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INFLUENCE OF POWDER STRUCTURE ON TEXTURE FORMATION IN SUPERCONDUCTING Bi(Pb)–Sr–Ca–Cu–O CERAMICS IN MAGNETIC FIELD

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Starting powder of Bi(Pb)–Sr–Ca–Cu–O superconductor consisting mainly of 2223-phase was separated into fractions by precipitation in toluene. The high-oriented stacking of grains of the monocrystalline fractions with different dispersity has been obtained in magnetic field of 2 T at room temperature. The size of the powder grains in the range of 2–20 μm has no strong influence on the texture. The best result of grain alignment has been obtained for the powder grains of 10 μm in size.

Keywords: Bi(Pb)–Sr–Ca–Cu–O; High-T_c; Texturing; Field-induced; X-ray diffraction

It is known that the electric charge transport in high-T_c superconducting ceramics takes place mainly in the *ab*-plane. That is why it is necessary to make strong texture in the material, to attain the high critical current density in these ceramics. The most evident way of grain alignment is deformation. However, when deforming the material, destruction of crystallites takes place besides their alignment, and the ceramics change gradually into amorphous state (Kanai *et al.*, 1990). That prevents a large deformation, and hence, one cannot obtain a very sharp texture in bulk samples. Consequently, it is important to discover

other methods of grain alignment without considerable destruction of crystallites. It is desirable to orientate the powder grains during the sample formation. In this case, a well-textured sample with insignificant deformation of crystallites can be obtained after pressing.

In several works, strong magnetic fields were used successfully to align the $YBa_2Cu_3O_7$ ceramic crystallites. For example, Farrell *et al.* (1987) and Nakagawa *et al.* (1989) obtained well-textured samples by use of magnetic fields of 9 and 7 T, respectively. The present work was undertaken to clear up the possibility of oriented stacking of the powder grains of Bi(Pb)–Sr–Ca–Cu–O ceramics with the aid of a magnetic field.

The starting powder of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ consisting of 2223- and 2212-phases was separated into five fractions as follows. The powder was stirred up in a large amount of toluene and precipitated on the bottom of a vessel for various time periods. The most coarse-grained precipitate (first fraction) was obtained after 3 s. The remaining suspension was poured off into another vessel, stirred up again, and the second fraction was precipitated on the bottom of the vessel after 15 s. Quite analogously the third, fourth and fifth fractions were obtained after 30 s, 1 and 10 min, correspondingly.

Textured samples were prepared from the separated fractions using a magnetic field in the following way. A quartz cuvette of 1 mm in depth was filled with the powder mixed with toluene. The cuvette was placed in magnetic field of 2 T for 20 min till the complete evaporation of toluene. A sample was a precipitate of about 0.3 mm in thickness on the bottom of the cuvette.

The X-ray diffraction patterns of the dry precipitates were taken. The texture was estimated from the ratio of the diffraction lines intensities of the investigated precipitate to the ones of the untextured sample. The cuvette was set always horizontally in the electromagnet. The magnetic field was directed vertically ($H \| g$) or horizontally ($H \perp g$).

Figure 1 shows X-ray diffraction patterns of the fraction No. 4. All the patterns were taken under the same conditions, they are represented on the same scale. As shown in Fig. 1(b), the precipitate texture in which the preferred c-axis orientation is perpendicular to the bottom of the cuvette arises in zero field. This is obvious from the strengthening of the $00L$ lines in comparison with the corresponding lines of the untextured sample (Fig. 1(c)). Additional investigations show that this

texture takes place on the surface of the precipitate and, evidently, arises because of the surface tension of the fluid during drying. When the samples were prepared in the vertical magnetic field, this texture became stronger (Fig. 1(a)). In the case of the magnetic field parallel to the bottom of the cuvette, a new texture with the preferred c-axis orientation parallel to the bottom appears. It results in a strengthening of the $H00$ and $HK0$ lines and a weakening of the $00L$ ones (Fig. 1(d)). Figure 2 shows the partial (200) pole figure of the sample prepared in horizontal magnetic field. It is obvious that the c-axis of the

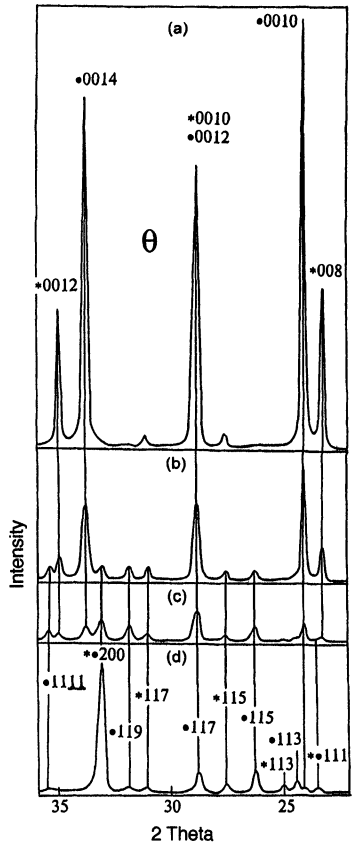

FIGURE 1 X-ray diffraction patterns of the samples prepared from the fraction No. 4: (a) in vertical magnetic field of 2 T, (b) in zero magnetic field, (c) untextured sample, (d) in horizontal magnetic field of 2 T. (•) 2223-phase, (*) 2212-phase.

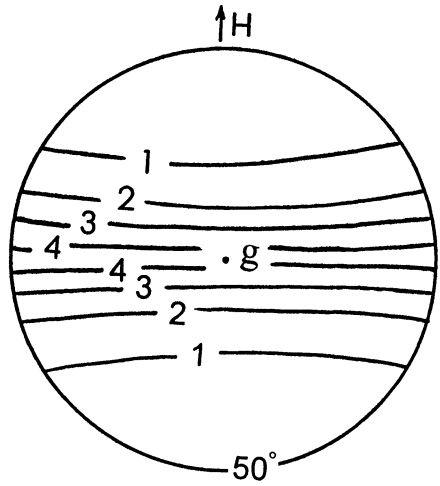

FIGURE 2 (200) pole figure of the sample obtained from the fraction No. 4 in horizontal magnetic field of 2 T. (*H*) direction of magnetic field, (*g*) direction of gravitation.

crystallites orientates along the field in the case of the vertical magnetic field as well.

The magnetic field was varied from 0 to 2.5 T to define its effect on the texture formation intensity which was estimated from strengthening of the •0010 and •*200 diffraction lines. As shown in Fig. 3, the dependence of the texture on magnetic field becomes weak in magnetic fields of more than 2 T, especially, in the case of vertical magnetic field. A field of 2 T was used in the present work.

To estimate the influence of the powder dispersity on the intensity of texture formation, all the fractions were investigated. The results are summarised in Table I.

One can see from Table I that the strongest texture is observed for the sample obtained from the fraction No. 4. Microscopic examination of powders shows that the fraction No. 4 consists mainly of monocrystalline plates of size from 5 to 10 μm in the *ab*-plane. Besides, there is a small quantity of clots of the same dimensions. Apparently, the latter are polycrystalline conglomerates. The amount of these conglomerates increases in the more coarse-grained fraction No. 3. The fraction No. 1 consists of the most coarse-grained conglomerates as a whole. From the microscopic and texture examinations, one can

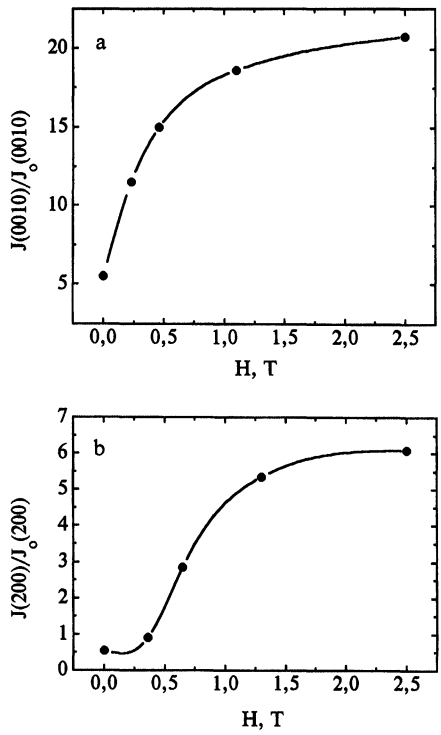

FIGURE 3 Dependence of the X-ray diffraction line intensities on grain alignment in the magnetic field: (a) $H \| g$, the 0010 line of 2223-phase; (b) $H \perp g$, the 200 line of 2223- and 2212-phases.

TABLE I Degree of orientation ($J(\bullet 0010)/J_0(\bullet 0010)$)* of the grains of the selected fractions in magnetic field

Fraction number	1	2	3	4	5	4
Size of the powder grains, μm	50–100	20–50	10–20	5–10	2–5	5–10
$J(\bullet 0010)/J_0(\bullet 0010)$	1.5	2.3	16	20	18	0.05
Field direction	$H \| g$	$H \| g$	$H \| g$	$H \| g$	$H \| g$	$H \perp g$

*$J(\bullet 0010)$ is the intensity of the 0010 diffraction line obtained from the 2223-phase of the precipitates, and $J_0(\bullet 0010)$ is the intensity of this line from the untextured sample.

conclude that weakening of the texture with increasing grain size of fraction is the result of increasing the amount of polycrystalline particles and decreasing the amount of monocrystalline ones. Apparently, such a powder structure is explained by chemical and structural heterogeneity of the starting material sintered before grinding.

Coarse-crystalline micro-parts of the sample are ground easily to monocrystalline plate-like grains of the 2223- and 2212-phases. Besides these phases, the rest of the micro-parts contained a noticeable amount of other compounds raising the strength of material. Apparently, these micro-parts remain, after grinding, in a shape of firm pieces which form the fractions No. 1 and No. 2. Therefore, after removing the coarse-grained fractions containing the particles of size more than 20 μm from the powder, one can believe that the remaining part of the powder is monocrystalline. Such a conclusion is correct only for the powder under investigation. In other powders of the same composition, this size fluctuates from 5 to 20 μm.

Thus, the high-oriented stacking in monocrystalline fractions of the Bi(Pb)–Sr–Ca–Cu–O ceramic powders of different dispersity has been obtained in a standard electromagnet at room temperature. The size of the powder grains in the range of 2–20 μm has no strong influence on the texture. The best result of the grain alignment has been obtained for the grains of size 10 μm.

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