Melt-Processing High-T_c Superconductors Under an Elevated Magnetic Field

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1. Introduction

Despite the fact that high critical current densities (J_c's) have been obtained in high temperature conductor (HTS) single crystals, the large-scale application of polycrystalline HTS, such as in wires, solenoids and magnets has been limited by the poor alignment of superconducting grains due to the presence of grain boundary weak links with low values of J_c [1].

Among the three major families of HTS, namely the Y-Ba-Cu-O (YBCO), Bi-Sr-Ca-Cu-O (BSCCO), the T1-Ba-Ca-Cu-O (TBCCO) systems, the best wires produced so far have been made of BSCCO material [2]. In this system, three superconducting phases are known, in particular Bi-2201 (T_c~20K), Bi-2212 (T_c~90K) and BI-2223 (T_c~110K). The crystallographic c-axis perpendicular to the longitudinal plane (Fig. 1). Although significant results in the fabrication of BSCCO thick films and tapes have been obtained [3-5], further improvements are required in order to render these compounds suitable for large-scale applications. Hence, it is important to fully understand the mechanisms that lead to a highly textured microstructure.

In the Bi-2223 compound it is difficult to align well the superconducting grains because of the complex formation mechanism of the Bi-2223 superconductor phase and the unsuccessful approach of melt-texturing the material, whereas in the fabrication process of Bi-2212, the sequence of partial melting and subsequent slow solidification is effective in obtaining a highly oriented c-axis grain microstructure. Thus far, four mechanisms for texture formation in melt-processed Bi-2212 have been suggested, namely a) heterogeneous nucleation on planar interfaces [6], b) buoyancy effects [7], c) opportunistic crystal growth [8] and d) anisotropy grain growth [9].

Despite these arguments [6-9], these models give an incomplete explanation of the various phenomena aobserved during the texture development of BSCCO materials. With respect to the mechanism of heterogeneous nucleation, the model excudes the state of texture in the center regions of films and tapes, and assumes the nucleation of superconductor crystals to occur preferentially for nuclei oriented with their c-axis perpendicular to the interface. Concerning the buoyancy effect, Hellstrom et al. [7,10] performed experiments in which the samples were partially melt-processed in different orientations with respect to the direction of the gravity force, and no significant change in texture was noted [7,10]. Furthermore, the buoyancy mechanism does not explain the enhancement in texture observed at the superconductor/Ag interface. These aspects imply that the buoynacy of 2212 is not a major factor inducing grain alignment. In the opportunistic crystal growth model [8], comaputer simulations [9] have shown that

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the effect due to this mechanism is too weak to explain the high degree of texture achieved in melt-processed samples. In addition, in accordance with the opportunistic model, one would expect to find in thick films or tapes various grains oriented with their c-crystallographic axis parallel to the interface. However, this type of orientation is rarely observed. Moreover, the model does not take into account heterogeneous nucleation and thus the role of the substrate is only considered as a limiting barrier. Finally, for the model based on anisotropic grain growth a direct observation has not yet been made. However, Bi-2212 grains in contact with each other and having their c-axes parallel with each other cannot explain the difference in texture as a function of thickness and the enhancement of texture at the free surface.

In general, although several mechanisms for the formation of texture in melt-processed BSCCO materials have been proposed [6-9], it seems that no model can explain all the observed microstructural features. Having this in mind, we propose in the following sections a model for the texture development of BSCCO materials based on interfacial energy considerations. As we shall see, although the interfacial mechanism of texture is very effective near the interface, the degree of alignment away from the interface is relatively poor, deteriorating with increasing thickness.

One possible route by which a strong crystallographic texture across the sample thickness can be produced is to melt-process the material under the effect of an elevated magnetic field. The magnetic alignment of non-doped superconductor grains results from anisotropy in the paramagnetic susceptibility associated with the Cu-O conducting planes. Since the susceptibility parallel to the superconductor grain crystallographic c-axis (χ_c) is higher than the susceptibility perpendicular to the c-axis (χ_{ab}) [11-13], non-doped superconductor grains should align with the c-axis parallel to the external field, H. For superconductors doped with magnetic rare-earth elements, the paramagnetic susceptibility is dominated by the R^{3+} ion, and the origin of anisotropy is single-ion anisotropy associated with crystal fields at the rare-earth site [14]. Grain alignment induced by a magnetic field has been confirmed by various groups [15-21], although very little work has been done on BSCCO thick films or tapes processed under elevated magnetic fields

The method of processing superconductor material at temperatures where a partially-molten state is achieved, facilitates the formation of the superconducting phases and enhances the degree of texture achieved. It is well established that if there are intergranular liquid phases, grains can be more easily oriented by the magnetic field. However, in the case of BSCCO/Ag films or tapes, a gradient in the liquid phase fraction may develop across the entire film thickness, as a result of the effect of Ag on the superconductor melting point [22]. As a result, it is possible that in thick films or tapes (>20 μm), stratified regions with a different degree of liquid phase may coexist across the whole thickness of the films. In other words, the presence of the Ag substrate in the tape or film configuration may change considerably the mechanism(s) by which grain alignment occurs in bulk materials.

In general, as a first step in providing suitable tools for the design of superconductor materials with tailored properties, it seems important to understand the effect of a magnetic field on the mechanism(s) by which superconductor grains are aligned. Thus, processing parameters such as temperature, degree of undercooling, and processing time and their related parameters such as grain size, grain shape and liquid volume fraction may affect the overall degree of alignment which can be produced by a magnetic field.

2. Results

2.1 A Model for Texture Development in the Absence of a Magnetic Field

We shall start by assuming that upon melt-processing, and during solidification, the nucleation of superconductor crystals occurs heterogeneously on the substrate. Furthermore, we shall consider that the nucleation is completely random, i.e., the c-axis orientation of superconductor nuclei has no preferred direction with respect to the substrate (Fig.2). In this fashion, and assuming anisotropic superconductor crystal growth behavior where growth along the ab-direction is preferred, as it has been shown in various investigations [23-25], one would predict after a time interval Δt two rather different situations should develop from the nucleation event assumed in Fig.2. It is assumed that during crystal growth the crystal shape develops into a plate-like configuration. Hence, in the case where the superconductor nucleus had its c-axis axis oriented parallel to the substrate, crystal growth would occur away from the substrate into the center (Fig.3), whereas for nuclei with their c-axis perpendicular to the substrate, crystal growth would follow the BSCCO/substrate interface (Fig.3). A comparison of the two cases shown in Fig.3 with experimental microstructural observations, indicate that crystal growth away from the substrate into the center of the film or tape is unlikely to occur. Thus, several consequences arise from these considerations which we will now discuss in the following section.

Based on the assumptions discussed above and an analysis of the texture reported by many experimental observations in which superconductor grains align and grow with their c-axis along a foreign surface, the following inequality may be written

$$\begin{aligned} &A_1\gamma_{Bi/S}(\underline{c})+2A_2\gamma_{Bi/L}(\underline{ab})+2A_3\gamma_{Bi/L}(\underline{ab})+A_1\gamma_{Bi/L}(\underline{c})< \\ &2A_1\gamma_{Bi/L}(\underline{c})+A_2\gamma_{Bi/S}(\underline{ab})+A_2\gamma_{Bi/L}(\underline{ab})+2A_3\gamma_{Bi/L}(\underline{ab}) \end{aligned} \quad (1)$$

where $\underline{c}$ is the vector orthogonal to the $\underline{c}$ surface, $\underline{ab}$ is the vector orthogonal to the $\underline{ab}$ surface, $\gamma_{Bi/S}(\underline{c})$ is the interfacial tension per unit area between a foreign solid surface and the $\underline{c}$ BSCCO surface, $\gamma_{Bi/L}(\underline{ab})$ is the interfacial tension per unit area between the $\underline{ab}$ surface of a BSCCO crystal and the peritectic liquid, $\gamma_{Bi/L}(\underline{c})$ is the interfacial tension per unit area between the $\underline{c}$ surface of a BSCCO crystal and the peritectic liquid, $\gamma_{Bi/S}(\underline{ab})$ is the interfacial tension per unit area between a foreign solid surface and the $\underline{ab}$ BSCCO surface, and A_1-A_3 are the respective interfacial areas. For the case of a plate-like crystal interchanging the surface areas A_2 and A_3 does not affect the results of equation (1).

Rearranging inequality (1) gives

$$A_1\gamma_{Bi/S}(c)+A_2\gamma_{Bi/L}(ab)<A_2\gamma_{Bi/S}(ab)+A_1\lambda_{Bi/L}(c) \qquad (2)$$

where the symbols have the same meaning as before. In other words, for superconductor crystals to grow along the substrate and not away from it, the sum of the interfacial tension between the superconductor c surface and a foreign surface, and the interfacial tension between the superconductor ab surface and the liquid needs to be less than the sum of the interfacial tension between the ab surface and the substrate, and the c surface of a superconductor crystal and the peritectic liquid.

As crystal growth progresses, and due to an anisotropy in the growth rates of the c and ab surfaces, the superconductor crystals develop into plates with aspect ratios of about 10 to 30. Therefore, if we assume $A_1 >> A_2$ we can approximate equation (2) to the form,

$$\gamma_{Bi/S}(\underline{c})<\gamma_{Bi/L}(\underline{c}) \qquad (3)$$

In other words, inequality (3) shows that the superconductor crystal minimizes its energy when its c surfaces remain in contact with a foreign surface. The foreign surfaces can be any surface with which a BSCCO crystal could come in contact, such as the silver substrate, particles of second phase, pores, other BSCCO platelets and the free surface. Assuming inequality (3) to be true, we may write for the case of heterogeneous nucleation on a foreign surface

$$\gamma_{Bi/S}(\underline{c})<\gamma_{Bi/L}(\underline{c})+\gamma_{S/L} \qquad (4)$$

where $\gamma_{S/L}$ is the solid/liquid interfacial energy per unit area of interface. If A is the area of contact, the change in free energy of the system can be written as

$$\Delta G = A\gamma_{Bi/S}(c) - A\gamma_{Bi/L}(c) - A\gamma_{S/L}<0 \qquad (5)$$

where the gain in energy is proportional to the area of contact (Eq.5). If the surface of the crystal in contact with the substrate is not the c surface, the area of contact is considerably smaller (typical aspect ratios for BSCCO crystals are between 10-30) and so the gain in energy associated with the contact can be expressed as

$$\Delta G_{Bi/S}(\underline{c}) < \Delta G_{Bi/S}(\underline{n}), \qquad \text{for} \quad \forall\ \underline{n}\neq\underline{c} \qquad (6)$$

where $\Delta G_{Bi,S}(\underline{n})$ is the energy gain when a BSCCO crystal surface with normal vector $\underline{n}\neq\underline{c}$ is in contact with a solid surface. Consequently, in this case, the contact is less stable and the superconducting crystal will more likely move away.

2.2 Melt Processing Bi-2212 Thick Films

SEM backscattered images of polished cross sections of various thick films with two different thicknesses, processed in zero field and a 10 T magnetic field, are shown in Fig.4 and Fig.5, respectively. When the films were processed under a zero magnetic field, the degree of texture decreased with increasing thickness of the film (Fig.4). In the case of the thick films melt-grown under a 10 T magnetic field, the degree of texture remains high with an increase in thickness (Fig.5). In this process, the grains align with the crystallographic c-axis (the direction of lowest growth rate) parallel to the magnetic field through the entire thickness of the film.

In Fig.6, the transport critical current densities of the films, measured at 4.2 K, are plotted as a function of their thickness for films processed under a zero and a 10 T magnetic field. When the thickness of the film increases, the J_c values decrease for both groups of films. However, the J_c values for the films processed under a 10 T magnetic field are higher than those obtained from the samples processed under zero field (Fig.6).

2.3 Melt-Processing Bi-2212 Tapes

Transport critical current densities of tapes processed under a 0T and 10 T magnetic field as a function of the maximum processing temperature T_m are shown in Fig. 7. It is clear that an increase in Jc for the taped processed under a magnetic field. In addition, the optimum processing temperatures T_m goes through a maximum around 883-885 C. SEM images of polished cross sections of the tapes processed under 0 and 10 T magnetic fields are shown in Fig.8. For the tapes processed under a zero magnetic field, many of the grains grow with their c-axis randomly oriented with respect to the Bi-2212/Ag, whereas for the tapes melt-grown under a 10 T magnetic field, a high degree of alignment is evident across the whole thickness.

2.4 A Model for Texture Development under a High Magnetic Field

In this model we are assuming that the magnetic field does not affect the process of nucleation and growth from the liquid. Instead, we suggest that rotation of superconductor grains in the early stages of growth under the presence of a magnetic field may be the cause for the increase in alignment.

This situation would be very similar to a rotation of particles in a free medium, since most of the material would be in the liquid state and thus particles can rotate without interacting.

Assuming that an anisotropic grain with a volume V is placed in a magnetic field H, the change in magnetic energy of the grain with a change in magnetic field can be written as

$$dE_m = -\vec{M}Vd\vec{H} = -(M_c \cos\theta + M_{ab} \sin\theta)VdH \quad (7)$$

where M is the magnetic moment per unit volume, which can be resolved in the two directions c and ab, and θ is the angle between the magnetic field and the c-axis of the grain. For high-T_c superconductors in their normal state, the magnetic moment M_c and M_{ab} are paramagnetic moments and thus, we can rewrite equation (7) as

$$dE_m = -(\chi_c \cos^2\theta + \chi_{ab}\sin^2\theta)VHdH \quad (8)$$

where χ_c is the paramagnetic susceptibility along the $\vec{c}$ direction and χ_{ab} is the paramagnetic susceptibility normal to the ab plane. On integrating equation (8) we obtain for the magnetic energy of a grain the expression

$$E_m = \int_0^H dE_m = -(\chi_c \cos^2\theta + \chi_{ab}\sin^2\theta)VH^2/2 \quad (9)$$

Rearranging gives

$$E_m(\theta,H) = -(\chi_{ab} + \Delta\chi\cos^2\theta)VH^2/2 \quad (10)$$

where $\Delta\chi$ is the difference in the volume susceptibilities of the grain.

Subsequent to the nucleation event, nuclei will start their growth under the influence of a magnetic field. In the early stages of growth, the grains will be completely surrounded by a liquid phase and thus, we shall treat the grains as small particles rotating in a free medium without interactions.

Let us start by considering the probability f(θ) that a grain has an orientation with angle θ under the influence of a magnetic field. This can be expressed, according to classic Boltzman statistics, as

$$f(\theta)d\theta = \frac{e^{-E(T,H,\theta)/kT}d\theta}{\int_0^{\pi/2} e^{-E(T,H,\theta)/kT}d\theta} \quad (11)$$

Let us now imagine a situation where the total number of grains is n. Thus, the mean number of grains with an orientation between θ and θ+dθ can be given by

$$n(\theta)d\theta = nf(\theta)d\theta = n\frac{e^{-E(T,H,\theta)/kT}d\theta}{\int_0^{\pi/2} e^{-E(T,H,\theta)/kT}d\theta} \quad (12)$$

The distribution n(θ) can be thus be related to an alignment parameter which can be used to quantify the degree of texture in melt-processed superconductor materials under the influence of a magnetic field. Let us define this alignment parameter F, such that F=1 for a completely aligned structure and F=0 for a completely random structure, in the form

$$F = 1 - \frac{s^2}{s_{H=0}^2} \qquad (13)$$

where s^2 is the variance of the distribution for a particular processing condition and $s_{H=0}^2$ is the variance of the distribution in the absence of a magnetic field.

In Fig. 9, the F factor is plotted as a function of the magnetic field for different grain sizes and a temperature of 875 C, which is approximately five degrees below the melting point of Bi-2212. The anisotropy in molar magnetic susceptibility $\Delta\chi^{molar}$ is approximately 22.5×10^{-5} cm^3/mol [12,13]. The anisotropy in volume magnetic susceptibility $\Delta\chi$ is 1.5×10^{-6}, if we assume a density of 5.5 g/cm^3 for the superconductor. As depicted in Fig. 9, when the magnetic field increases, there is a tendency for the texture to increase, except in the cases where the grain size is too small. Therefore a high degree of alignment can be obtained by increasing the magnetic field and the grain size. It is also evident that in the case of larger grain sizes, the magnetic field tends to saturate and thus increasing the magnetic field has only a negligible effect on the degree of texture.

2.5 Short Application of a High Magnetic Field

The previous model suggests that the enhancement in texture is primarily obtained through grain rotation during the early stages of crystal growth from the liquid. If the results of this model are valid, a short time exposure to the magnetic field during the initial stages of crystal growth could be sufficient to enhance the texture of HTS materials. Consequently, the opportunity of employing a magnetic field for processing HTS could be considered for large-scale industrial applications.

In Fig.10, the transport critical current density of the films, measured at liquid helium temperature, are plotted as a function of the processing conditions. The maximum value in J_c occurs in films where the magnetic field was present during the entire cooling process (film type A). The application of a magnetic field during the solidification from the molten state (film type B) results in higher J_c's than film type C, where the magnetic field is present during grain growth. The process in the absence of a magnetic field (film type D) exhibits the lowest J_c, although the value is very close (within the standard deviation) to the critical current density obtained for film C. The improvement in J_c for the thick films A and B is a consequence of the enhancement in the degree of texture.

3. Discussion

In general, BSCCO crystals that have nucleated on a foreign surface may have their c-axis oriented randomly with respect to the interface. However, crystals which have their c-axis perpendicular to the nucleating surface have a high probability, during crystal growth, of conserving their parallel orientation with respect to the interface, in order to reduce their overall interfacial energy. On the other hand, crystals which have their c-axis parallel to the interface are restrained from growing and are probably consumed by grains aligned with their c-axis perpendicular to the interface (Fig.11). Consequently, in the absence of other concurrent nucleation phenomena, the parallel alignment of crystals in contact with the substrate (or free surface) will propagate from the substrate (free surface) inward into the film, leading to a highly textured sample. In addition, secondary phases, such as the Bi-free phase, always present in the peritectic liquid, can also act as a good source for the nucleation of 2212 grains. However, due to the arbitrary shape of second phase particles, their surfaces are randomly oriented with respect to the substrate, and consequently, 2212 crystals nucleating on secondary phases are not aligned with respect to the silver substrate. In summary, even when the mechanism of homogeneous nucleation is absent, heterogeneous nucleation cannot be responsible for the texture observed in Bi-2212 samples.

Long range order may be attained when planar constraints are present, such as in the case of thick films or tapes. In these conditions, superconductor crystals will preferentially adhere to the silver substrate (or the free surface) with their c planar surface, resulting in a c-axis orientation perpendicular to the silver substrate (or free surface). Moreover, grains aligned with the substrate may transfer their c-axis alignment to adjacent grains. In this way, the orientation of the superconductor crystals may propagate across the oxide film. Evidently, the influence of the silver substrate or free surface on the texture of the grains will decrease with distance from the surface. In this manner, thicker films exhibit a lower degree of texture.

The development of texture is also influenced by large secondary phase particles (or pores) during crystal growth. Since superconducting crystals can decrease their interfacial energy by having their planar surface in contact with a secondary phase particle, and secondary phases have commonly an arbitrary shape, general alignment with the substrate is more unlikely to occur. As a consequence, the texture of the superconducting phase in the proximity of large secondary phase particles will be poor. Furthermore, since Bi-2212 thicker films contain a significant amount of impurity phase, the misalignment effect due to the presence of impurities will be greater in these samples. Therefore, besides the fact that the presence of secondary phases reduces the total superconducting volume fraction, the more significant influence on the transport properties of BSCCO superconductors is its deleterious impact on the development of texture.

Finally, the fact that the texture development in BSCCO/Ag tapes could follow from interfacial effects suggests that other materials which do not react with the BSCCO material could also constitute a suitable substrate. Gold could be a good candidate, because it has the same crystal structure and similar electronic configuration as silver.

The improvement in Jc for the thick films and tapes processed under a 10 T magnetic field is a consequence of the enhancement in the degree of texture. The fact that the employed processing temperatures are above the melting point of Bi-2212 lead us to suggest that the mechanism for alignment seems to be closely related to the early stages of growth of superconductor grains from the liquid. In this regime the grains are still surrounded by a liquid phase, and thus the magnetic energy will rotate the c-axis of the grains towards a direction parallel to the magnetic field. Additional growth of the grains occurs mainly perpendicular to the c-axis, but with the c-axis aligned parallel to the field. Thus, when the magnetic field is applied during the early growth of superconductor grains, a high degree of alignment is produced. As shown in Fig.7, grains of the order of 600 A can be highly aligned by the application of a 10 T magnetic field.

The reason for a J_c decrease with increasing thickness is probably due to the fact that for greater thicknesses, the number of grains not aligned with the magnetic field will more likely grow longer in the case of thicker films which deteriorates the process of alignment. In addition, it might be that as the thickness increases, there is a larger amount of grain boundary area, which if poorly connected, affect the J_c performance.

The model described here is only valid if the superconductor material is processed under a magnetic field at a temperature where there is a large fraction of liquid phase. In the case where the superconductor is never processed above the melting point in the presence of a magnetic field, or when the magnetic field is applied during the late stages of growth, an interaction energy between the grains needs to be considered.

Under these conditions, the degree of alignment achieved is a function of the distance and angle between the grains, and the grain aspect ratio.

The results from the last experiments confirm that a magnetic field is more effective in enhancing the texture development of Bi-2212 thick films during the solidification regime than during the grain growth process. In this case, although the application of a magnetic field was limited to 20 minutes, a J_c enhancement of approximately 50% was achieved in comparison with samples processed in the absence of magnetic field. Since preferred nucleation is negligible, crystal rotation under a magnetic field must be the mechanism leading to the crystal alignment. It can be noticed that the magnetic driving force is proportional to the volume of the crystal, thus it is expected that larger grains are more easily oriented. However other factors may hinder the orientation induced by the torque, such as thermal agitation, impingement with other crystals, the presence of impurity phases and the viscosity of the surrounding liquid.

4. References

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Figures

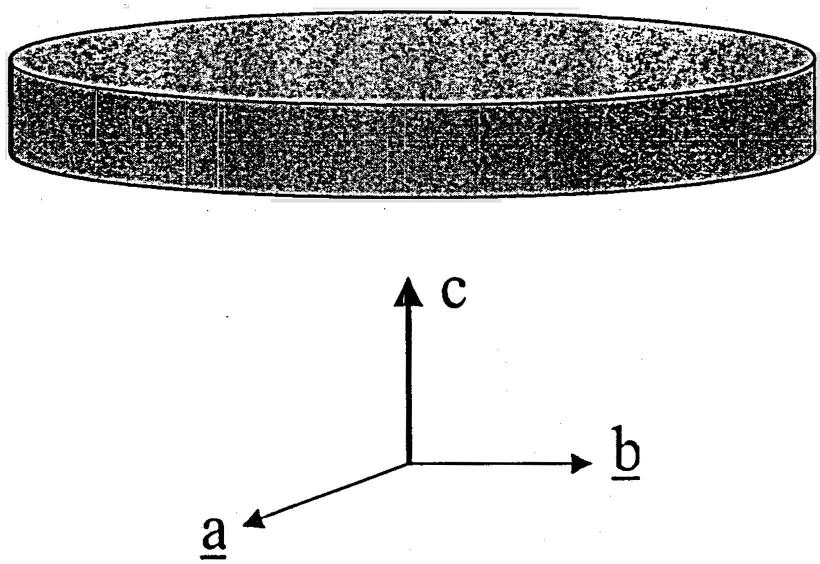

Figure 1: Schematic illustration of a Bi-2212 platelet-like grain. a, b and c are the crystallographic directions.

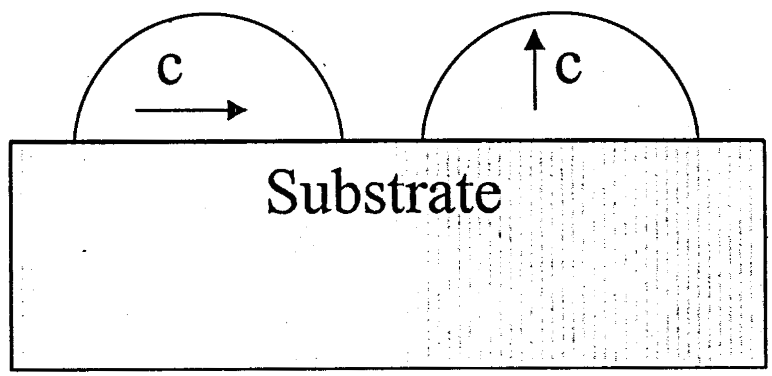

Figure 2: Random heterogeneous nucleation of Bi-2212 crystals with respect to the c-axis orientation.

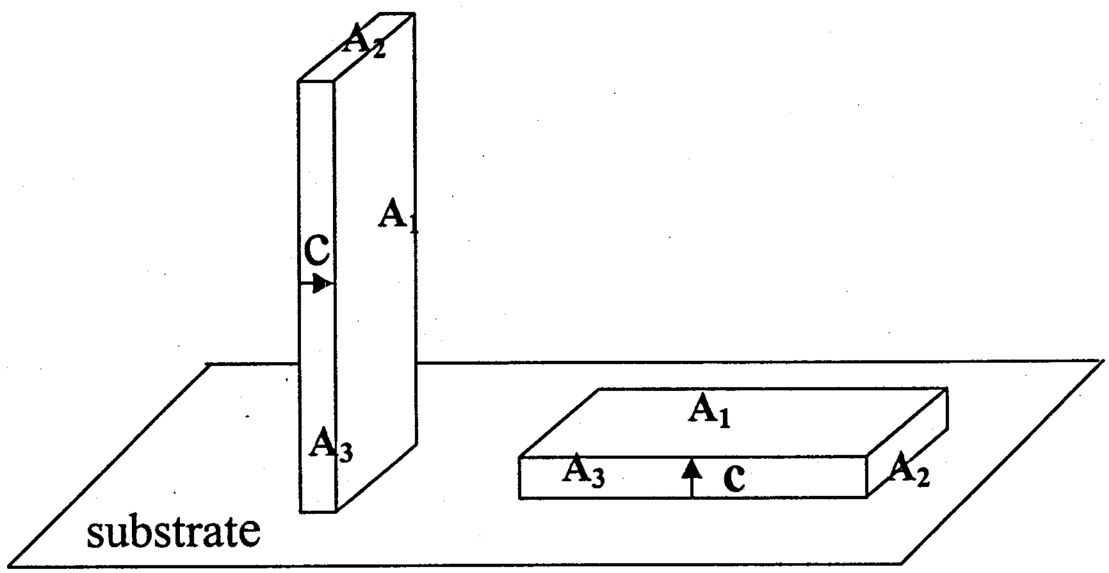

Figure 3: Anisotropic crystal growth of Bi-2212 nuclei with the c-crystallographic axis parallel and perpendicular to the interface.

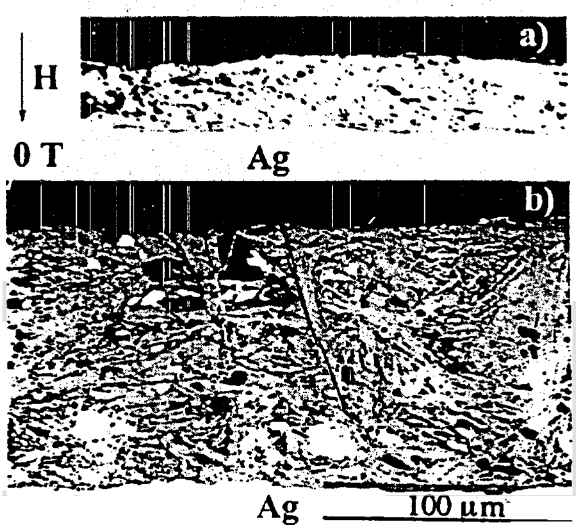

Fig.4 : SEM images of BI-2212 cross-sections melt-processed in the absence of a magnetic field.

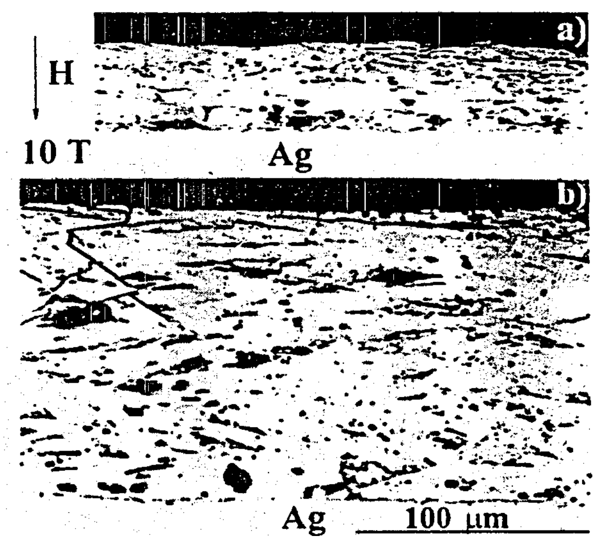

Fig.5: SEM images of BI-2212 cross-sections melt-processed under a 10 T magnetic field.

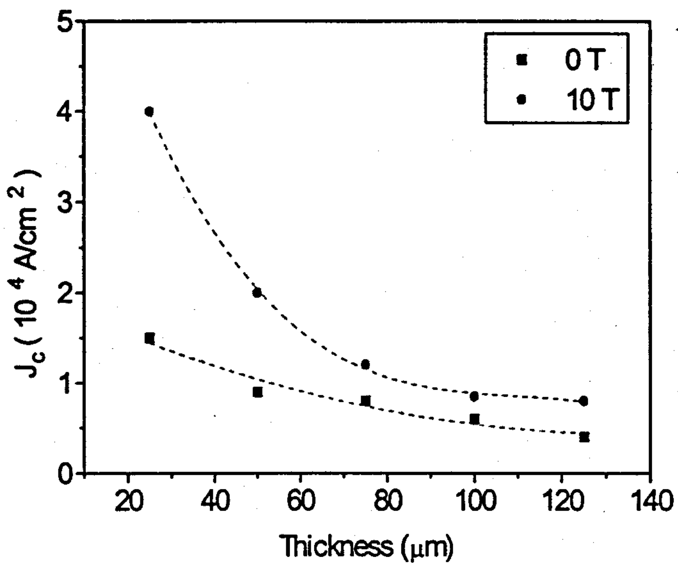

Fig.6: Transport current density at 4.2K, zero field, as a function of thickness for films processed under a 0 and 10 T magnetic field.

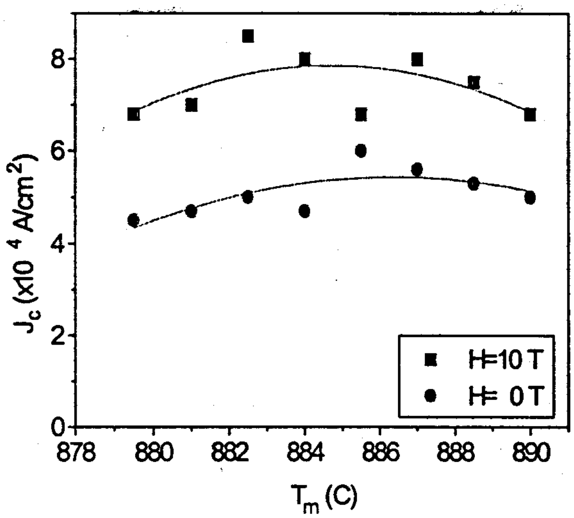

Fig.7: Maximum temperature Tm dependence of Jc at 4.2 K, zero field, for the tapes processed following thermal sequences under zero and 10 T magnetic fields.

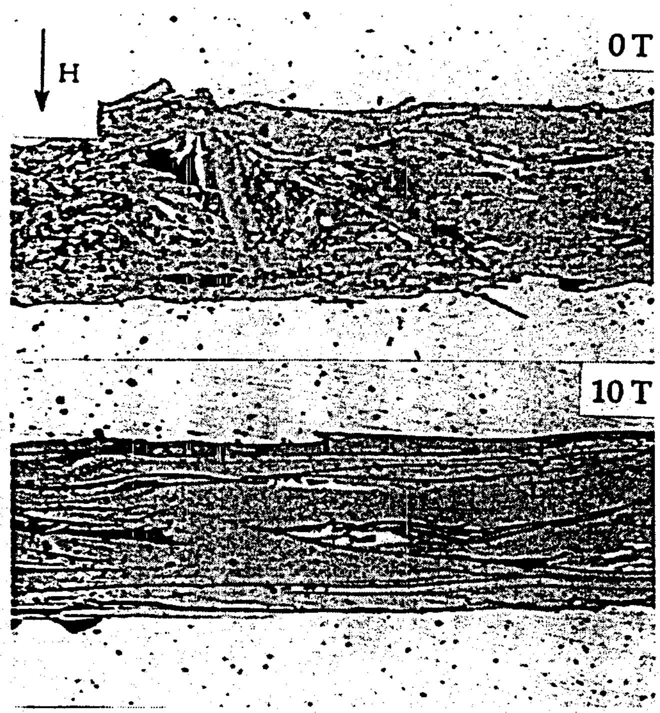

Fig.8: SEM images of cross-sections of Bi-2212 tapes melt-grown under a 0 T and 10 T magnetic field.

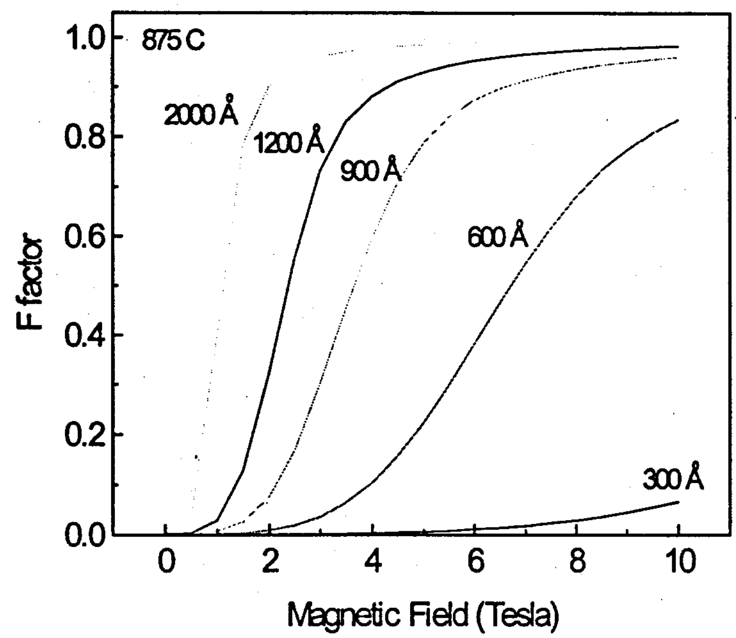

Fig.9: F factor as a function of magnetic field for various grain sizes. The processing temperature is 875 C.

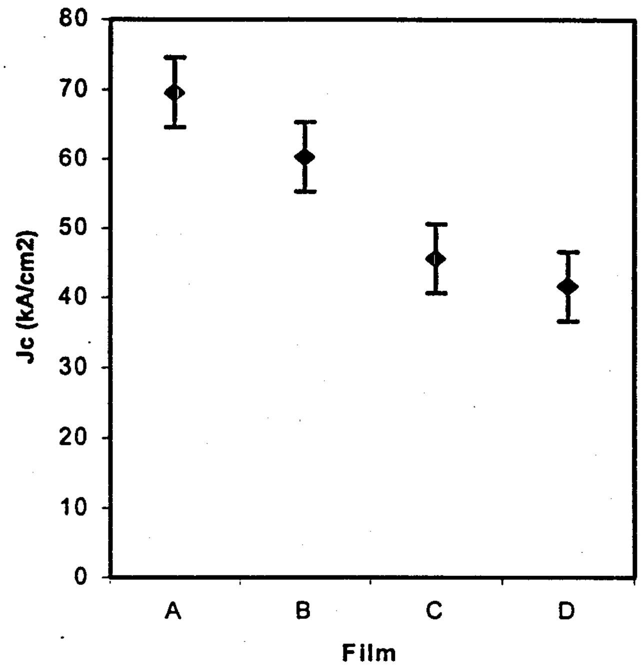

Figure 10: Average Critical Current Density (4.2 K, self field) of films A, B, C and D, calculated on the basis of three samples. Measurements were performed with the 4-probe DC method. A criterion of 1μV/cm was used to determine J_c.

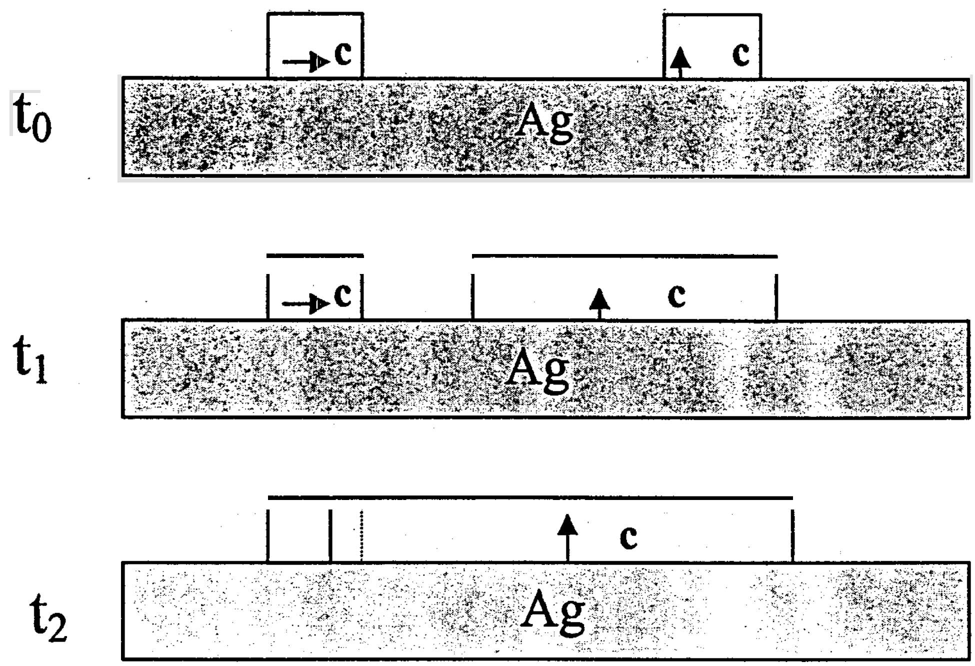

Figure 11: Sequence of events where BSCCO grains oriented with their c-axis perpendicular to the substrate will eventually consume crystals oriented with their c-axis parallel to the substrate. The symbols t_0-t_2 correspond to increased time intervals.