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Gaps between the platelets in melt-processed $R\mathrm{Ba}_2\mathrm{Cu}_3\mathrm{O}_7$ (R=rare earth) superconductors

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The origin of the ''cracks'' between the platelets in melt-processed $R\mathrm{Ba}_2\mathrm{Cu}_3\mathrm{O}_7$ superconductors is investigated experimentally. The results convincingly demonstrate that these cracks actually develop during solidification. The relevance of this to the microstructural evolution during melt processing and to the critical-current densities is discussed. [S0163-1829(98)02609-5]

I. INTRODUCTION

The occurrence of parallel platelets separated by ''cracks'' is a distinctive feature of the microstructure of melt-textured $R\mathrm{Ba}_2\mathrm{Cu}_3\mathrm{O}_7$ (123: R=Y, Sm, Gd, Nd, etc.) superconductors.[1–3] In the samples processed in the absence of a temperature gradient along the sample, a few-millimeter-sized domains, each containing a group of platelets with a common c axis, occur. The domains themselves do not have a common orientation. In the presence of an imposed temperature gradient, the number of such domains decreases with the domains becoming larger and elogated with a preferred crystallographic orientation along the applied gradient. With suitable processing conditions like a steep temperature gradient, a single domain will result. The texturing of the platelets is considered to be an important factor contributing to the observed high current densities in optimally melt-processed 123 superconductors.[4]

The origin of the parallel platelets has been explained in different ways in the literature. The predominant view in the literature is that the platelets are formed by the cracking up of a single domain due to the stresses developed after the solidification is completed.[5–9] The gaps between the platelets are then a result of the cracking. The stresses have been attributed to the tetragonal to orthorhombic transformation occurring during the oxygenation of the samples in the solid state below 600 °C,[5,6] to the thermal expansion anisotropy of the 123 grains.[7–9] The hypothesis that the interplatelet cracks are formed after the solidification is completed has an important consequence: The domains are then essentially single crystals formed from single nuclei. The observed texture is a result of the fact that a single domain has cracked up.

Alternatively, it has been proposed that the platelets are sympathetically nucleated side to side during the solidification.[10,11] The formation of gaps also has been attributed to the presence of 211 particles ahead of the 123 growth front.[12–14] A mechanism of texture development by competitive growth due to a much larger growth velocity in the ab plane than in the c direction has been discussed.[15] This assumes independent nucleation of platelets in the liquid or on 211 particles. The interplatelet gaps could then develop during the solidification.

It is not definitively known at present where 123 nucleates. No coring of 211 has been observed. Isolated large domains of 123 have been observed. The properitectic $R_2\mathrm{BaCuO}_5$ particles are not supposed to be the favorite site for 123 nucleation.[10,16,17] 123 has been proposed to nucleate at perturbations in the liquid as domains.[16] Sympathetic nucleation on the side of platelets has also been proposed.[10,11] Understanding whether the interplatelet separation occurs during solidification or after the solidification process is completed is of critical importance in understanding how 123 nucleates and grows, how the various microstructural features evolve during melt processing, and how to control those parameters which are of importance in deciding the physical properties of the superconductors.

This paper reports the results of some experiments which were done with the objective of deciding unambiguously in favor of one or the other of the above views.

II. EXPERIMENT

123 powder used for melt processing was prepared by a chemical route and subsequent vacuum calcination. The phase purity of the powder was confirmed by powder x-ray diffraction. The powders are uniaxially pressed into pellets of size 15 mm×15 mm×7 mm under a pressure of 10 ton in a steel die. The pellets are then melt processed in a horizontal tube furnace, which has a temperature accuracy of ±0.5 °C, in ambient atmosphere. There is no measurable temperature gradient in the vertical or horizontal direction, in the space reserved for the sample in the furnace.

1-mm-thick slices, separated by a low-speed diamond saw from the bulk, were used for microstructural studies. They were embedded in a cold setting resin and polished on a fine cloth mounted on a rotating disk with different grades of diamond paste as grinding media. A Leitz optical microscope with polarized light and a JEOL JSM-840 scanning electron microscope (SEM) were used for the microstructural investigations. No etching was done to the surfaces.

III. RESULTS AND DISCUSSION

The samples were processed at different cooling rates from 1 and 15 °C/h from the peritectic temperature. All other experimental conditions were kept the same. No subsequent oxygenation was done. It was anticipated that faster cooling rates would induce instabilities at the solidification front and affect the platelet formation if the platelets were to be formed at that stage, and, otherwise, the platelet formation was likely to be unaffected by the cooling rate through T_p. Figures 1(a) and 1(b) compare the microstructures of melt-processed Y-

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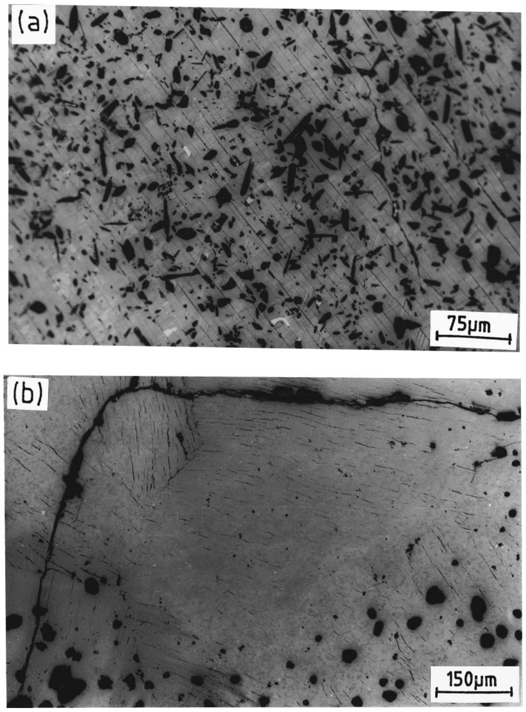

FIG. 1. Polarized light micrographs of partially oxygenated melt textured $YBa_2Cu_3O_{6+k}$ processed by cooling at (a) 1 °C/h and (b) 15 °C/h from the peritectic temperature. The bending of platelet gaps in the case of the 15 °C/h cooled sample can be observed.

123 cooled through T_p at 1 and 15 °C/h. The platelets and the interplatelet gaps in the slow-cooled sample are straight whereas they are zigzag in the faster-cooled sample. A region which has undergone the tetragonal to orthorhombic transformation can be seen in Fig. 1(b) as a gray region. The oxygen from air has diffused into the domain from a domain boundary and partially transformed the domain into the orthorhombic phase. The fact that the interplatelet gaps occur in the tetragonal as well as the orthorhombic region without any change in their width is significant because it proves that they are not a result of stresses accompanying the transformation. Figure 2 shows a partially transformed 123 sample with two differently oriented domains from a 1 °C/h cooled sample. The twins within the transformed region can be seen. It can also be noticed that the rate of oxygen diffusion and accompanying crystal structure transformation are greater when the cracks are perpendicular to the domain boundary than when they are parallel. This also proves that the cracks existed before the structural transformation.

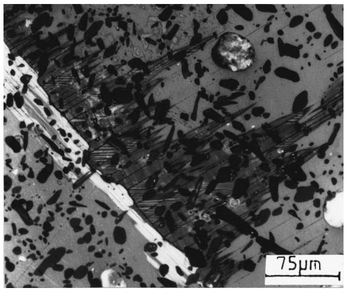

FIG. 2. Polarized light micrograph of partially oxygenated $YBa_2Cu_3O_{6+k}$ illustrating the growth of twins from domain boundary into the domains. The dark region, where twinning can be seen, is also transformed. The difference in the color of the dark regions under polarized light is because the two domains have different orientations.

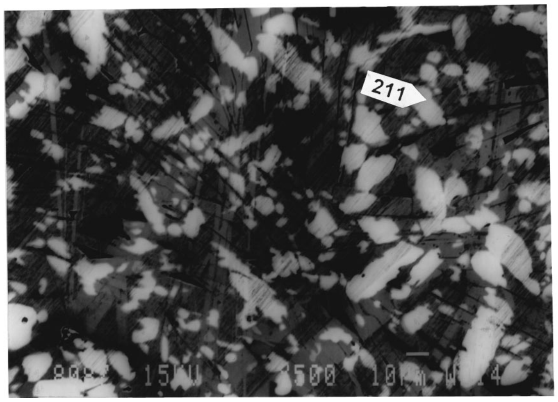

FIG. 3. Spherulitic growth of a 123 domain at relatively higher undercoolings (50 °C/h). An important observation that can be made from the figure is that some particles of 211 have been fully cut and some have been partially cut by the liquid phase. The partially cut 211 particles exist here unlike in slow-cooled samples probably because of the rapid transit of the solidification front in the present case due to the large undercooling.

In Fig. 3, we show a scanning electron micrograph from a sample that was cooled at 50 °C/h from the peritectic temperature to 930 °C. It shows the nucleation and growth of a spherical 123 domain (spherulitic growth) from the comparatively highly undercooled liquid. The reason for the spherical morphology of the domains in this case will be discussed elsewhere.[18] It can be noticed that the platelets and the gaps between them occur with almost the same width as in the earlier slower-cooled samples. A mechanism invoking stresses is difficult to argue in this case to account for the interplatelet gaps. It can be noted from the figure that some 211 particles have been partially consumed by the liquid in the gaps. Some other particles have been fully divided. It is likely that, in this case of high undercooling, the solidification front passed the particles so fast that some particles were only partially consumed. This is again a convincing proof for the presence of liquids during the formation of platelets and of the gaps between them. This also would account for the absence of large 211 particles in the melt-processed microstructure. Such particles would be divided into a size equal to the platelet width during melt growth.

IV. CONCLUSIONS

It has been shown that the platelets and the gaps between them, occurring in the microstructure of melt-processed R-123 superconductors, form during the solidification stage. As a consequence, the 123 platelets must be separately nucleated. Their textured growth could then occur according to the hypothesis of Muller and Freyhardt,[15] due to the difference in their growth velocities in different crystallographic directions in the local temperature gradient.

We have given an explanation for the observation that the average 211 size in the melt-processed microstructure is equal to the platelet width.

The parallel platelets are a distinctive feature of the microstructure of melt-‘‘textured’’ R-123 superconductors. It might be noted that the peritectic formation of η phase in the Cu-Sn system[19] (considered a model system for 123 formation) and that of Pr-123 (Ref. 20) (which occurs in a system which does not have the equivalent of an R_2BaCuO_5 phase), when subjected to a heat-treatment schedule equivalent to the melt growth process, yield large grains with properitectic phase inclusions, but there is no substructure within the grains. The microstructural features such as platelet width, the width of the interplatelet gaps, the size of the 211 inclusions, etc., are quantitatively interconnected;[21] as are the shrinkage accompanying melt processing and the size of the micropores,[22] with the platelet size. The width of the platelets is correlated to the R_2BaCuO_5 precipitate size in the final microstructure,[22] and through that to the flux pinning.[3] All these play an important role in determining the critical-current densities and a knowledge of the manner in which the platelets nucleate and grow is important in controlling such material properties.

ACKNOWLEDGMENT

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