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High pressure-high temperature effect on melt-textured $YBa_2Cu_3O_{7-\delta}$ high temperature superconductive material

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High pressure-high temperature (HP-HT) treatment of melt-textured $YBa_2Cu_3O_{7-\delta}$ at 2 GPa, and in the 800–950 °C pressure-temperature range for 15–30 min in contact with monoclinic pre-annealed zirconia induces: (1) the increase of the material density from 5.7 to 6.3 g/cm^3 (by 9%), (2) the increase of critical current density in the direction of *c*-axis of $YBa_2Cu_3O_{7-\delta}$ grains from 3–3.5 $\times$ 10^3 up to 7 $\times$ 10^3 A/cm^2 (in the self-field at 77 K) while in the *ab*-plane it remains unchanged (10^4 A/cm^2), (3) the increase of Vickers microhardness from 3.95 up to 5.3 GPa (estimated under the 4.91-N load). The increase in dislocation density in the (001) planes of HP-HT treated $YBa_2Cu_3O_{7-\delta}$ grains from 10^8 up to 10^{12} cm^{-2} may be one of the reasons of the increase in critical current density. The spaces between twin domains in $YBa_2Cu_3O_{7-\delta}$ before treatment were 100–150 nm. Completely detwinned wide areas or regions, where each second twin domain was narrowed to approximately 20 nm or tapered down to zero thickness within the first domain and the disappearance of 1/6 ⟨301⟩ stacking faults have been observed in the treated samples.

1. Introduction

The paper discusses high pressure-high temperature—induced variations of structure as well as superconductive and mechanical properties of melt-textured $YBa_2Cu_3O_{7-\delta}$ (MT-YBCO). The 1–5 GPa pressure and 650–1300 °C temperature ranges have been studied.

Among known bulk high-temperature superconductive materials, MT- YBCO possesses the highest level of cryomagnetic properties. Controlled seeding and melt-textured growth of YBCO results in trapped fields up to 0.9 T at 77 K and up to 6.9 T at 12 K [1]. Attained level of properties of MT-YBCO makes it possible

to construct HTSC electromotors with massive MT-YBCO rotors with maximum output power of about 19 kW at 3,000 rpm [1]. Such electromotors are smaller than conventional ones and have more than 50%-higher energy density on rotor surface.

For large scale applications the superconductive (SC) and mechanical properties of MT-YBCO, as well as resistance to degradation, call for further improvement.

The structure of MT-YBCO samples mainly consists of a few large textured grains or magnetic domains of $YBa_2Cu_3O_{7-\delta}$ (Y123) with finely dispersed inclusions of the non-superconductive Y_2BaCuO_5 (Y211) phase. Y123 grains are well oriented relative to each other, so that their *ab*-planes and *c*-axes are almost parallel. The presence of Y211 grains which can serve as pinning centers in the Y123 matrix leads to an increase in critical current density. A melt-textured material usually has micro- and macrocracks in the structure. Macrocracks appear during cooling process after melt texturing because of the difference in the coefficient of thermal expansion of Y123 and Y211 phases, while microcracks form during the oxygen saturation because of volume variation, for example, during the tetragonal into orthorhombic transformation of Y123 phase [2]. Microcracks are usually parallel to the *ab* planes of Y123 crystallites and do not practically affect the critical current density in the *ab* planes. Material density reaches 85–90% of the theoretical one.

K. Salama *et al.* [3, 4] have shown that a pressure treatment of specially oriented samples of MT-YBCO ($2 \times 2 \times 10$ mm, cut from single-domained blocks) under 25 MPa (uniaxial pressing at approximately 930 °C for 12 h) and a subsequent annealing in flowing oxygen (at 500 °C for 48 h) allows the increase of critical current density (j_c) at all field orientations with j_c at H$\|c$-axis (of Y123) being five times higher than that of the undeformed sample. It has been concluded that the changes in critical current density are directly correlated with the variation in defect density and especially in dislocation density. High temperature deformation increases the dislocation density and leads to the improvement in the current transport properties. After treatment under 25 MPa the 2×10^{10} cm^{-2} dislocation density, which is one order of magnitude higher than that in the initial sample has been observed [3].

We have shown previously [5–8], that after high pressure (2–5 GPa)-high temperature (900–1000 °C) treatment for 10–120 min of $MeBa_2Cu_3O_{7-\delta}$ (Me = Y, Nd, Gd, Sm, Eu) samples in contact with pre-annealed zirconia, their oxygen content and orthorhombic superconductive structure may be saved. Besides, sample's density, superconductive and mechanical characteristics may be essentially improved. Thus, using high pressure (under ⟨⟨high pressure⟩⟩ we mean pressures higher than 1 GPa)-high temperature treatment for MT-YBCO, we supposed to create higher defect density and to reduce essentially the treatment time. Temperature treatment under high pressure can affect the material structure for a shorter period than in the case of lower pressure. After the process there wouldn't be necessary any annealing in oxygen for superconducting properties restoration, because the proposed high pressure conditions may prevent oxygen liberation from a superconductor structure. It should be also mentioned that a treatment under lower pressures (hot or isostatic pressing) and a subsequent annealing in oxygen to restore superconductive properties leads to the reduction of material density (down to 10%) [9]. It is well known, that a higher superconductive material density corresponds to a higher resistance against degradation.

The present study of the HP-HT effect on MT-YBCO continues our earlier investigations [10–13].

2. Experimental technique

High pressure (1–5 GPa)-high temperature (650–1300 °C) conditions have been created with the help of a 2000 tons press in a recessed-anvil-type high pressure apparatus, described in [5–7]. After pressure application the samples have been heated by the direct current flow through the anvils and graphite heater. The accuracy of pressure and temperature measurements were ±0.15 GPa and ±20 °C, respectively. To protect samples from reaction with a graphite heater, during the treatment they were in contact with previously annealed (at high temperature) and precompacted monoclinic zirconia powder. The right choice of the material, which is in contact with the treated sample is of great importance. The recessed-anvil-type high-pressure apparatus is not hermetic with respect to gases. Under definite conditions zirconia may be an oxygen supplier and prevent oxygen losses from Y123 phase and thus, the transformation of the Y123 orthorhombic superconductive structure into the Y123 tetragonal nonsuperconductive one. When we use, for example, hexagonal boron nitride instead of zirconia, Y123 structure decomposition to liberate copper occurs in a short time, while the contact with zirconia under the same conditions allows us to prepare samples with high superconductive properties. But at 1 GPa pressure, even the contact with zirconia can't prevent the oxygen losses from Y123 structure. The importance of the material, in contact with the Y123 sample during HP-HT treatment is supported also by [12] where the liberation of oxygen from the Y123 structure under 2–9 GPa was observed even after heating at 600 °C for 10–120 min [17]. In an Ag container the liberation of copper from the Y123 structure occurred after treatment under 2 GPa, and 950 °C for 1 h [14]. The presence of silver oxide as an oxidizer (in an Ag container) does not provide necessary oxidizing environment (silver oxide does not change), but stronger oxidizers ($KClO_3$) induce the intense corrosion of the Ag container. In a Pt container under the same conditions, only tetragonal nonsuperconductive Y123 phase has been obtained [14]. The addition of an oxidizer into the Pt container was impossible because of the intense Pt corrosion. Only a low-temperature orthorhombic modification (with $T_0 = 90$ K) of Y123 phase with 5.1 g/cm^3 density has been observed in the Au container (if $KClO_3$ has been added as an oxidizer) after treatment under 1.5–3 GPa, 450–1100 °C for 1 h. All orthorhombic samples demonstrated the pronounced Meissner-Ochsenfeld effect at the liquid nitrogen temperature, but very often samples were crashed into very small pieces after the treatment. X-ray patterns of the

samples exhibit the orthorhombic modification of Y123 with exception of (104) reflection, whose position in all samples under study does not agree with the calculated one [14].

As the starting material in the present investigation, MT-YBCO samples of 9 mm in diameter, 7 mm in height and 30 mm in diameter, 17 mm in height have been used. At least 5 samples were treated under the same pressure-temperature-time conditions and the averaged results are presented.

MT-YBCO samples have been prepared by a modified Melt-Textured Method [16]: precalcinated 123 starting powder with an addition of Y_2O_3 ($Y_{1.5}Ba_2Cu_3O_{7-\delta}$), and about 1 wt% PtO_2 and/or CeO_2 are pressed uniaxially into cylindrical blocks; the cylinders are placed in a chamber furnace with a six side heater; after the growth onset temperature is attained, the samples (up to 30 in one batch) are cooled down in a small temperature gradient with 1 K/h; finally the samples are oxidized for 4 days in a separate process. PtO_2 and/or CeO_2 are added in order to reduce the size of Y211 inclusions and to disperse them uniformly. It is well known, that the homogeneous distribution of finely dispersed Y211 grains in Y123 matrix increases the material critical current density. The resulting material mainly consists of one or five textured Y123 grains with finely but nonuniformly distributed 211 inclusions (up to 30 wt %). Some copper, barium-copper containing phases are present in the part of the MT-YBCO sample is last to crystallize. The material density, according to hydrostatic weighing, is about 5.7 g/cm^3.

The sample structure and phase composition were examined by X-ray, using polarized optical and transmission electron (at 200 kV) microscopes. The oxygen content of $YBa_2Cu_3O_{7-\delta}$ phase was determined according to the equation of regression:

$$\delta = 60.975c - 71.134,$$

where c is the unit cell parameter measured in nm.

The SC transition temperatures were estimated by ac-susceptibility and the critical current densities from magnetization hysteresis loops measured by an Oxford Instruments 3001 vibrating sample magnetometer using Bean's model. The samples density (γ) was determined by hydrostatic weighing according to the equation:

$$\gamma = \frac{p_1 \gamma_{\text{water}}}{p_2 - p_3}, \text{ g/cm}^3,$$

where p_1— the weight of the sample in air, p_2— is the weight of the sample in air after immersion it in paraffin (in order to protect the penetration of water into the sample pores) and p_3— is the weight of the sample (previously immersed in paraffin) in water, γ_{water}— is the water density at the temperature of weighing.

Vickers microhardness was measured employing a Matsuzawa Mod. MXT-70 microhardness tester under 0.491- and 4.91-N loads. The fracture toughness of a starting sample, was estimated from the length of the radial cracks emanating from the corners of an indent (4.91-N load) using equation [15]:

$$K_{1c} = 0,0725 P/c^{1,5},$$

where P— is the indentation load and c— is the length of a radial crack emanated from the corner of the indent.

3. Results and discussion

Fig. 1 shows the structure of the starting MT-YBCO sample (a, b) and as-HP/HT treated samples (c, d, e, f, g, h) under the different P-T-τ conditions.

Three main regions that differ in the amount of Y_2BaCuO_5 (Y211) inclusions (green particles) in $YBa_2Cu_3O_{7-\delta}$ (Y123) matrix might be indicated in the structure of the starting samples (Fig. 1a): I—without inclusions; II—with a small amount of inclusions and III—with a high density of inclusions. The regions without (I) or with a small amount (II) of inclusions repeat the shape of the starting powder particles. One can see also a lot of cracks, which appeared during oxygenation (Fig. 1a). The structure of the starting sample also contains some pores (Fig. 1b), which sometimes reach rather large sizes. The HP-HT treatment may increase sample density up to the theoretical one (by 9%, see Table I). The results of microhardness tests, presented in Fig. 2 and the microscopic observations point to the structure homogenization after the HP-HT treatment. To assess the hardness of a material and not of the individual phases, of which the material is composed, it is necessary to conduct the study under the high loads. Under the 9.81-N load, to measure the indent was impossible because of the damage around it. Therefore, the investigations have been done under the 4.91-N load.10 indents have been made at 200-micrometers intervals in order to compare samples before and after the HP-HT treatment. As a result of the treatment the average value of microhardness (estimated under the 4.91-N load) increased from 3.95 up to 5.3 GPa (Fig. 2, Table I). The hardness of the initial sample has changed from 1 up to 6 GPa, while that of treated one, from 4 up to 6 GPa.

The microhardnes tests of the region III (with a large amount of Y211 inclusions) of the starting sample showed that it was very soft, the hardness under the load of 0.491 N was equal to 0.52 GPa. The hardness of the region II (with a small amount of Y211 inclusions) was higher and equaled approximately to 6.0 GPa (under 0.491-N load), the radial cracks from the corners of the indents obtained under the load of 0.491 N in this phase were absent. Under the 4.91-N load, the length of radial cracks was 39.1 μm, which corresponds to the fracture toughness of 1.46 MN $\times$ m$^{-3/2}$. The hardness (estimated under the 0.491-N load) of the regions with a small amount of Y211 inclusions after HP- HT treatment. increased from 6 up to 9 GPa.

Sometimes in the starting sample, regions without inclusions of the Y211-phase (marked I in Fig. 1a) are found. They are very brittle. Their hardness is 6.7 GPa, the radial cracks from the corners of the indents in this phase have been observed even at loads of 0.491 N. The fracture toughness of regions I was 0.71 MN $\times$ m$^{-3/2}$.

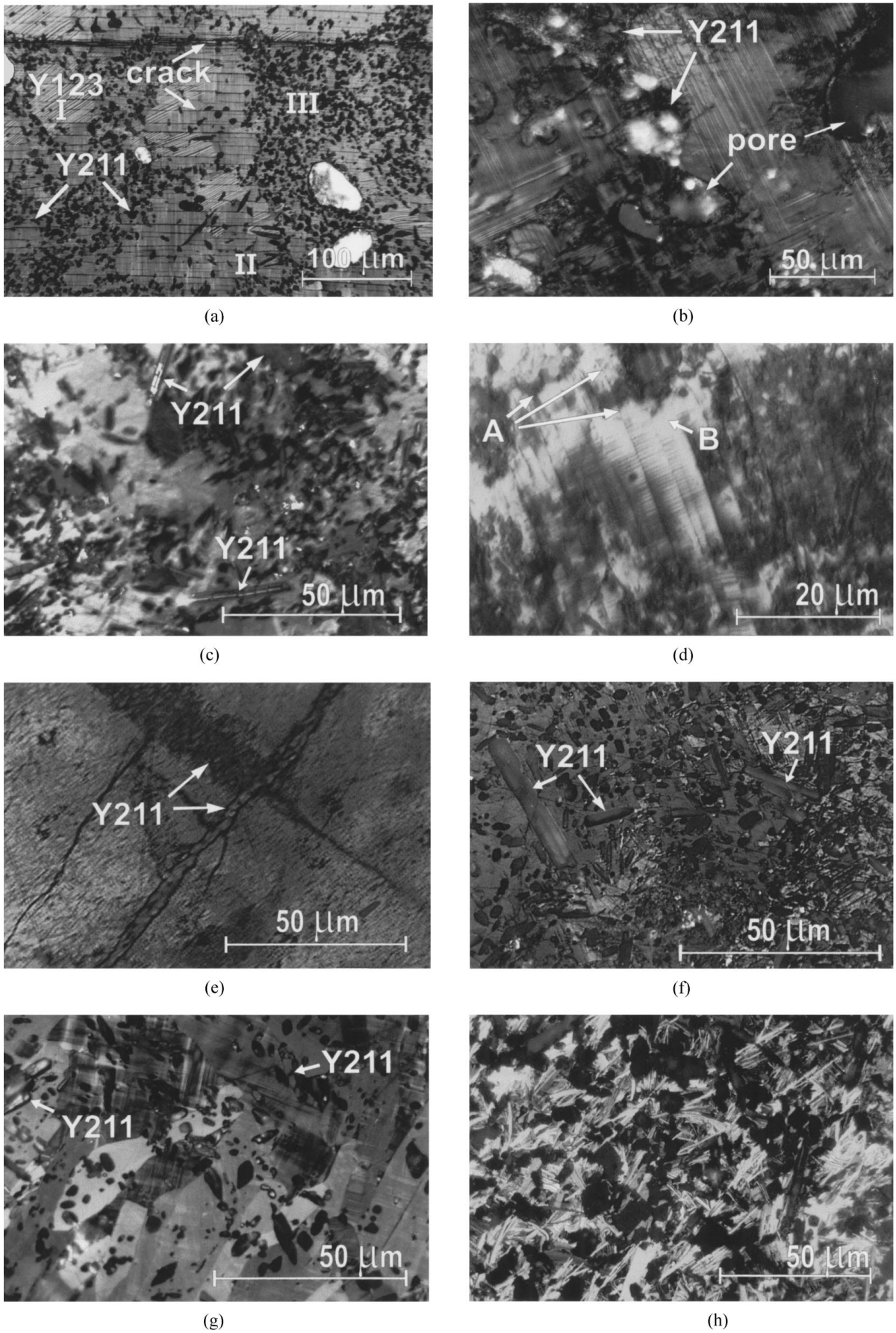

Figure 1 Structure of starting (a, b) and as-HP/HT treated (c, d, e, f, g, h) MT-YBCO samples under: (c) 2 GPa, 900 °C , 30 min; (d) 2 GPa, 900 °C, 15 min; (e) 5 GPa, 800 °C, 15 min, (f) 1 GPa, 900 °C, 15 min, (g) 2 GPa, 1200 °C, 10 min, (h) 2 GPa, 1300 °C, 10 min.

TABLE I Critical current density (j_c), microhardness (H_V), fracture toughness (κ_{1c}), Young modulus (E) and density (γ) of MT-YBCO before and after HP/HT treatment under the optimal conditions (2 GPa, 800-950 °C, 15–30 min)

j_c, A/cm^2, in zero magnetic field at 77 K		H_V, GPa, at 4.9 N		κ_{1c}, MN × m$^{-3./2}$, at 4.9 N		E, GPa at 0.05 N		γ, g/cm^3	
Before	After	Before	After	Before	After	Before	After	Before	After
10^4 ($\|ab$), 3×10^3 ($\|c$)	10^4 ($\|ab$), 7×10^3 ($\|c$)	3.95	5.5	0.71–1.46	No cracks	5–175	198	5.75	6.33

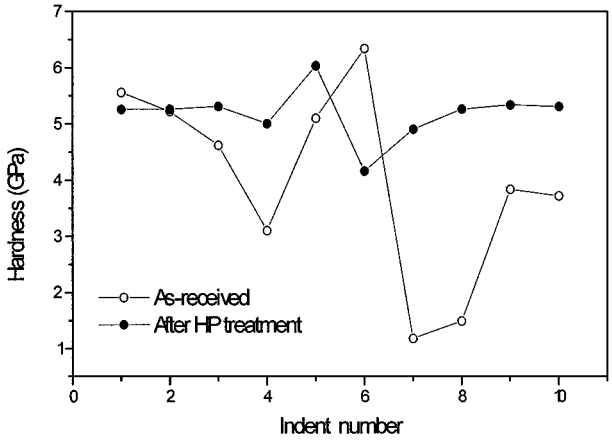

Figure 2 Microhardness of starting and treated at 2 GPa, 900 °C, 15 min MT-YBCO samples estimated under the 4.91-N load; the space between the indentor marks is 200 μm.

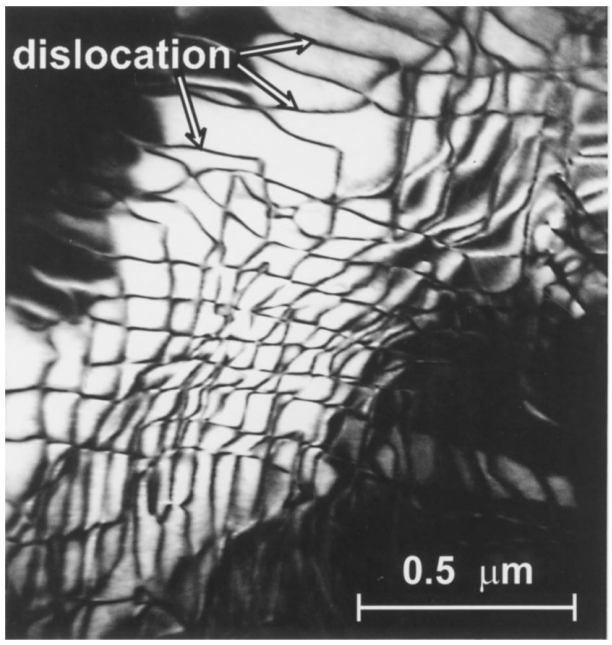

Figure 3 TEM image showing a wide detwinned region with the high density of dislocations lying in the (001) plane of Y123 in MT-YBCO (treated at 2 GPa, 900 °C, 15 min).

Fig. 1c, d, e, f, g, h present the variations in the structure after HP-HT treatment under different P-T-τ parameters, observed with an optical polarized microscope. Fig. 3 presents the results of TEM studies of the sample with the highest level of superconductive properties after HP-HT treatment (at 2 GPa, 900 °C, 15 min). Fig. 4 shows the *a*, *b*, *c* unit cell parameters of the Y123 phase of MT-YBCO before HP-HT treatment and their variations versus treatment parameters (pressure and temperature). Fig. 5 is a schematic representation of the critical current density variations related to the treatment pressure, temperature and time.

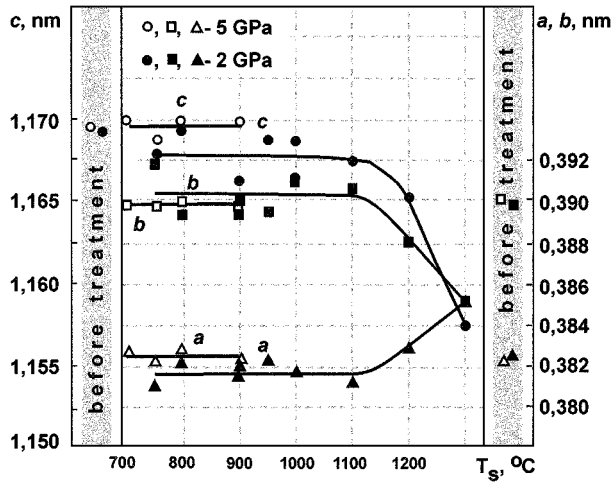

Figure 4 Unit cell parameters *a*, *b*, *c* of Y123 phase of MT-YBCO before and after HP/HT treatment under 2 and 5 GPa versus treatment temperature, T_s.

Fig. 1c, f show the structures of samples treated at 2 GPa and 900 °C for 30 min and at 1 GPa and 900 °C for 15 min, respectively. One can see big elongated recrystallized inclusions of the Y211 phase. The sample treated under 1 GPa has zero (at 77 K) critical current density (j_c) and the one treated under 2 GPa exhibits $j_c = 10^3$ A/cm^2 in the *ab*-plane (H$\|c$), i.e. an order of magnitude lower than that of the initial sample. The sample treated under 1 GPa demonstrates also some oxygen liberation from the Y123 structure as witnessed by the increase in the c-unit cell parameter (up to 1.1710 nm).

The samples treated at 2 GPa for 30 min at 800 °C and at 900 and 950 °C for 15 min exhibit the highest values of the critical current density $j_c = 10^4$ A/cm^2 in *ab*-plane (H$\|c$) and $j_c = 6 - 7 \times 10^3$ A/cm^2 in the direction parallel to the *c*-axis (H$\|ab$) (Fig. 5). We have mentioned some increase in j_c parallel to the *c*-axis of Y123 grains as compared with the initial sample, while in the *ab*-planes, it remains practically unchanged. The structure of these samples (Fig. 1d) exhibits neither variations in shape and size of the Y211 inclusions nor essential changes in the Y123 unit cell parameters (Fig. 4). But one can see the deformation-induced flexion of twin bands (marked ⟨⟨A⟩⟩ in Fig. 1d) and

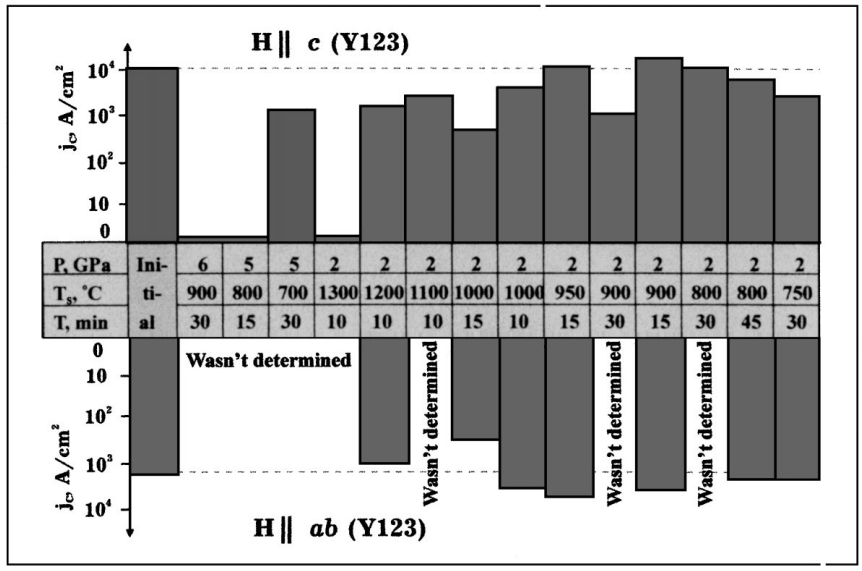

Figure 5 Schematic representation of the critical current density variations at 77 K and zero magnetic field value in *ab*-plain (H‖*c*) and in the direction of *c*-axis (H‖*ab*) of Y123 related to the treatment pressure, temperature and time.

detwinned regions (marked ⟨⟨B⟩⟩ in Fig. 1d). The TEM studies of the sample treated at 2 GPa, 900 °C for 15 min (Fig. 3) show that the density of perfect dislocations in the (001) plane of Y123 phase drastically increased from 10^8 up to 10^{12} cm^{-2} and even higher in some places, thus reflecting a high degree of plastic deformation. In the HP-HT treated structure, the dislocation lines outside the easy glide plane (001) have not been found. In the initial sample, the dislocation density on the (001) plane of Y123 phase is relatively low ($\sim 10^8$ cm^{-2}) and inhomogeneous, being larger typically around 211 inclusions. Large $1/6\langle 301\rangle$ stacking faults, elongated parallel to the screw component of their Burgers vectors, have been found to be attached to the 123–211 interfaces as well as subgrain boundaries, as commonly found in well oxygenated sample. The initial MT-YBCO samples were homogeneously twinned, the twin spacing being between 100 and 150 nm. The HP-HT treated samples represent either completely detwinned wide areas, or regions, where every second twin domain is narrowed from 100–150 nm to ∼20 nm or tapered down to zero thickness within the first twin domain. The $1/6\langle 301\rangle$ stacking faults disappeared. The observed four order of magnitude increase in the dislocation density on the (001) plane of Y123 may be the reason of more than twice increase in the critical current density (from 3×10^3 up to 7×10^3 A/cm^2 at 77 K, 0 T) in the direction parallel to the *c* axis of $YBa_2Cu_3O_{7-\delta}$.

The unit cell parameters of Y123 phase of MT-YBCO remain unchanged over the wide range of treatment conditions (Fig. 4), but a considerable variation in critical current density have been observed. We suppose that these variations are due to the Y211 grains coarsening and Y211 phase redistribution. As the pressure increases (up to 5 GPa), a large amount of macrocracks filled in with recrystallized green Y211 phase appeared (see Fig. 1e). The appearance of such big regions of nonsuperconductive phase leads to degradation of j_c (Fig. 5). At 2 GPa, the holding time longer than 30 min at 800 °C or longer than 15 min at 900 °C as well as an increase in the treatment temperature up to 1200 °C causes the Y211 phase grains to coarse and the j_c to decrease (Figs 1c, g and 5).

The further increase in the treatment temperature up to 1300 °C results in the Y123 phase decomposition as is demonstrated by the X-ray diffraction and microscopy examinations (Figs 1h and 4). In this case, the decomposition of Y123 occurs through the supersaturation of the structure with oxygen, i.e. the phases with more than 7 oxygen atoms per one unit cell form as witnessed by the decrease in the c-unit cell parameters. The same phenomenon for the case of single-phase Y123 ceramics has been described and argued by us in [6].

The variations of MT-YBCO superconductive properties are in close connection with the HP-HT treatment conditions and with the following structural variations: (1) the size of the Y211 phase inclusions; (2) the presence and size of cracks in the material, in which the Y211 phase recrystallizes; (3) the dislocation and twin densities, (4) oxygen content of the Y123 structure.

4. Conclusions

1. High pressure-high temperature treatment in the 800–950 °C temperature range for 30–15 min at 2 GPa may increase the sample density practically up to the theoretical one, homogenize the material structure, more than twice decrease the anisotropy of critical current density due to the increase in j_c (in the direction parallel to the *c*-axis of Y123 grains), essentially improve mechanical properties (microhardness, fracture toughness).

2. The complex investigations of HP-HT influence on the lattice parameters, structure, superconductive and mechanical properties of MT-YBCO have been done. The correlation between the treatment conditions, structural variations and material critical current density have been established.

3. The region of optimal HP-HT treatment conditions (to attain a higher j_c) is restricted primarily by the recrystallization and coarsening of Y211 grains.

4. As a result of HP-HT treatment, the increase in dislocation density in the (001) planes of $YBa_2Cu_3O_{7-\delta}$ grains from 10^8 up to 10^{12} cm^{-2} and essential narrowing of twin domains occur. We think that the increase in dislocation density along with the increase in material density are responsible for the observed increase in critical current density.

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