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Compression of a magnetic flux in the intergrain medium of a YBa₂Cu₃O₇ granular superconductor from magnetic and magnetoresistive measurements

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A method to determine a value of the effective field in the intergrain medium of a granular superconductor is proposed. The space between superconducting grains is considered to be a Josephson medium where passage of the transport current causes dissipation and the effective field is superposition of external field H and the field induced by magnetic moments of superconducting grains. The method proposed is based on the comparison of hysteresis field dependences of magnetoresistance and magnetization and their relaxation at H = const. By the example of granular YBa₂Cu₃O₇, it is shown that, in the region of weak fields, the effective field in the intergrain medium exceeds by far the external field, i.e., compression of a magnetic flux occurs. © 2011 American Institute of Physics. [doi:10.1063/1.3657775]

I. INTRODUCTION

It is quite a complex problem to study a pattern of penetration and distribution of a magnetic flux in granular superconductors by direct experimental methods because of a small geometric size of the intergrain boundaries, which are centers of the break of scale invariance of the order parameter. If the coherence length of a superconductor is about the length of the intergrain boundary, as in high-temperature superconductors (HTSC), then the set of these boundaries is considered to be a Josephson medium.¹ Depending on the ratio between external field H, first critical fields H_{C1} of superconducting grains (or, more exact, the first penetration field) which depend on a grain shape, and Josephson medium field H_{C1}, distribution of the magnetic flux in a granular material will be different. A few theoretical approaches to describe the behavior of the Josephson medium in the case $H_{C1J} < H < H_{C1}$ were proposed.²⁻⁶ Since the condition $i_{CI}(H) \ll i_{C}(H)$ for respective densities j_{CJ} and j_{C} of the critical current of the Josephson medium and of superconducting grains should be met, then, in the region of the intergrain boundaries, force lines of the magnetic flux may dense. It is clear that, for a real microstructure of a granular sample with the size distribution of grains, the density of the magnetic flux lines will be very inhomogeneous and hard, maybe impossible, to calculate. Possibility of densing the magnetic flux in the intergrain medium of a granular HTSC was taken into account in the model consideration in study.⁶ Some estimations of the degree of densing the magnetic flux for a granular Y-Ba-Cu-O (hereinafter, referred to as YBCO) were made in Ref. 7, where the authors investigated the dependence of the critical current on the external field and estimated magnetization of grains using the Bean model.

Indeed, the magnetoresistive effect is a useful tool to study the processes of trapping the flux and distribution of the flux in granular superconductors. In the mentioned case $i_{CI}(H) \ll i_{C}(H)$, one can use the magnetic field range in which the entire dissipation during passage of the transport current is caused only by collapse of carriers in the intergrain medium. For the granular YBCO discussed in this study, the external field range is up to $\sim 10^4$ Oe at the liquid nitrogen boiling temperature and the typical values of the above-mentioned parameters are as follows: H_{C1J} is of about that of the Earth or smaller,¹ $H_{C1} \sim 10^1 \div 10^2 \text{ Oe}, \quad j_{CJ} \sim 10^1 \div 10^3 \text{ A/cm}^2, \text{ and}$ $j_{\rm C} \sim 10^5 \div 10^6 \,\text{A/cm}^2$. We will show how to estimate the effective field in the intergrain medium by comparing experimental data on magnetization and magnetoresistance of granular HTSCs. In this case, the value of the effective field may exceed by far the external magnetic field in which a granular HTSC is placed.

The approach proposed here is based on the interrelation between the hysteresis behavior of magnetoresistance and magnetization of granular HTSCs. Previously, we unambiguously established that flux pinning in the intergrain medium does not contribute to the hysteresis behavior of magnetoresistance.^{8,9} The hysteresis of R(H) is caused by the effect of magnetic moments of HTSC grains, which make additional contribution to the field in the intergrain medium.^{8–10} To analyze the hysteresis R(H) dependence, we may use a considerable model simplification, introducing effective field B_{eff} in the intergrain medium of the material under investigation. Magnetoresistance is determined by a value of this field.

II. EXPERIMENTAL

The measurements were performed on a polycrystalline YBa₂Cu₃O₇ sample prepared using ceramic technology. The sample was tested by X-ray analysis, which showed the reflexes of the 1-2-3 structure and no additional reflexes of

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FIG. 1. Typical SEM image of the granular YBCO sample under study.

foreign phases. Scanning electron microscopic (SEM) study (Fig. 1) revealed a typical granular microstructure with an average crystallite size of $\sim 3 \,\mu m$ and a sample density of 5.8 g/cm³ which amounts to 91.2% of the theoretical value. The transition temperature determined from the magnetic measurements is $\approx 93 \,\text{K}$.

The magnetoresistance R = U/I, where U is the potential drop and I is the transport current, was measured by a standard four-probe method using pressure gilt electric contacts. The samples were $\sim 12 \times 1 \times 1 \text{ mm}^3$ in size. The R(T) dependences at different values of the external field (Fig. 2) were obtained on a PPMS-6000 facility (Quantum Design). During these measurements and the R(H) measurements at T = 82.5 and 90 K (insert in Fig. 2), the measuring transport current value was I = 1 mA.

During the R(H) measurements at high densities of the transport current, the sample was placed inside a copper solenoid in the liquid nitrogen medium to avoid the effect of heating at a stable transport current of 1 A. The external field

was applied parallel to the direction of the transport current. This configuration ensured the same demagnetizing factor of the entire sample in both electric and magnetic measurements. The magnetic measurements with a vibrating magnetometer were performed on a cylinder with a length of $\sim 6 \text{ mm}$ and a diameter of $\sim 0.6 \text{ mm}$ cut from the sample on which the R(H) dependences were taken.

The measurements of the hysteresis dependences of magnetoresistance and magnetization, including the relaxation measurements, followed the same algorithm for the R(H) and M(H) dependences. The sample was cooled in zero external field (The Earth's magnetic field was not shielded). The external field changed with the same rate dH/dt = 0.5 O e/s for the R(H) and M(H) measurements. Hereinafter, we denote the increasing and decreasing external field by respective symbols ↑ and ↓. The external field grew from $H_{\uparrow} = 0$ to $H_{\uparrow} = 100$ Oe; then, the time dependence of R(H) or M(H) was recorded for t = 3000 s; after that, the external field changed with the same rate to the next value $H_{\uparrow} = 200 \text{ Oe}$, at which the time dependence was recorded again. Following this procedure, the measurements were performed with a pitch of 100 Oe. After approaching $H_{\uparrow} = H_{max}$, where H_{max} is the maximum applied field, the external field decreased (relaxation at $H = H_{max}$ was not recorded), and relaxation of R(H) or M(H) was recorded for t = 3000 s at the same values of external field H₁, including $H_{\downarrow} = 50 \text{ Oe and } H_{\downarrow} = 0 \text{ Oe.}$

III. RESULTS AND DISCUSSION

The R(T) dependences in different external fields (Fig. 2) revealed the behavior typical of granular HTSCs.^{11,12} The sharp jump of resistance at $T_C \approx 93.5$ K corresponds to the superconducting transition in HTSC grains. Noticeable broadening of this R(T) portion is observed only in strong fields. The smooth portion of the R(T) dependence broadens in weak fields, which reflects the resistive transition in a subsystem of



FIG. 2. (Color online) R(T) dependences of the YBCO sample in different magnetic fields and R(H) dependences (inset) at indicated temperatures. The measuring current is I = 1 mA. The vertical line at T = 77.4 K shows the value of magnetoresistance R at I = 1.0 A, H = 1 kOe, and T = 77.4 K.

the intergrain boundaries (weak Josephson bonds).^{11–13} The R(H) isotherms shown in the insert in Fig. 2 confirm that it is correct to consider a granular HTSC to be a two-level superconducting system.⁴ First, dissipation occurs in a subsystem of the intergrain boundaries, magnetoresistance almost saturates, and then, after a certain value of the external field corresponding to the point where curvature of the R(H) dependence changes its sign, dissipation occurs in HTSC grains. It is obvious that magnetoresistance R of the subsystem of the intergrain boundaries depends strongly on the value of transport current. Experimental data in the Fig. 2 are obtained at I = 1 mA. The value of R at I = 1.0 A, H = 1 kOe, and T = 77.4 K relative to the R(T) dependences (I = 1 mA) in the external fields is shown in Fig. 2 as a vertical line at T = 77.4 K.

Figure 3 demonstrates the hysteresis M(H) dependence of the sample at 77.4 K. This dependence includes also the relaxation measurements observed as splashes. The data are given in Gs. Note that the value of magnetization of the granular HTSCs, including YBCO, can be determined by both intragrain pinning and microstructure of the sample. For the sample under investigation, at weak fields, the M(H) dependence is close to ideal: -4π M \approx H. The M(H) dependence is asymmetrical relative to the abscissa axis, which is typical of the HTSC materials at sufficiently high temperatures and may be caused by surface barriers¹⁴ and depinning.¹⁵ The character of relaxation is typical of the materials based on the yttrium HTSC (Ref. 14): in all the cases, relaxation is of the Anderson type, $M = M(t_0) (1 - (kT/Up) \times \ln(t/t_0))$; the values of pinning potential Up are consistent with the known literature data.¹⁴

The critical current density of the investigated sample obtained from the transport measurements is $\approx 150 \text{ A/cm}^2$ at T = 77.4 K. The magnetic measurements (Fig. 2) allow to determine the value of the intragrain critical current density using the well-known expression from the Bean model:¹⁶ $j_C = 30 \Delta M$ (emu/cm³)/d (cm), where ΔM is the height of the magnetization hysteresis loop in zero field and d is the average size of crystallites. Using the data presented in Fig. 3, we



FIG. 3. Hysteresis dependence of the magnetic moment of the YBCO sample. Splashes correspond to relaxation measurements for 3000 s. Arrows indicate the direction of the external field variation. Dashed line is the ideal diamagnetic response.



FIG. 4. Hysteresis dependence of magnetoresistance R(H) = U(H)/I (I = 400 mA) of the YBCO sample. Splashes correspond to the relaxation measurements for 3000 s. Arrows indicate the direction of the external field variation. Horizontal lines give an example of determination of the field hysteresis width $\Delta H = H_{\perp} - H_{\uparrow}$ in the marked points.

obtained the value $j_C(T = 77.4 \text{ K}, \text{ H} = 0) \approx 5 \times 10^5 \text{ A/cm}^2$. Thus, the condition $j_{CJ} \ll j_C$ (see Sec. I) is met.

Figure 4 shows the hysteresis dependence of magnetoresistance, R(H), of the sample at 77.4 K obtained under the conditions identical to those in Fig. 3. Apart from the pronounced R(H) hysteresis, this dependence has the sharp maximum when the external field grows. Similar to the magnetic data, the relaxation measurements are seen as splashes. To describe the magnetoresistance hysteresis, we consider a model representation of the behavior of a granular superconductor in the external magnetic field.

Let the superconducting grain in the external magnetic field possesses magnetic moment M_G , negative at $H = H_{\uparrow}$. Beyond the grain, the field $B_{ind}(r) = 4\pi M_G K(r)$ is induced whose direction and value are the functions of coordinates. The field is determined by both a demagnetizing factor of the grain and the field distribution inside the grain at $H > H_{C1}$, which can be taken into account in the function K(r). In the space between two grains (Fig. 5) close to one



FIG. 5. Schematic of the magnetic induction lines in the intergrain medium of the granular superconductor. Ovals correspond to the superconducting grains. M_G are the magnetic moments of the grains, B_{ind} is the field induced by M_G , and H_1 and H_1 are the external fields.

another, the distribution of the magnetic flux induced by the grains will be strongly inhomogeneous and the force lines of the magnetic flux will dense. The induced field for the area beyond the superconducting grains can be formally expressed as $B_{ind}(r) = 4\pi$ ($M_{G1} K_1(r) + M_{G2} K_2(r)$), where $K_1(r)$ and $K_2(r)$ involve demagnetizing factors and the effect of magnetic moments M_{G1} and M_{G2} on one another. For a real polycrystal, the situation is more complex. However, the pattern of the dense magnetic flux lines can be significantly simplified assuming that (i) all the superconducting grains have the same magnetic moment $M_G (M = \Sigma M_G)$ and (ii) instead of coordinate-dependent $K_i(r)$ functions, we deal with the effective field in the intergrain medium of the entire sample. Then, the effective field in the intergrain medium for the entire sample can be written as

$$\boldsymbol{B}_{\rm eff}(\mathbf{H}) = \boldsymbol{H} - 4\pi \boldsymbol{M}(\mathbf{H})\boldsymbol{\alpha}(\mathbf{H}). \tag{1}$$

Here, we take into account that, at M < 0 (growing external field), the magnetic induction lines from the superconducting grains in the area of the intergrain boundaries we are interested in are co-directed to the external field (Fig. 5(a)), while at M > 0 (decreasing external field, Fig. 3), the induced field is directed opposite to the external field (Fig. 5(b)). Parameter α arises due to the considerable simplification (ii) and includes averaging over local fields in the intergrain boundaries, the effect of demagnetizing factors, etc. Since the magnetic flux inside the superconducting grains is redistributed upon magnetization reversal, introduced parameter α can be, strictly speaking, a function of the field different for the increasing and decreasing field. Below, we will consider how the introduced simplifications (i) and (ii) may influence the description of the experiment.

Magnetoresistance caused by dissipation, i.e., by breaking Cooper pairs, in the intergrain boundaries will be a function of the modulus of this effective field: $R(H) \sim f(IB_{eff}(H)I)$. Since M(H) is the hysteresis function (Fig. 3), $B_{eff}(H)$ will also be a hysteresis function of the external field. The aforesaid qualitatively explains the observed R(H) hysteresis of the granular superconductors (Fig. 4). To describe the hysteresis R(H) dependence, it is reasonable to operate with such a parameter as the field hysteresis width.^{8,9} Indeed, for two points taken on different branches of the hysteresis R(H) dependence at which $R(H_{\uparrow}) = R(H_{\downarrow})$, we will have $B_{eff}(H_{\uparrow}) = B_{eff}(H_{\downarrow})$. Then, from expression (1) we obtain $\Delta H = H_{\downarrow} - H_{\uparrow}$; i.e., the hysteresis width of R(H) is determined as

$$\Delta \mathbf{H} = 4\pi\alpha(\mathbf{M}(\mathbf{H}_{\uparrow}) - \mathbf{M}(\mathbf{H}_{\downarrow})). \tag{2}$$

This expression is written on the assumption $\alpha = \text{const.}$ (field-independent). In real experiments on granular HTSCs, the density of the transport current is smaller than the density of the intragrain critical current by several orders of magnitude ($j \ll j_C$, which is satisfied in our case) and the transport current cannot affect the magnetic fluxes of HTSC grains. Consequently, parameter Δ H of the classical HTSC systems is independent of the transport current.⁹

To clarify the interrelation between the R(H) and the magnetic data, we varied value α to attain agreement between parameter Δ H of the R(H) hysteresis dependence (under the



FIG. 6. Hysteresis dependence $B_{eff}(H)$ of effective field in the intergrain medium calculated by expression (2) with the use of the M(H) data from Fig. 3 and $\alpha = 12$. Arrows indicate the direction of the external field. Horizontal lines give an example of determination of the field hysteresis width $\Delta H = H_{\downarrow} - H_{\uparrow}$ in the marked points corresponding to the external field values from Fig. 4. Dashed line is $B_{eff}(H) = H$.

condition $R(H_{\uparrow}) = R(H_{\downarrow})$ and the same parameter for the hysteresis $B_{eff}(H)$ dependence (under the condition $B_{eff}(H_{\uparrow})$) $= B_{eff}(H_{\perp})$). It was found that, for the data presented in Figs. 3 and 4, good agreement is attained at $\alpha \approx 12$, i.e., at the fieldindependent value of α . The field dependence of B_{eff} modulus obtained from the experimental data on magnetization (Fig. 3) at $\alpha = 12$ is shown in Fig. 6. The hysteresis of B_{eff}(H) manifests itself in the hysteresis of R(H), since $R(H) \sim f(|B_{eff}(H)|)$. In addition, the $B_{eff}(H_{\uparrow})$ dependence has the maximum at H_{\uparrow} ≈ 200 Oe, i.e., at the same value of the external field as the $R(H_{\uparrow})$ dependence (Fig. 4). One may conclude that the local maximum of the field dependence of magnetoresistance, sometimes observed for the granular HTSCs,^{6,17–21} reflects the local minimum of the M(H) dependence of superconducting grains. In cited works,^{6,17} this maximum was related to H_{C1} which was not quite correct.

An example of determination of the field hysteresis width is given in Figs. 4 and 6. For $H_{\downarrow} = 700 \text{ Oe}$ (point D' in Figs. 4 and 6), the value $\Delta H = H_{\downarrow} - H_{\uparrow}$ corresponds to the length of the DD' segment. Figure 7 demonstrates the values $\Delta H = H_{\downarrow} - H_{\uparrow}$ from the R(H) (Fig. 4) and $B_{eff}(H)$ (Fig. 6) data in the range $H_{\downarrow} = 0 \div 770 \text{ Oe.}^{22}$

During relaxation measurements (H = const.), the magnetic moment changes in time. This leads to time variation of the effective field in the intergrain medium: $B_{eff}(H, t) = |H - 4\pi \ M(H,t) \ \alpha|$ and, consequently, to the time dependence R(t) at H = const. The value of ΔH can be considered as the length of the horizontal line between the value R(t = 3000 s) and the point of intersection between this curve and the R(H) dependence, as is shown in Figs. 8(a) and 8(b) for the relaxation measurements at $H_{\downarrow} = 600 \text{ Oe}$ and $H_{\downarrow} = 300 \text{ Oe}$. The example of determination of the values of ΔH from the $B_{eff}(H,t)$ dependences at $H_{\downarrow} = 600 \text{ Oe}$ and $H_{\downarrow} = 300 \text{ Oe}$ is given in Figs. 8(c) and 8(d), respectively. In Figure 7, these parameters are compared for the relaxation measurements at $H_{\downarrow} = \text{const.}$, it is also reasonable to compare the values of ΔH . For this



FIG. 7. (Color online) Field hysteresis width of R(H) (open symbols) and $B_{eff}(H)$ (closed symbols) from data in Figs. 4 and 6, respectively, as a function of H_{\downarrow} (the ordinate axis on the left). $\Delta H(H_{\downarrow})$ data (the ordinate axis on the right) from the relaxation measurements $R(H_{\downarrow} = \text{const.}, t)$ (open symbols) and the $B_{eff}(H_{\downarrow} = \text{const.}, t)$ dependences (closed symbols) in Fig. 8.

purpose, Figs. 4 and 6 show the points of intersection of the horizontal lines at $R(H_{\uparrow} = \text{const.}, t = 3000 \text{ s})$ and $B_{eff}(H_{\uparrow} = \text{const.}, t = 3000 \text{ s})$ with the corresponding R(H) and $B_{eff}(H)$ dependences. It can be seen that the lengths of segments AA*, BB*, and B*B' for the data from Figs. 4 and 6 nearly coincide (there is the difference for A*A'). In addition, segment CC' intersects with good accuracy the relaxation data on R(t) and $B_{eff}(t)$ at t = 3000 s for $H_{\uparrow} = 400, 500$, and 600 Oe.

Thus, at $\alpha = 12$, the values of parameters ΔH obtained from consideration of relaxation of magnetoresistance and the effective field are close. This indicates that we used the adequate approach to describe the hysteresis of magnetoresistance of the granular superconductors. As can be seen in Fig. 6, the value of the effective field in the intergrain medium may exceed by far the value of the external field, which confirms compression of the magnetic flux in the intergrain medium.

Nevertheless, we should note that the above simplifications (i) and (ii) yield, in some cases, not a completely adequate picture. As is known, the hysteresis M(H) dependence is determined by superposition of the Meissner currents and, at $H > H_{C1}$, by the trapped flux, which makes positive contribution to magnetization. With decreasing external field, the magnetic moment as an integral characteristic passes through the zero value $M(H_{\perp}) = 0$ in which the contributions of the Meissner currents and Abrikosov vortices have equivalent values. However, the distribution of the trapped flux inside the grains is inhomogeneous. As a consequence, outside the grains the induced field will be strongly inhomogeneous. In other words, in the vicinity of $M(H_{\perp}) = 0$ the contribution of the magnetic moments to the effective field in the intergrain medium can be much larger than it is predicted by dependence (1).

The aforesaid is illustrated in Fig. 9 where the weak-field portions of the R(H) and B_{eff}(H) dependences are shown. The R(H) dependence in this figure was measured at a transport current of 1 A at which the R(H_↓) minimum with decreasing field is already observed. The data presented in Fig. 7 for Δ H in the range H < 200 Oe were obtained from this dependence (I = 1 A). The B_{eff}(H) dependence has the minimum at H_↓ = 65 Oe and the R(H_↓) dependence has the minimum near H_↓ \approx 32 Oe. At the same time, intersection of the forward and backward paths of the R(H) and M(H) dependences (in this point Δ H = 0) takes place at the same value of H.

It is known that the M(H) dependence of the polycrystalline HTSC sample is determined, apart from intragrain pinning, by microstructure of the sample, i.e., M(H) of separated grains may differ from M(H) of a polycrystal. In this case, the assumption $\alpha = \text{const.}$ in the description of the



FIG. 8. (a) and (b) Portions of the R(H_↓) dependences, (c) and (d) $B_{eff}(H_{\downarrow})$ (B_{eff} is obtained by expression (2) from the M(H, t) data in Fig. 3 at $\alpha = 12$) including the relaxation measurements for t = 3000 s. In (a) examples of the initial (t=0) and final (t = 3000 s) points of the relaxation measurements at H_↓ = 600 Oe are shown. Examples of determination of field hysteresis width Δ H (the length of the horizontal line) from the relaxation measurements are given. Data on Δ H for different values of H_↓ are shown in Fig. 7.



FIG. 9. (a) Portions of the hysteresis R(H) dependences for I = 1 A and (b) $B_{eff}(H)$ (Fig. 6) in the region of weak fields including the relaxation measurements at $H_{\downarrow} = 50$ and 0 Oe. Arrows indicate the direction of the external field variation. Examples of determination of parameter ΔH from the relaxation measurements at $H_{\downarrow} = 0$ Oe are given.

field hysteresis width cannot yield good agreement. The sample investigated in this study exhibits a considerable diamagnetic response. For this sample, the initial path of the M(H) dependence in weak fields follows the nearly ideal line -4π M = H (Fig. 3). We investigated the YBCO polycrystals obtained using different technological regimes and exhibiting lower diamagnetic response (the values of the M(H) modulus near the extremum and M(H₄=0) are by 50–80% smaller than those for the YBCO sample in Fig. 3). It appeared that, to describe parameter Δ H from the experimental data on M(H) of these samples, it is necessary to consider the field dependence of introduced parameter α . Although even on the assumption $\alpha = \text{const}$, the path of the Δ H(H₄) dependence as well as the order of the Δ H values qualitatively coincide.

It is important that for all the investigated polycrystalline HTSC samples, including HTSCs of the bismuth and lanthanum systems, the values of parameter α obtained from comparison of the field hysteresis width and the experimental M(H) dependences is much larger than unity. Consequently, the effective field in the intergrain medium exceeds by far the external field (similar to the data in Fig. 6), especially in the region of weak fields, which is the manifestation of magnetic flux compression in the intergrain medium.

IV. CONCLUSIONS

In this study, the hysteresis dependences and relaxation of magnetoresistance and magnetization of polycrystalline YBa₂Cu₃O₇ have been investigated. Analysis of the results within the model of granular HTSCs proposed previously^{8,9} unambiguously shows that, indeed, in the intergrain medium the magnetic flux is compressed and, in the region of the weak external fields, the effective field can exceed the external field by an order of magnitude. This conclusion concerns the HTSC polycrystals, at least, to those prepared by a standard technique.

In our opinion, the described procedure of comparing parameter ΔH of the hysteresis dependences of magnetoresistance and the effective field can be used to estimate a degree of magnetic flux compression in the granular superconductors. It concerns, certainly, the materials in which the intergrain boundaries serve as weak (Josephson) bonds.

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