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Features of the Transport Properties of Superconducting Magnesium Diboride Samples

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A comparative analysis of the experimental data on the microstructure and transport properties of the created and investigated samples of the high-temperature superconductor magnesium diboride MgB_2 is carried out. The differences in the pinning of long elastic Abrikosov vortices in the investigated thin-film and bulk samples are discussed.

Key words: high-temperature superconductor, Abrikosov vortice, magnesium diboride, pinning.

Проведено порівняльний аналіз експериментальних даних щодо мікро-структурних та транспортних властивостей створених та досліджених зразків високотемпературного надпровідника дибориду магнію MgB_2 . Обговорюються відмінності піннінгу довгих еластичних вихорів Абрікосова в досліджених тонкоплівкових та масивних зразках.

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1. INTRODUCTION

Electrical products from superconducting MgB_2 can be fabricated using relatively cheap and widely used synthesis technologies [1–3]. In superconducting products synthesized based on high-temperature cuprate superconductors, the factor limiting the critical superconducting current density in them is the barriers that form at the grain boundaries; magnesium diboride is free from this drawback. Studies of the pinning of Abrikosov vortices in high-temperature superconductors and an increase in the critical superconducting current density in them have shown that the most effective from this point of view are linear defects, namely, columnar defects arising from irradiation of samples with heavy ions with high energies of the order of 10 GeV [4], such defects as edge and screw dislocations [5–7], as well as such as nanorods that grow in the process of self-organization of various phases in the HTSC matrix [8].

It has been shown that such defects play an important role in terms of enhancing the pinning of Abrikosov vortices and increasing the critical current density not only in cuprate HTSCs but also in other superconducting materials, for example, in superconductors-ferropnictides [9].

The features of the implementation of pinning in the fabricated samples of magnesium diboride are discussed in this article.

2. EXPERIMENTAL DETAILS

Thin films of magnesium diboride MgB_2 are deposited on sapphire substrates with a size of $8 \times 8 \times 0.2 \text{ mm}^3$ and orientation (0001) in an argon atmosphere at a pressure of about 1 Pa by magnetron sputtering of magnesium diboride MgB_2 targets. The targets are prepared by hot pressing (at a pressure of 30 MPa, a temperature of 800°C for 1 hour from a mixture of amorphous boron and magnesium shavings taken in a 1:2 ratio (magnesium and boron are mixed and ground in a high-speed planetary activator)). As shown by X-ray studies using the Rietveld method, the fraction of inclusions of the MgO phase in the target made of magnesium diboride MgB_2 reached 24 at.% and 76 at.% for MgB_2 , while the critical current density of the target in zero magnetic fields in the temperature range of 10–33 K varied from $5 \cdot 10^5$ to $6 \cdot 10^4 \text{ A/cm}^2$, respectively, sputtering of these targets is carried out

using a dc magnetron (direct current magnetron), as a result of which magnesium diboride is deposited on the aforementioned substrates; the substrate temperature could vary from room temperature to 800°C.

Then (after deposition on a cold substrate), the films are annealed *in situ*. The material of the used target had a high thermal conductivity, also water cooling is used, the target is heated slightly (the temperature on the target surface did not exceed 100° C). According to the phase diagram of the Mg–B system, the MgB₂ phase forms at sufficiently high temperatures at which Mg intensively sublimes, including from the substrate during deposition. Therefore, the films are deposited on substrates at room temperature, followed by a short (~5 min) annealing in an argon atmosphere at a pressure of $P \sim 10$ Pa and a temperature of $T \sim 600$ –650°C.

Samples of superconducting materials based on MgB₂ obtained by synthesis at high pressures and temperatures (2 GPa, 600–1050°C) from a stoichiometric mixture of magnesium and boron powders and alloying additives in an amount of 10% wt. The powders are crushed and mixed in a planetary activator in an argon atmosphere until a homogeneous mechanical mixture is formed, which then placed in an anvil-type high-pressure apparatus with a depression. Heating is carried out under pressure, as well as a significant part of the cooling after completion of the synthesis. The synthesis is carried out in contact with hexagonal boron nitride under quasi-hydrostatic conditions. The phase composition is determined by powder X-ray diffraction using full-profile analysis by the Rietveld method, scanning electron microscopy with X-ray microscopy analysis, and also by Auger spectroscopy.

3. RESULTS AND DISCUSSION

Figure 1 shows typical microstructures (after cleaning their surface with argon ions in the chamber of the used microscope) of a thin film of MgB₂ (Fig. 1, *a*) and bulk samples synthesized based on Mg:2B (amorphous) mixture (Fig. 1, *b–d*): in an argon flow at 800°C (Fig. 1, *b*), at 1050°C and hydrostatic pressure of 2 GPa (Fig. 1, *c*), at 1050°C and hydrostatic pressure of 50 MPa by spark plasma sintering (Fig. 1, *d*). Similar microstructures of the studied samples are discussed in articles [10, 11] also.

The differences in colour in Fig. 1 correlate with changes in magnesium, boron, and oxygen at these places. The superconducting characteristics of the samples under study (j_c , B_{C2} , B_{irr} , T_C) are shown in Fig. 2. Variations in stoichiometry at the nanoscale level have been observed in the samples (as evidenced by color changes and qualitative image analysis).

The visible interfaces between regions with different concentrations

of elements (Fig. 1) are responsible for the pinning and high values of the density of the superconducting critical current of the studied MgB_2 material.

To analyze the peculiarities of the pinning of Abrikosov vortices in the samples under study, we plotted the dependence of the normalized pinning force $F_n(b)$ on the applied magnetic field and analyzed them within the framework of the Dew-Hughes model [12], comparing the main pinning mechanism responsible for the J_c value with the position of the peak in the dependence $F_n(b)$ (see Fig. 3). Here $F_n = F_p/F_{p,\max}$; $F_p = J_c H$; $b = H/H_{\text{irr}}$. At $T = 10$ K, the dependences of the vortex pinning force plotted for both thin-film samples (Fig. 3, *a*) and massive ones (Fig. 3, *b*, *c*).

It should be noted that the plots of the $F_n(b)$ dependence of thin-film samples demonstrate the position of the peak in the dependence close to 0.18–0.20. By following the Dew-Hughes model [12], this corresponds to pinning of vortices at grain boundaries in a superconductor, which correlates with the data obtained by studying samples by the SEM method (Fig. 1). Those in the thin-film samples under study, the main contribution to the pinning of vortices appears from the grain bounda-

Fig. 1. Typical microstructures of MgB_2 thin film (*a*), bulk samples (*b–d*). The materials contain Mg, B, O and a small amount of C. Brighter tones in the images correspond to lower boron concentrations and higher oxygen concentrations. Oxygen-rich inclusions and nanolayers are denoted by ‘I’ and ‘L’, respectively.

ries. For the studied massive samples, the graphs of the $F_n(b)$ dependence show the position of the peak on the dependence close to 0.1, which is close to the previous value, but somewhat less than it.

This indicates that the main contribution to the pinning of vortices is still made from the grain boundaries (this also correlates with the data from the SEM samples), but in this case, the pinning force is smaller.

Fig. 2. Dependences of the critical current density, j_c , on the magnetic field B for thin films (a), bulk samples (c, d); b— B_{c2} , B_{irr} temperature dependences of a thin film for two orientations: parallel to the substrate surface ($H//ab$) and perpendicular to it ($H⊥ab$), and e— B temperature dependences of bulks synthesized at high pressure.

In the theoretical paper [13], the pinning of Abrikosov vortices in superconductors at the most effective linear defects with a radius of the order of the coherence length ξ of the superconductor is discussed.

The efficiency of pinning on defects of this type is determined by their ability to pin a vortex along its entire length in the case when the magnetic field directed along the axis of a linear defect. In [13], the behaviour of an elastic long Abrikosov vortex and its exit from the potential well of a linear defect under the action of an inhomogeneous force studied theoretically. Model [13] is developed for the one-vortex approximation, that is, for the magnetic field in the superconductor $B \ll B_\phi = n_d \phi_0$; ϕ_0 is the quantum of magnetic flux, n_d is the two-dimensional concentration of linear defects in the material under study, that is, each vortex is pinned by a linear defect. It is theoretically shown in [14, 5] that at a finite sufficiently high temperature, the depinning (exit) of Abrikosov vortices from linear defects in superconductors occurs due to the formation of vortex fluctuations in the form of a partially depinned vortex loop in massive superconductors or in the form of vortex ‘tongues’ near the sample surface, having a size ex-

Fig. 3. Graphs of the dependence of the normalized pinning force $F_n = F_p/F_{p,max}$ on the applied magnetic field $b = H/H_{irr}$.

ceeding a certain critical value $l_c(J)$ and, accordingly, with an energy greater than a certain critical value $U_c(J)$: $l_c(J) \sim U_c(J) \approx J^{-1}$ [13]. These loop-like or tongue-like excitations are responsible for the instability of the pinned state of the vortex at $l > l_c(J)$ since such a loop grows in size under the action of the current-induced Lorentz force, creating a vortex creep from linear defects [13]. This scenario of thermally induced vortex escape under the action of the Lorentz force and the appearance of the subsequent vortex creep in superconductors with linear defects has been confirmed by numerous experiments performed on HTSC single crystals [15, 16].

It follows from the above that in the considered case of thin MgB_2 films, the grain size in them is commensurate with the thickness of the investigated film; as a result, the grain boundaries cross the film through and through, which ensures efficient pinning of Abrikosov vortices at these grain boundaries and, accordingly, a relatively high value of the density of the superconducting critical current in these films. In the case of massive samples with the same grain sizes, the grain boundaries often do not cross the sample through and through, as a result of which the vortices can more easily be depinned from such grain boundaries due to their high elasticity and tendency to stretch, to form loops and, as a result, depinning. This behaviour of vortices makes it possible to qualitatively explain the fact that, with approximately the same microstructure, the investigated superconducting thin films of MgB_2 have a relatively large pinning force of Abrikosov vortices and, as a result, an order of magnitude larger J_c (the density of the superconducting critical current) than the bulk samples under study.

4. CONCLUSION

1. Samples of high-temperature superconductor magnesium diboride MgB_2 in the form of thin films and the form of bulk samples have been created and experimentally investigated.
2. A comparative analysis of the microstructure of the created and investigated samples carried out.
3. At a temperature close to the boiling point of liquid helium, the transport characteristics of the created superconducting samples have been investigated.
4. It is shown that the main mechanism of pinning of Abrikosov vortices in the studied materials is pinning at grain boundaries, which provides a high value of the critical superconducting current density in them. The differences in the pinning of long elastic Abrikosov vortices in the investigated superconducting thin-film and bulk samples are discussed.

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