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Regularities in the temperature evolution of magnetic flux trapped by the intergranular medium of a high-temperature superconductor

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Granular high-temperature superconductors (HTSC) are characterized by the coexistence and interaction of two superconducting subsystems: HTSC granules and intergranular boundaries (Josephson medium). Depending on the thermomagnetic prehistory, the magnetic flux can be trapped either by both subsystems together or separately, or only in the weak subsystem of intergranular boundaries. In this paper, the conditions for the implementation of all these cases for the yttrium HTSC system are experimentally determined. The main attention is paid to the case when the Meissner state is realized in the HTSC granules, and the magnetic flux is trapped only by the Josephson medium. A previously undetected regularity in the temperature evolution of the remanent magnetization $M_r(T)$ is discovered in the case of flux capture only by the subsystem of intergranular boundaries. Namely, the temperature dependence of the normalized remanent magnetization $m_r(T) = M_r(T)/M_r(T = 0)$ is identical for different values of the maximum applied field, despite the significant difference in the corresponding values of $M_r(T = 0)$. At the same time, in a wide temperature range from 4.2 to $\sim 80\,K$ (the transition temperature of the subsystem of intergranular boundaries TCGB $T_{CGB} \approx 90\,K$), the functional dependence $m_r(T)$ follows the power law $\propto (1 - T/T_{CGB})^{0.5}$.

Keywords: magnetic hysteresis, remanent magnetization, Josephson vortices.

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1. Introduction

The hysteresis effects in behavior of magnetization of type-II superconductors are determined by magnetic flux trapping by the superconductor upon external field increasing above the first critical H_{c1}, or upon superconductor cooling in external field from temperature above critical temperature T_c. State of superconductor in zero external field after field application is characterized by remanent magnetization M_r, which reflects amount of magnetic flux trapped by superconductor. This value is important parameter for practical applications of superconducting materials as sources of constant magnetic field [1,2]. Remanent magnetization of superconductors is determined by pinning of the magnetic vortices, and value M_r is proportional to density of critical current J_c of superconductor, this ensures the ability to develop methods of proximity determination of J_c of superconducting materials. Behavior of remanent magnetization of granular high-temperature superconductors (HTSC) as function of maximum applied field H_{max}, value of field H_{FC}, in which sample cooling occurs from $T > T_c$, and temperature evolution M_r were rather widely studied for different classes of superconductors [3–6].

The granular oxide HTSC are characterized by presence of two superconducting subsystems. First subsystem, obviously, is HTSC-granules, second subsystem — mesh of intergranular boundaries, which exists due to Josephson coupling between HTSC-granules. In this subsystem the magnetic flux exists in form of Josephson vortices, and its critical parameters (H_{c1}, J_c) are significantly below the same for subsystem of HTSC-granules. So contributions from „weak“ superconducting subsystem (intergranular boundaries) and „strong“ subsystem (granules) occur on loop of magnetic hysteresis $M(H)$ of granular HTSC in different ranges of external field. Main characteristic features of loop of small hysteresis corresponding to subsystem of intergranular boundaries, were described and qualitatively explained rather long ago [7–12]. But recent detailed studies of dependences $M(H)$ ensure determination and explanation of features associated with interaction of subsystems of granules and intergranular boundaries [13–15]. Namely, subsystem of HTSC-granules forms „weak“ superconducting subsystem of intergranular boundaries, and at the same time, at definite magnetic pre-history, the effect of magnetic moments of granules on intergranular boundaries facilitates complete disappearance of the magnetic hysteresis associated with the subsystem of intergranular boundaries [13–15]. At that we can rather reliably determine the range of external magnetic field (from $H = 0$ to $H = H_{max}$), where dependence $M(H)$ of granular HTSC is determined either (i) only by subsytem of intergranular boundaries, or (ii) by both subsystems, or (iii) by subsystem of granules only.

There are many studies of temperature dependence of remanent magnetization of granular HTSC at different thermomagnetic pre-history [6,8,12,16–19]. But we can not say that there is clear understanding of features in behavior of dependences $M_r(T)$, especially for the case (i). Moreover, during the superconductor cooling in the external field the case (i) certainly is not met. In this paper we measure the dependences $M_r(T)$ of granular HTSC of yttrium system at such different values H_{max}, which lead to implementation of cases (i), (ii) and (iii). Main task was to determine regularities in temperature evolution of the remanent magnetization of granular HTSC linked with subsystem of intergranular boundaries, i. e. for the case (i).

2. Experiment

The polycrystalline HTSC with composition $Y_{0.75}Gd_{0.25}Ba_2Cu_3O_{7-\delta}$ was studied, it was prepared as per standard ceramic technology. According to X-ray diffraction data the sample is single-phase; according to results of energy-dispersive X-ray spectroscopy gadolinium in granules is distributed uniformly. The scanning electron microscope showed clear granular structure with average size of granules $\sim 2.7\,\mu$m; details of synthesis and characterization are given in [20].

The magnetization was measured on the original unit [21], where for the external fields up to $H_{max} \approx 1$ kOe creation the copper solenoid was used (loaded in cryostat with liquid helium), this excludes effect of the remanent field typical for superconducting solenoids. In range of fields $1-20$ kOe, for which the effect of remanent field in superconducting solenoid is insignificant as compared to field of the solenoid, the superconducting solenoid was used. Sample cooling was performed in zero external field; no special measures on Earth field screening were taken. Measurement report includes (1) dependences $M(H)$ at $T = 4.2$ K at different values of maximum applied field with further increasing of value H_{max}, and (2) temperature dependences of remanent magnetization $M_r(T)$ in zero external field, after application of field with value H_{max} and field decreasing to zero. The field change rate during measurement of dependences $M(H)$ was ~ 1 Oe/s for range of fields up to 400 Oe, ~ 5 Oe/s for range of fields $400-1000$ Oe and ~ 50 Oe/s for range of fields $1-20$ kOe.

3. Results and discussion

3.1. Contributions to magnetic hysteresis from subsystems of intergranular boundaries and granules

Figure 1, $a-c$ shows the hysteresis dependences $M(H)$ at $T = 4.2$ K at values H_{max}, corresponding to different ranges of external field. Figure 1, a illustrates the development of hysteresis loop upon H_{max} further increasing to 50 Oe. At $H_{max} = 10$ Oe noticeable hysteresis of dependence $M(H)$ is not observed, but at $H_{max} = 20$ Oe we can say about presence of irreversible behavior of magnetization, as can be seen from the upper insert of Figure 1, a. Then, at values H_{max}, equal to $30-50$ Oe, the difference between branches of increasing and decreasing external field of hysteresis dependence $M(H)$ becomes significant. In its turn, upon H_{max} increasing to 200 Oe, in range $50-200$ Oe the hysteresis again becomes evanescently low, as shown in the lower insert in Figure 1, a. The described evolution of hysteresis in region of low fields ensures statement that the hysteresis dependences in Figure 1, a are associated with contribution from subsystem of intergranular boundaries, i. e. small magnetic hysteresis. This hysteresis exists in range of fields to some threshold value $H_{1T} = 55 \pm 10$ Oe. Note that opening of the loop of small hysteresis occurs on the background of linear over field diamagnetic signal from HTSC-granules.

With further H_{max} increasing to 400, 600 and 1000 Oe the magnetization hysteresis becomes visible in all range of magnetic fields, see Figure 1, b. And this is associated with the opening of the hysteresis loop from the subsystem of granules. At that the contribution to general dependence $M(H)$ from subsystem of intergranular boundaries (small hysteresis) appears in form of arc-like features in region of small fields ($H < H_{1T}$) only at $H_{max} = 400$ Oe, and at $H_{max} = 600$ Oe contribution of small hysteresis in region $H < H_{1T}$ becomes evanescently low. this behavior is shown in top insert in Figure 1, b (marked as „arc"). Therefore, we can specify one more threshold field $H_{2T} = 550 \pm 100$ Oe, and if $H_{max} > H_{2T}$, then small hysteresis does not appear also in region of low fields.

Such behavior called „collapse of small loop of magnetic hysteresis" [13] is explained by the effect of magnetic moments of granules on the intergranular environment, as result the intergranular Josephson couplings are broken. Main reason of such collapse is the magnetic flux compression in the intergranular environment [22–27]. Based on some experimental results [22–27], the effective field B_{eff} in the intergranular environment is determined by superposition of external field H and field induced by the magnetic moments of granules M_G: $B_{eff} = H + M_G$. For scalar value B_{eff} the following expression is valid [22–27]:

$$B_{eff}(H) = H - \alpha \cdot 4\pi \cdot M_G(H). \qquad (1)$$

Here $M_G(H)$ — field dependence of magnetization on subsystem of granules, and parameter α shows magnetic flus compression in intergranular environment, as result of which the value of parameter α reaches $12-20$ [22–27]. Then the dependence $M(H)$ of granular HTSC as superposition of contributions from subsystems of granules M_G and intergranular boundaries M_{GB} will be determined by following expression:

$$M(H) = M_G(H) + M_{GB}(H - \alpha \cdot 4\pi \cdot M_G(H)). \qquad (2)$$

According to expression (2), the contribution of the second term will rapidly decrease with field increasing due to the increase in the magnetization modulus $|M_G|$ and large value of parameter α, and in region of fields $H > H_{1T}$ value $M_{GB}(H - \alpha \cdot 4\pi \cdot M_G(H))$ will be evanescently low. Similarly, if the maximum applied field H_{max} exceeds value H_{2T},

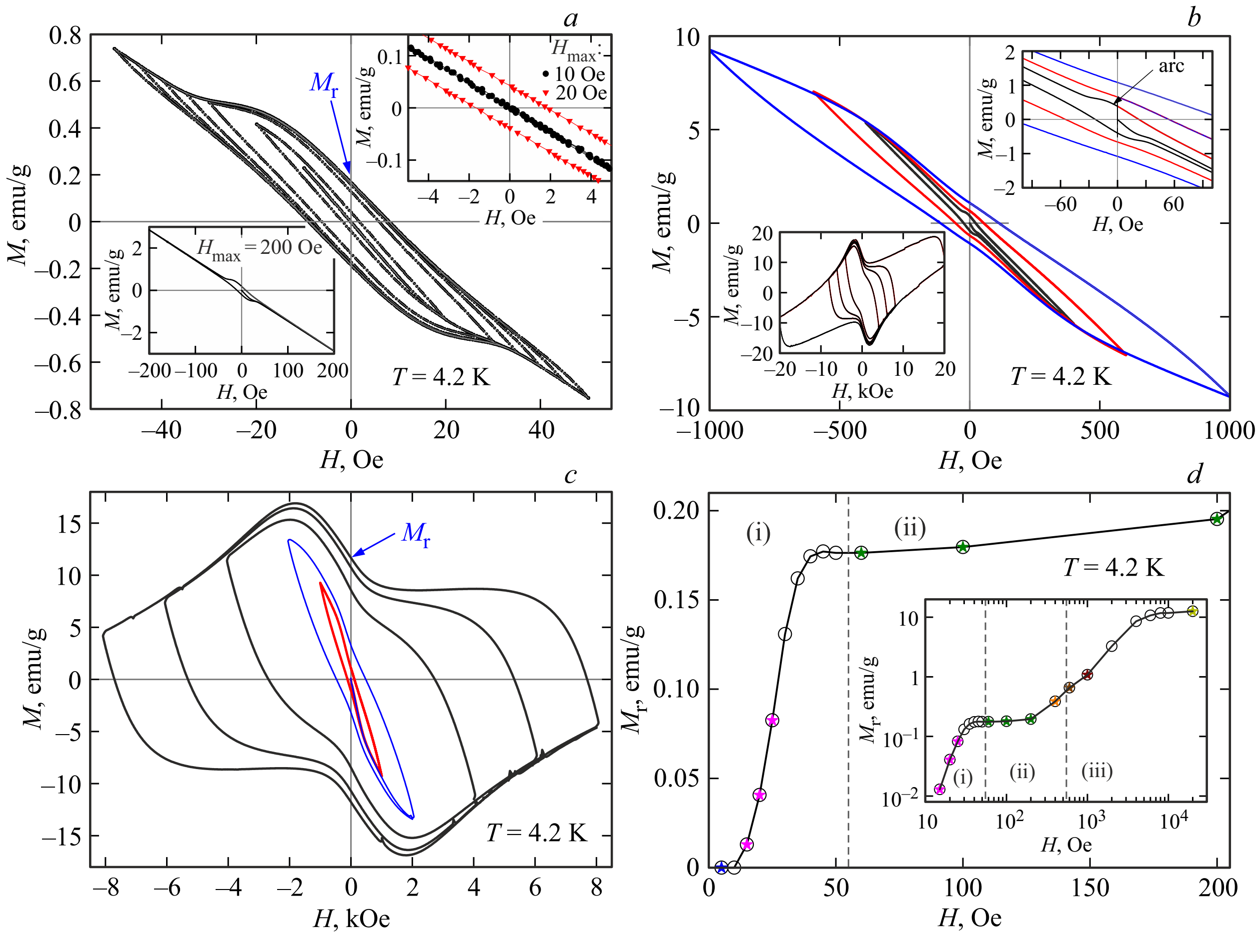

Figure 1. $(a)-(c)$ and bottom inserts to (a) and (b) — magnetization hysteresis loops of studied sample for different ranges of values of maximum applied field H_{max} — 50, 1, 8, 200 and 20 kOe respectively. In top inserts to (a) and (b) — details of behavior of dependences $M(H)$ in ranges of fields up to 5 and 100 Oe. In (d) the dependence of remanent magnetization $M_r(H_{max})$ is shown from H_{max} in range up to 200 Oe; insert to (d) illustrates the dependence $M_r(H_{max})$ in double logarithm coordinates. Vertical dashed lines separate the discussed cases (i), (ii), (iii), corresponding to different ranges of values H_{max}.

the contribution of second term in expression (2) will be evanescently low already in region of small fields (arc-like feature is not observed, see top insert in Figure 2, *b*) [13].

During further increasing of the maximum applied field (to 8 kOe) the magnetic hysteresis effectively develops from a subsystem of granules, and dependences $M(H)$ vie becomes typical for type-II superconductors, see Figure 1, *c*. In larger fields, up to 20 kOe, in form of dependence $M(H)$ the paramagnetic contribution from gadolinium ions appears, see bottom insert in Figure 2, *b* (see details in [20]).

As per set of measured dependences $M(H)$ at different values of H_{max} the dependence of remanent magnetization M_r on H_{max} was obtained, see Figure 1, *d*. In main field of this Figure the dependence $M_r(H_{max})$ is shown in dynamic range up to $H_{max} = 200$ Oe, and for entire range of fields H_{max} this dependence is shown in insert in Figure 1, *d* in double logarithm coordinates. Function $M_r(H_{max})$ has „two-stage view", characteristic for granular HTSC [12–15,18,27,28]. Such form of dependence $M_r(H_{max})$ reflects the presence of two superconducting subsystems, this was discussed above. The „first plateau" is reached in region of fields H_{max}, slightly lesser 50 Oe, this corresponds to complete opening of small hysteresis loop. Further M_r increasing is associated with beginning of field entrance into HTSC-granules, and exit to „second plateau" in field about 10 kOe corresponds to complete opening of hysteresis loop from HTSC-granules. Stop of $M_r(H_{max})$ increasing indicates that the magnetic flux reached the center of all granules.

So, from earlier performed data analysis Figure 1 we can distinguish three ranges of fields H_{max} (correspond to cases (i), (ii), (iii)), where contributions from subsystems of intergranular boundaries and granules are different:

(i) $|H| \leq H_{1T}$ ($H_{1T} = 55 \pm 10$ Oe) — contribution from intergranular boundaries only;

(ii) $H_{1T} \leq |H| \leq H_{2T}$ ($H_{2T} = 550 \pm 100$ Oe) — contribution from intergranular boundaries and granules;

(iii) $|H| > H_{2T}$ — contribution from granules only.

Figure 1, *d* shows dashed lines separating these ranges. The schematic presentation of trapped magnetic flux in

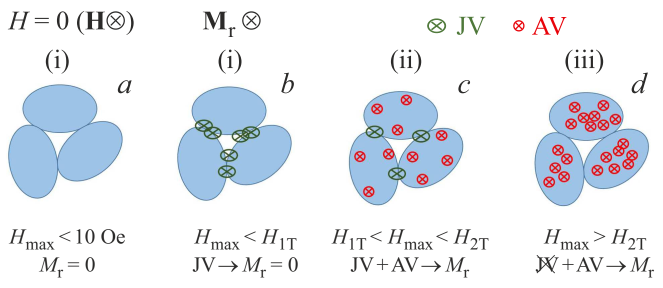

Figure 2. Schematic presentation of magnetic flux in granular HTSC in zero field after application of field H_{max}. Ellipses — superconducting granules, region between ellipses — intergranular boundaries (Josephson environment). On top the experimental conditions are provided (direction of applied external field), and designations are shown: JV — Josephson vortices, AV — Abrikosov vortices. The considered cases (i), (ii), (iii) are shown at specified ratios H_{max} and characteristic fields H_{1T} and H_{2T}, see text.

granular HTSC in zero external field after application of field H_{max} in ranges corresponding to cases (i), (ii), (iii), is shown in Figure 2. For (i) completely Meissner state (complete dynamic screening) is implemented, if H_{max} does not exceed the field $H_{C1GB} \approx 10$ Oe, see Figure 2, *a*.

Case (i) corresponds also to maximum applied field $H_{C1GB} < H_{max} < H_{1T}$ (55 ± 10 Oe), and at that the trapped flux is determined only by Josephson vortices, see Figure 2, *b*. Or case (ii) at $H = 0$ and $H_{1T} < H_{max} < H_{2T}$ (550 ± 100 Oe) the trapped flux is determined by both Josephson vortices and by Abrikosov vortices, see Figure 2, *c*. For (iii) at $H = 0$ and $H_{max} > H_{2T}$ all trapped flux — only Abrikosov vortices, see Figure 2, *d*. Contribution from Josephson vortices is negligible.

3.2. Temperature dependences of remanent magnetization for cases (i), (ii), (iii)

Temperature dependences of remanent magnetization $M_r(T)$, in conditions $H = 0$, after application of field H_{max} in ranges corresponding to (i) and (ii), are given in Figure 3, *a* and *c*. We can note the visible difference in form of dependences $M_r(T)$ (change in sign of curvature) for small and large values of field H_{max}.

At $H_{max} = 5$ Oe the hysteresis loop $M(H)$ is not opened yet, and value $M_r(H_{max} = 5\,\text{Oe})$ coincides with values of magnetization after cooling in zero external field. So, these measurements correspond to temperature evolution of magnetization associated with cooling in the Earth field. In this case the Earth field enters both in intergranular boundaries, and in granules, and sample demonstrates weak negative signal, practically independent from temperature in range up to ~ 90 K, see Figure 3, *b* and *d*. At the same time, from data in Figure 3, *b* and *d* we can see that dependences $M_r(T)$ obtained at values H_{max} in range of 15 to 200 Oe initially decrease to temperature ~ 90 K, and then repeat behavior of $M_r(T)$ at $H_{max} = 5$ Oe until temperature 92.5 K (when state with „zero magnetization" is implemented). This temperature is transition temperature of HTSC-granules T_{CG} (shown in Figure 3, *b* and *d*). It is obvious that described nonmonotonicity of dependences $M_r(T)$ (at H_{max} in range of 15 to 200 Oe) is associated with contribution from the trapped Earth field. But this contribution can be assumed as insignificant as already for dependence $M_r(T)$ at $H_{max} = 20$ Oe value $M_r(T = 4.2\,\text{K})$ by modulus exceed practically by order of magnitude the value $M_r(T = 4.2\,\text{K})$ at $H_{max} = 5$ Oe. During further analysis from dependences $M_r(T)$ at different H_{max} the dependences $M_r(T)$ at $H_{max} = 5$ Oe will be subtracted.

The dependences $M_r(T)$ at H_{max}, equal to 400 and 600 Oe, decrease up to temperature T_{CG}, and no features at temperature 90 K are observed. From the described behavior of dependences $M_r(T)$ in Figure 3, *a* and comparison of values $M_r(T = 4.2\,\text{K})$ at different values of H_{max}, we can conclude that transition in the superconducting state in subsystem of intergranular boundaries occurs at temperature ~ 90 K (further — T_{CGB}, as it is shown in Figure 3, *b*), which is somewhat lesser the transition temperature of granules $T_{CG} \approx 92.5$ K. If contribution to remanent magnetization from the subsystem of intergranular boundaries is significant, then in dependences $M_r(T)$ the transition temperature of intergranular boundaries T_{CGB} appears; and if contribution from subsystem of granules prevails, the transition at T_{CGB} either appears weakly, or is not visible. This agrees with consideration of the collapse of small hysteresis, see Sub-section 3.1 and expression (2).

To plot the normalized temperature dependences of the remanent magnetization from dependences $M_r(T, H_{max})$ the dependences $M_r(T, H_{max} = 5\,\text{Oe})$ were subtracted, and by extrapolation in low-temperature region the values $M_r(T = 0)$ were obtained. The dependences $m_r = M_r/M_r(T = 0)$ on normalized temperature $t = T/T^*_C$ ($T^*_C = T_{CGB}$ for data at H_{max} to 200 Oe; $T^*_C = T_{CG}$ for data at $H_{max} > 200$ Oe) are shown in Figure 4, *a* and *b*.

Behavior of dependences $m_r(t)$ for different values of H_{max} is in good agreement with ranges corresponding to cases (i), (ii), (iii), see Sub-Section 3.1 and Figure 1, *d*. And here we can speak about the discovered regularities in the behavior of the temperature dependences of remanent magnetization. The dependences $m_r(t)$ either fit on two master-curves for ranges $15\,\text{Oe} \le H_{max} \le 25\,\text{Oe}$ and $60\,\text{Oe} \le H_{max} \le 200\,\text{Oe}$ (Figure 4, *a*), or have different form for range $H_{max} \ge 600$ Oe (Figure 4, *b*).

For case (i) values H_{max} are 15, 20 and 25 Oe, at that values $M_r(T = 0)$ for $H_{max} = 15$ and 25 Oe differ by several times, and at the same time dependences $m_r(t)$ are identical. For $H_{max} = 60$ Oe dependence $m_r(t)$ is located somewhat below, and with it the dependences $m_r(t)$ at $H_{max} = 100$ and 200 Oe coincide. We can suppose that if value $M_r(T = 0)$, at least, is not so closed to value $M_r(T = 0)$ in vicinity of „first plateau" of dependence $M_r(H_{max})$, see Figure 1, *d*, then we can speak about some universal temperature dependence $m_r(t)$. In this case the

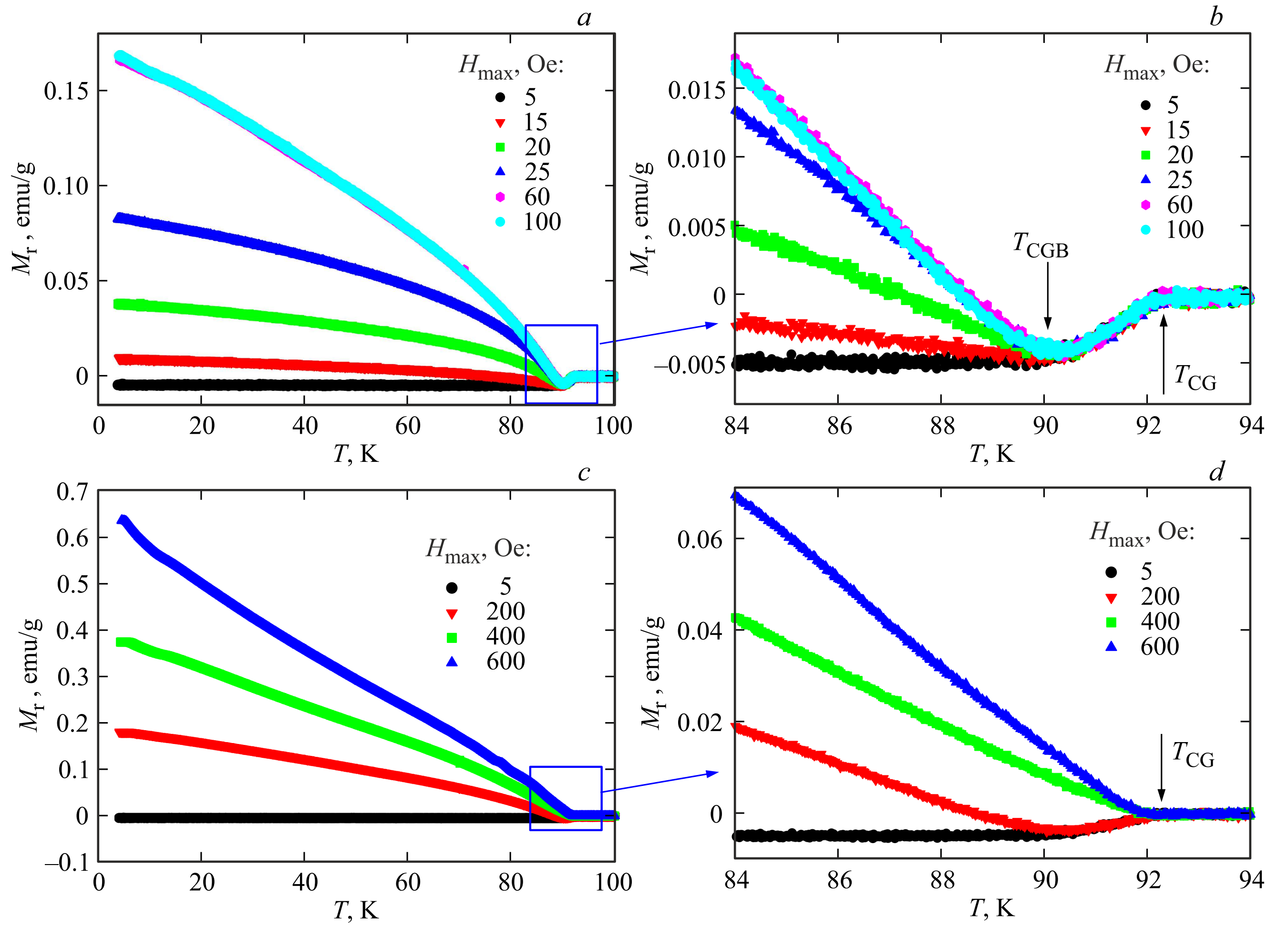

Figure 3. Temperature dependences of remanent magnetization $M_r(T)$ after application of field H_{max} — (*a*) and (*c*). In (*b*) and (*d*) — details of dependences $M_r(T)$ in region of high temperatures.

remanent magnetization is determined by the magnetic flux trapped in intergranular boundaries only.

For the case (ii), where there are contributions from both subsystems (both boundaries and granules), upon H_{max} increasing we can expect change in behavior of dependences $m_r(t)$. Coincidence of dependences $m_r(t)$ for fields H_{max} in range 60–200 Oe, apparently, is explained by approximately similar contributions to the remanent magnetization from two superconducting subsystems. This is confirmed by small difference of values $M_r(T = 0)$ for fields H_{max} in this range (see Figure 1, *d*). With field H_{max} further increasing the contribution from magnetic flux trapped by granules becomes prevailing. At that amount of trapped flux increases, this is reflected in change of functional dependence $m_r(t)$ with H_{max} increasing, see Figure 4, *b*.

Dependence $m_r(t)$ for H_{max} = 15, 20 and 25 Oe (case (i)) in wide temperature range follows exponential law:

$$m_r(t) = (1 - t)^n \tag{3}$$

with exponent $n \approx 0.5$. This is evident from Figure 4, *c*, where data m_r along axes of coordinates and $(1 - t)$ along axis of abscisses are indicated in double logarithm coordinates. Straight lines in Figure 4, *c* are plotted at $n = 0.5$ and 0.7. We see that in temperature range up to $\sim 0.9T_{CGB} \approx 80$ K, the dependence $m_r(t)$ follows the exponential law (3) with exponent $n \approx 0.5$, and near T_{CGB} the exponent n becomes equal to ~ 0.7.

The temperature evolution of the remanent magnetization of different HTSC-materials was discussed in many papers [4,6,11,12,17,19]. Here we note that, at least, in many cases in cited papers M_r was determined by the magnetic flux trapped by two subsystems — by intergranular boundaries, and by granules. I.e. it was not proved that case (i) was met. Exclusion is data in [12] obtained for first generation tapes of bismuth HTSC. Note that from model of critical state the value M_r is proportional to critical current J_c, it seems expedient to compare dependence $m_r(t)$ with the theoretical dependences $j_c(T) = J_c(T)/J_c(T = 0)$ for Josephson couplings. We note that agreement with the classical Ambegaokar-Baratov (for tunnel ransition), Kulik-Omel'yanchuk (for metallic-type transitions in „dirty" and „clean" limits) [29] or other theories [30,31] is not observed.

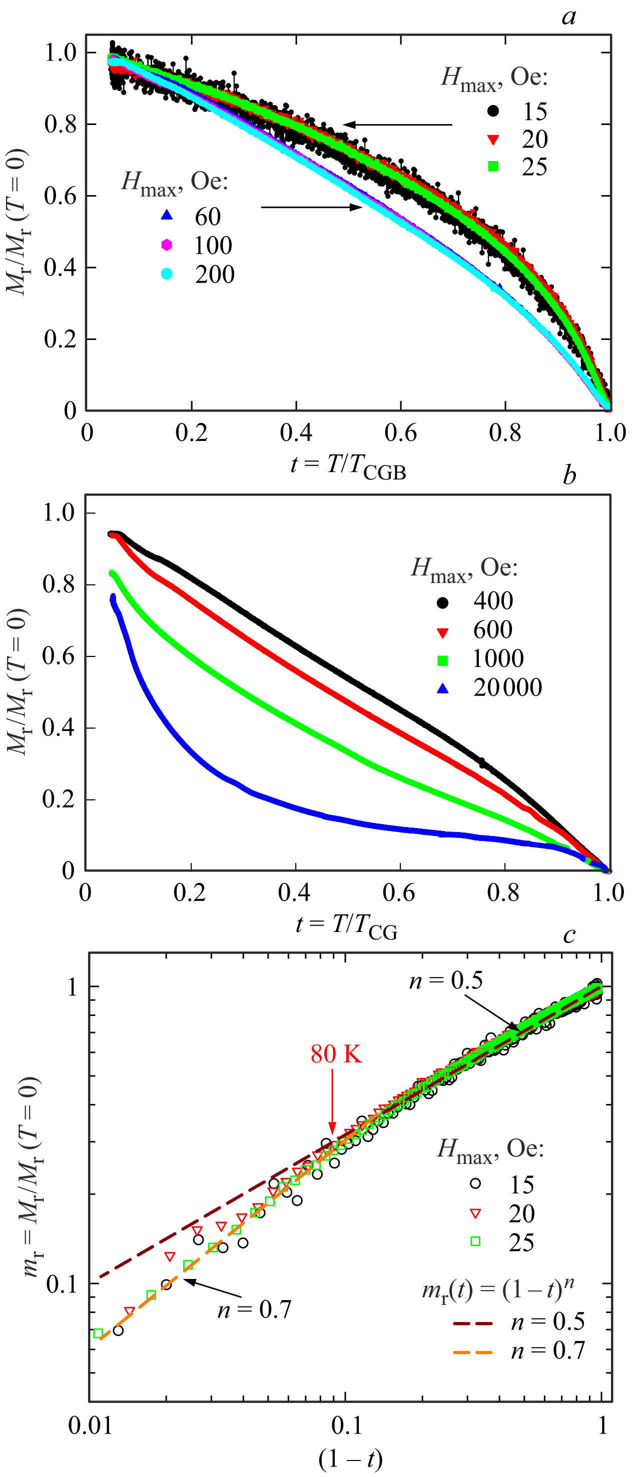

Figure 4. (a) and (b) — normalized dependences of remanent magnetization $m_r(t)$ at specified in inscriptions values of field H_{max}. In (c) — m_r on $(1 - t)$ in double logarithm coordinates (symbols); straight lines are plotted as per expression (3) at specified values n.

Studies on other granular systems may provide an answer in what extent the obtained functional dependence $m_r(t)$ is „universal". On other hand, may be the exit of Josephson vortices from the sample, determining the remanent magnetization decreasing with temperature rise, does not represent the behavior of densities of intergranular critical current density.

4. Concluding remarks

As a result of detailed measurements of magnetic hysteresis of granular HTSC of yttrium system in region of weak fields, the ranges of external field corresponding to magnetic flux trapping (i) in intergranular boundaries only, (ii) in intergranular boundaries and granules, and (iii) in HTSC-granules only were clearly identified And if the last two cases can be considered sufficiently studied, then for case (i) the following regularity was identified for the first time. In spite that after application of the external field 15, 20 and 25 Oe (at $T = 4.2$ K) the values of the remanent magnetization differ by times, the temperature evolution of normalized remanent magnetization $m_r(t)$ is identical for specified conditions. And this can be assumed as typical feature of the magnetic flux trapping by subsystem of intergranular boundaries of granular HTSC.

The functional dependence $m_r(t) = (1 - t)^n$ at $n \approx 0.5$ occurs in wide range of temperatures of 4.2 to ~ 80 K ($\sim 0.9T_{CGB}$), and at high temperatures the exponent n becomes equal to ~ 0.7.

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Conflict of interest

The authors declare that they have no conflict of interest.

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