, is used to determine the hardness of the material. The unloading part of the curve is essentially elastic and with some analysis gives information about the elastic material properties. The important quantities are the peak load (P_{max}), the maximum depth (h_{max}), and the final depth (h_f).

Accurate measurement of the contact area (A_{max}) is critical to the measurement of the hardness and elastic modulus by microindentation. This is particularly important since the contact area may be underestimated if material pile-up, due to plasticity, takes place at the edges of the indentation. Indentation sink-in may also occur in relatively brittle materials, and this may result in an overestimation of the contact area. Fig. 2 shows schematic illustrations of pile-up and sink-in around a sharp indenter. Giannakopoulos and Suresh [15] have examined this problem by three-dimensional simulations.

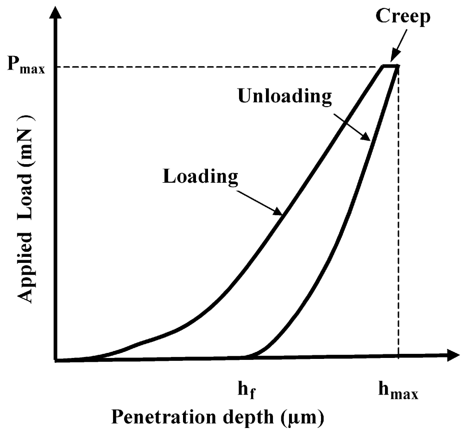

Fig. 1. Typical indentation cycles showing load–unload curves.

Table 1
Experimental data of the final depth, the maximum depth and the ratio of h_f/h_{max}

Samples	P_{max} (mN)	h_{max} (µm)	h_f (µm)	h_f/h_{max}
0% Ti-added MgB_2 wire	100	1.41	0.98	0.70
	200	2.34	1.56	0.67
	300	3.40	2.26	0.67
	400	4.56	3.19	0.70
	500	6.03	4.38	0.72
5% Ti-added MgB_2 wire	100	0.82	0.58	0.71
	200	1.29	0.86	0.67
	300	1.70	1.08	0.64
	400	2.05	1.28	0.62
	500	2.36	1.44	0.61
10% Ti-added MgB_2 wire	100	0.74	0.55	0.74
	200	1.13	0.77	0.68
	300	1.43	0.94	0.66
	400	1.72	1.10	0.64
	500	1.99	1.27	0.64

Parameter S is related to the ratio of h_f to h_{max} by [15]

$$\frac{h_f}{h_{max}} = 1 - d^* S \tag{1}$$

where $d^* = 5$ for the Vickers pyramid indenter. Using the value of h_f/h_{max} (Table 1), the S values are calculated by Eq. (1). Obtained values of S is put into Eq. (2) and then the contact area (A_{max})

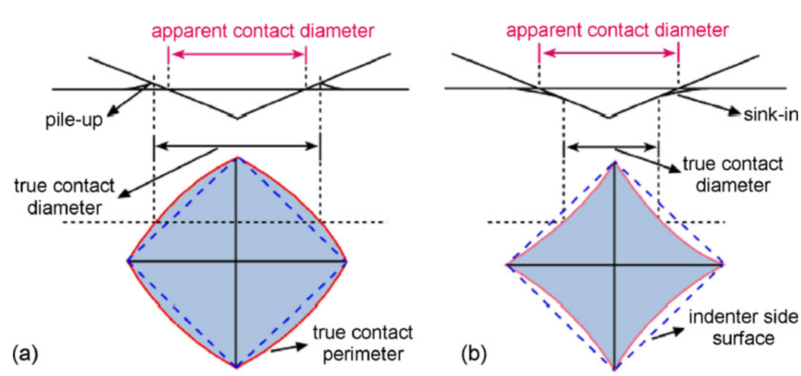

Fig. 2. Schematic illustration of: (a) pile-up and (b) sink-in around a sharp indenter [15].