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Powder Synthesis and Fabrication of Ag-Sheathed Bi-2223 Tapes†

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An alternate processing route is described for the production of uniform, fine-grained precursor mixture for the fabrication of Bi-2223 tapes. The precursor powder was produced by aerosol spray pyrolysis of nitrate salts. It was found that the phase content of the powder is sensitive to several processing parameters including temperature and oxygen partial pressure. Bi-2223 tapes were manufactured using these powders by a powder-in-tube method. Supereonducting properties of these tapes are presented. A novel nondestructive transmission x-ray diffraction study showed that the texturing process of the superconducting phase is nearly complete by the end of very early stage of heat treatment. Pressing was found to piay little rote for the texturing process.

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Gaining control oi the phase conversion reaction by which a mixed phase powder of overall 2223 composition is converted. to an aligned 2223 microstructure is one of the central issues underlying the development of high critical current density conductors of 2223. It is well recognized that such control can not be achieved without a homogeneous starting powder with uniform chemical composition and morphology. Aerosol spray pyrolysis is a versatile technique for producing ceramic fine powders with uniform chemical composition and morphology. In this paper, we present results on the Bi-2223 tapes fabricated using powders produced by this technique.

A novel nondestructive transmission x-ray diffraction technique was used for determining the degree of texturing of superconductors in the c-axis. This technique has a clear advantage over the conventional x-ray technique performed on peeled samples. Peeling usually results in two samples with different thickness of the superconductor core. It is well knowm that the Bi-2223 is best textured at the Ag interface and the degree of texturing decreases for the superconductor located far from the interface [1]. Thus comparing peeled samples sometimes lead to confusing results. In addition, the poor sintered superconductor core at the early stage of heat treatment usually falls apart when the tape is peeled, making

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study difficult. In this study, samples produced at a wide range of heat treatment time as well as the as-rolled sample were carefully studied. The result leads to reexaminatlon of the role for mechanical deformation on the texturing process in the Bi-2223 tapes. The result also sheds some light into the formation mechanism of the Bi-2223 phase.

Powders with the norminal composition of $Bi_{1.8}Pb_{0.4}Sr_2Ca_{2.2}Cu_3O_x$ were produced by aerosol spray pyrolysis of nitrate salts. The experimental details have been described elsewhere [2,3]. All powders were produced in either air or N_2 atmosphere with the temperature set in the range of 700-780 "C.

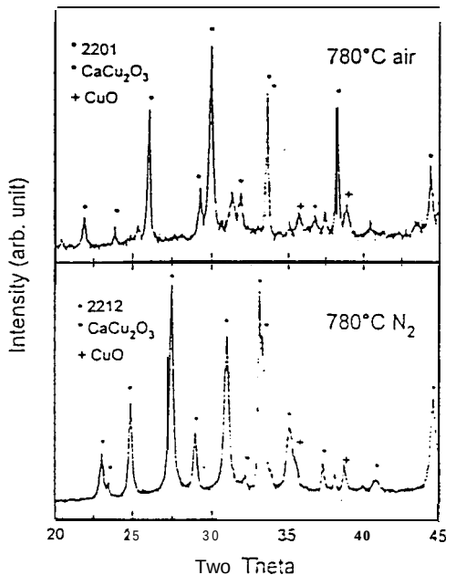
The tapes were produced by the well established oxide powder in tube (OPIT) technique. Samples with length 4-5 cm were cut from the tapes and heat treated in air at 845 °C with different duration period. The samples were uniaxially cold preessed 3 times at ≈ 2 Gpa at every 50 h interval of heat treatment. Transport current density J_c was measured by a standard four-probe technique using a *dc* method. The two voltage leads were 1 cm apart. The criterion for J_c was $1\,\mu V$ cm-l.

Conventional XRD was performed on peeled samples to determine the volume fraction of the Bi-2223 with respect to the Bi-2212 by comparing the integrated intensity of the $(0\ 0\ 10)_{2223}$ peak to the $(0\ 0\ 8)_{2212}$ peak. The transmission x-ray diffraction experiment was performed at the National Synchrotron Light Source at beam line X-27A. Ihe experimental details have been described elsewhere [3,4]. The high energy and high intensity x-ray makes it able to penetrate the silver sheath and probe the entire superconductor volume. The sample was positioned in such a geometry that the long axis of the tape is perpendicular to the incident x-ray. The rocking curves of the (2 2 0) peaks from both 2212 and 2223 phase we&analyzed to determine the half width at half maximum.

Figure l(a) and l(b) shows the XRD pattern of powder produced at 780 °C in air and N_2, respectively. No 2223 phase was detectet in either of the spectrum. For the powder produced in air, 2201 exists as the major phase; for the powder produced in N_2, 2212 domininates the spectrum. Other secondary phase were identified as CuO and $CaCu_2O_3$. For powders produced at 700 and 750 °C (not shown), the spectra look similar to Fig. l(a) for powders produced in air while the spectra were mixed with 2212 and 2201 for powders produced in N_2. It has been reported by many authors that best result is obtained when the starting powder is a mixture consisting of 2212 and (Ca, Cu)-rich secondary phases and the 2223 phase is synthesized "in-tape" [1,5,6]. The powder produced in Fig. l(b) was then chosen for making tapes for further study. Figure 2 is an SEM micrograph showing the morphology of this powder. The powder has an average particle size of $\sim 1\,\mu m$ and a flaky appearance.

Figure 3 shows the Bi-2223 phase conversion ratio as a function of annealing time. In this study, all samples were annealed at 845 °C in air. It was seen the Bi-2223 phase increases rapidly during the early stage, then increases slowly during the intermediate stage, and finally plateaus to a maximum value after prolonged annealing times. The origin of this Bi-2223 formation kinetics will be discussed in the future and is beyond the scope of the current study.

It was noted that for samples annealed for the same period of time (250 h), the sample undergoing the cold pressing process yields more Bi-2223 phase. Also, samples processed with intermediate pressing show higher J_c values than the unpressed counterparts. The



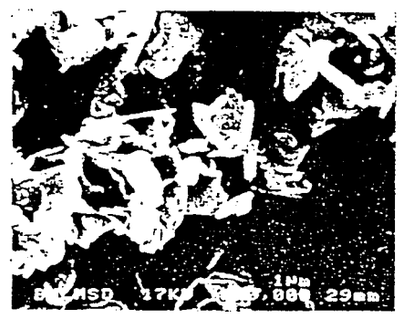


FIG. 1. XRD patterns of powder produced at 780°C in (a) air and (b) N_2. For the powder produced in air, Bi-2201 shows as major phase; in N_2, Bi-2212 is the primary phase.

FIG. 2. An SEM micrograph showing the fine and flaky morphology of the powder produced in N_2.

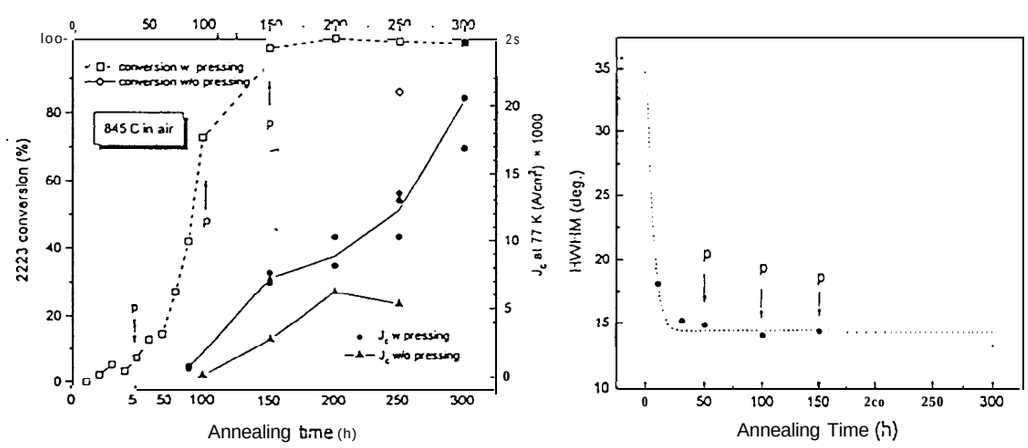


FIG. 3. Evolution of the 2212-to-2223 transformation and J_c *as* a function of annealing temperature. A "p" indicates the sample was cold pressed at the end of the specified annealing stage.

FIG. 4. HWHM (half width at half maximum) of the (2 2 0) peak from both 2212 and 2223 phases shoring the evolution of c-axis texturing for samples heat treated at different stages. A "p" also refers to cold pressing as in Fig. 4.

role of such mechanical deformation on the development of the final tape product has been proposed by Parrell et. al [7]. Def ormation is thought to break up the larger second-phase particles and to densify the superconductor core, such that the subsequent rcaction to 2223 is faster. It is also proposed that the c-axis texture of the grains is improved by these additional deformation step. To test this hypothesis, the result of the transmission x-ray diffraction rocking curve measurements is illustrated in Fig. 4. It is clearly shown that the texturing process of the superconductor is complete at the very early stage of annealing. The texturing is essentially unchanged after the first 30-50 h of heat treatment. The cold psessings employed later clearly did not change the overall texturing of the superconductor in c-axis direction. Since very little Bi-2223 phase forms at this stage (Fig. 3), the texturing process of the supereonductor was carried out by realigning the Bi-2212 grains. The texturing mechanism of Bi-2212 grains in the silver tape is still not clear. With the current result, we demonstrate cold pressing, and we believe mechanical deformation in general, have no effect on the texturing process of the Bi-2223 phase. However, judging from the HWHM value of the as rolled sample (time zero), the mechanical deformation (rolling in this case) seems, able to align the 2212 powder to some extent during shape forming of the tape.

It is generally believed that the 2223 phase forms by a reaction between the 2212 phase and a eutectic liquid associated wiwh the (Ca, Cu)-rich phase [8,9]. However, a generally agreed microscopic model has yet to be developed. From Figs. 3 ant 4, it was demonstrated that the Bi-2223 phase at the end of the annealing cycle has essentially the same texture as the Bi-2212 phase st the early stage of annealing. This suggests that the final .Bi-2223 phase in the tape inherits its microstructure from the Bi-2212 phase at the early heat treatment stage. This makes Bi-2223 formation mechanism such as nucleation and growth of Bi-2223 embryos less likely. Our previous TEM study suggests that the Bi-2223 is formed by the intercalation of a single CuO/Ca bi-layer, one at a time, into the CuO/Ca/CuO blocks of the Bi-2212 structure; sometimes the random insertion of the CuO/Ca bi-layer results in a distinct phase, Bi-4435 [9]. A detailed study on the formation mechanism of the Bi-2223 phase is still underway.

It has been demonstrated in this study that aerosol spray pyrolysis is a suitable technique for producing homogeneous fine powders for fabrication of Bi-2223 tapes. The phase content in the powders was found to be sensitive to temperature and oxygen partial pressure. The desirable 2212 and (Ca, Cu)O_x mixture forms at low oxygen pressure atmosphere. By using synchrotron radiation, we demonstrated a non-destructive transmission x-ray diffraction rocking curve study is feasible for studying the c-axis texturing of the superconductor embedded under the silver sheath. The texturing process of the Bi-superconductor was found to be complete at the very early stage of heat treatment. Mechanic1 deformation aids 2212 alignment during the shape forming stage of the tape, improves both grain connectivity and 2223 formation kinetics, but plays little role in the texturing of 2223 grains.

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