case of 18 kN, a considerable portion of the particles are mechanically broken. The structure is condensed nearly without cavities. It might be useful to prepare pellets of high density, as the diffusion coefficient of tritium in Li_2SiO_3 or Li_4SiO_4 is on the order of 2 magnitudes higher than in the other lithium compounds.[3]

$Li_2SiO_3 \cdot nH_2O$ is X-ray amorphous.[1] A considerable portion of the powder has already been crystallized after powder calcination at 600°C. Material sintered at a temperature of 1100°C has crystallized completely (Fig. 2).

Conclusions

The preparation processes including the spray-dry technique[2] can be recognized as an approved method for preparing fine powders of lithium meta- and orthosilicate. In contrast to standard procedures,[4,5] an inherent source of chemical impurities has been excluded.

With increasing compacting pressure from 10 kN, the particles were highly compressed, the cavities dissolve, and the structure becomes relatively homogeneous in the case of spray-dried lithium silicate powders.

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Texture Development in $YBa_2Cu_3O_x$ by Hot Extrusion and Hot-Pressing

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Pronounced textures have been found in hot-extruded and hot-pressed $YBa_2Cu_3O_x$ powders. The basal plane is preferentially oriented normal to the maximum compression axis in hot deformation. Thus, the extrusion texture of these ceramics should facilitate maximum current-carrying capability along the extrusion axis, suggesting hot extrusion or drawing as a favored method of producing superconducting wires.

Several research groups have recently demonstrated a very high critical current density in the high-T_c $YBa_2Cu_3O_x$ compound using nearly-single-crystal thin films.[1,2] These results confirm that the structural anisotropy in this compound dictates its transport properties and that only the basal plane can sustain a very high current density. It is now obvious that precise textural control must be exercised in polycrystalline superconducting components to achieve the optimal current density in their application. Hot-deformation practices such as hot extrusion and hot-pressing are known to produce textures in both cubic and noncubic polycrystalline metals and ceramics. The characteristic texture obtained depends on the deformation method and materials. We report here an exploratory study of hot extrusion and hot-pressing using $YBa_2Cu_3O_x$ powders. As will be shown later, the resultant texture from hot extrusion is of particular interest, in that it favors the alignment of the superconducting path along the extrusion axis.

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Experimental Procedure

Starting powders of orthorhombic $YBa_2Cu_3O_x$ were prepared using several methods, including solid-state reaction and coprecipitation. Hot deformation was conducted using cylindrical dies made of alumina, in an environmental chamber of controlled atmosphere. An extrusion ratio of 3 in area and a diameter-to-height ratio of 4 in hot-pressing were used. The experimental setup is schematically shown in Fig. 1. Typically, the powder samples were heated in air with a small preload, then deformed between 850° and 950°C, under a constant stress between 10 and 40 MPa. After hot deformation, the samples were either cooled in air or cooled in argon, in order to control the tetragonal-to-orthorhombic transformation.[3–7] Samples were examined by polarized reflected light microscopy and scanning electron microscopy. Textures were evaluated by X-ray diffractometry (XRD) using Cu$K\alpha$ radiation, by comparing the diffracted intensities of various peaks along transverse and longitudinal cross sections. Several samples were further annealed in argon or in oxygen and similarly characterized. In addition, densities and microhardness were measured. Presintered pellets were also hot-extruded and hot-pressed in this study.

Results

Phase Identification

Although the starting powders are orthorhombic, they are expected to become tetragonal above 750°C in air,[3–7] i.e., before hot deformation commences. X-ray diffractometry indicates that all the hot-deformed samples are tetragonal whether they were cooled in air or in argon. Thus, no phase transformation occurs during cooling. Transformation to the orthorhombic phase can be achieved if these samples are subsequently annealed in oxygen. The indexing convention used in this paper is, for tetragonal, $a = b < c/3$, and for orthorhombic, $a < b = c/3$.[7]

Textures

The XRD pattern of a hot-extruded sample is shown in Fig. 2. In the as-cooled (tetragonal) state, (006), (103), and (013) reflections were found depressed on the plane transverse to the extrusion axis but enhanced on the plane parallel to the extrusion axis. Thus, the **c** axis lies preferentially normal to the extrusion axis. This texture was found to be preserved in the superconducting orthorhombic phase formed by post-hot-deformation heat treatment of 900°C in flowing oxygen followed by slow cooling. This can be seen from the XRD pattern shown in Fig. 2. Since the basal plane is the superconducting plane, we expect the extrusion direction to coincide with the most favorable current direction in this texture.

The texture of hot-pressed samples was also investigated. The XRD pattern of a hot-pressed sample is shown in Fig. 3. In the tetragonal state, (006), (103), and (013) reflections were found enhanced along the hot-press plane, and vice versa on the longitudinal plane. Thus the **c** axis is preferentially aligned perpendicular to the hot-press plane. A similar texture was recently reported after sinter forging of $YBa_2Cu_3O_x$ powders,[8] which lacks the transverse confinement that hot-pressing provides. For this texture, the resistivity in the normal state along the plane was found to be 1 order of magnitude smaller than that perpendicular to the plane.[8] The maximum current density in the superconducting state is expected to flow along the plane.

Taken together, the above results from hot extrusion, hot-pressing, and hot-forging suggest that *the basal plane is preferentially oriented normal to the maximum principal compression axis in hot deformation* (see Fig. 1).

Kinetics of Hot-Pressing

Kinetics of hot-pressing were determined in this study. Essentially, we found that a pressure of 40 MPa and a temperature of 900°C are required for hot deformation in closed dies, both in hot-pressing and in hot extrusion, in order to facilitate densification in approximately 1 h. Some data of densification are shown in Fig. 4 to illustrate this point. Note that after 1 h, the densification rate is very low for all cases, as can be seen in Fig. 4(*B*). It seems that the closed die confinement in hot-pressing helps to effect densification, but demands higher pressure. The steep temperature dependence above 850°C is probably

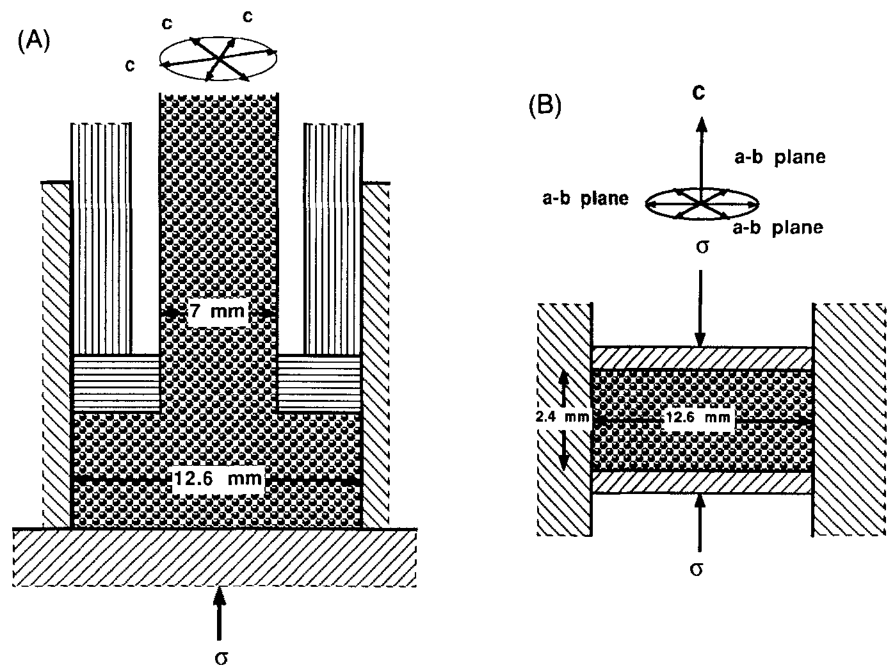

Fig. 1. Experimental setup of (*A*) hot extrusion and (*B*) hot-pressing, with the expected textures shown schematically.

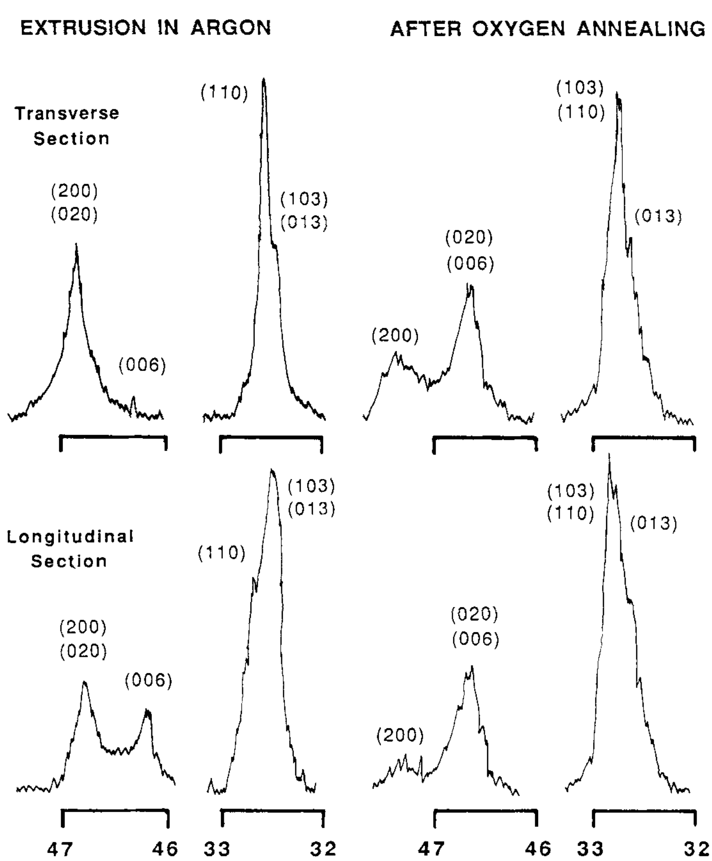

Fig. 2. XRD intensity of {200} and {103} reflections (using Cu$K\alpha$ radiation) along transverse (top) and longitudinal (bottom) cross sections of hot-extruded $YBa_2Cu_3O_x$: (left) as-extruded, tetragonal phase; (right) annealed in oxygen, orthorhombic phase.

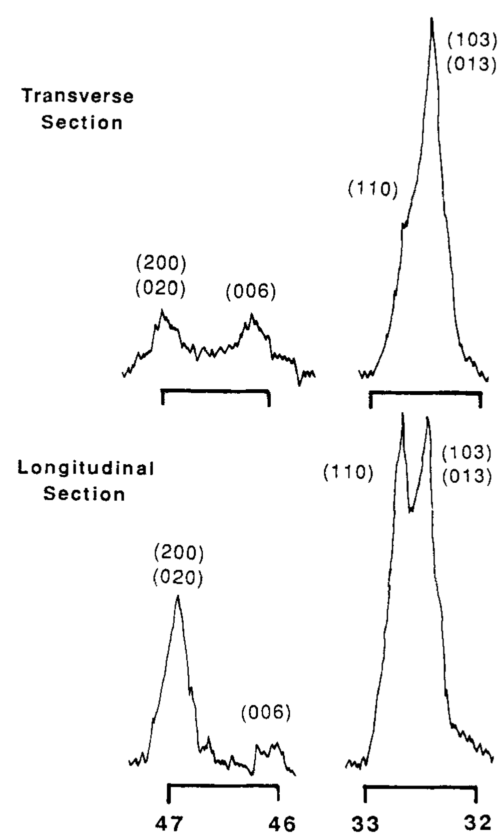

Fig. 3. XRD intensity of {200} and {103} reflections along transverse (top) and longitudinal (bottom) cross sections of $YBa_2Cu_3O_x$ hot-pressed (900°C, 40 MPa) in air. Tetragonal phase.

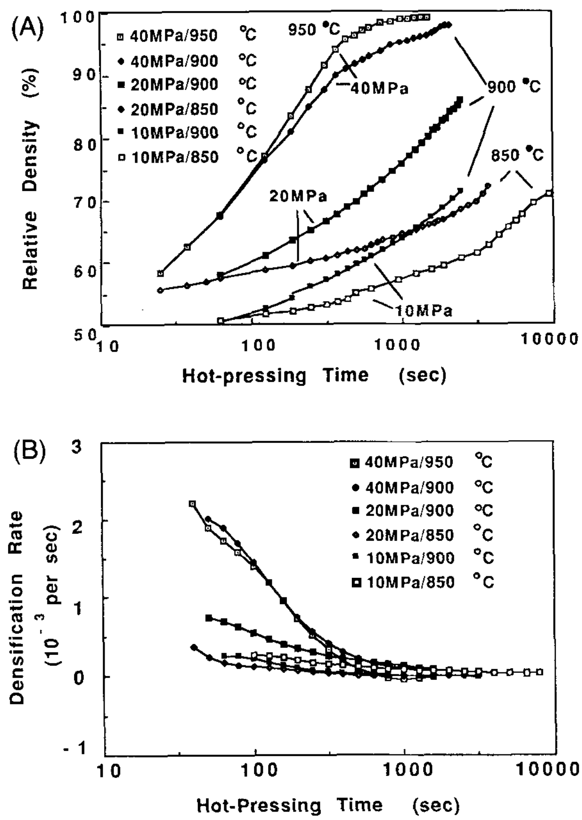

Fig. 4. (*A*) Densification curves in hot-pressing; (*B*) densification rates.

closely related to the formation of a liquid phase in this system.[9,10] Further details of hot-pressing kinetics will be reported elsewhere.

Microstructure and Hardness

Hardness of relatively high-density samples obtained from hot-pressing were measured on both the transverse and the longitudinal cross sections. Hardness was found to be higher by 10% along the longitudinal cross section (H_v=5.3 GPa on transverse cross section and 5.8 GPa on longitudinal cross section). These values are 30% lower than that reported for single crystal along the basal plane (8.7 GPa)[11] but are much higher than typically reported for sintered samples of lower density.[12,13]

As noted above, hot deformation between 850° and 950°C is believed to be conducted in the tetragonal-phase regime, and apparently no transformation occurs during cooling. The microstructure of the as-deformed samples consists of many elongated grains reminiscent of orientation textures caused by hot deformation. Twinning was not observed in this state. After subsequent annealing and the resultant transformation into the orthorhombic phase, both grain growth and twinning have taken place. Despite these changes, by careful control of annealing conditions, texture can be retained as shown previously in Fig. 2.

Preliminary experiments in this study indicate that no strong texture was produced by hot-deforming presintered pellets and that textures could be lost because of excessive grain growth in subsequent annealing. Further study is required to clarify these aspects.

Conclusions

Pronounced textures have been found to develop during hot extrusion and hot-pressing of $YBa_2Cu_3O_x$ powders. The basal plane is preferentially oriented normal to the maximum compression axis in hot deformation. In particular, the extrusion texture of this ceramic material provides a favorable grain orientation such that the superconducting planes are parallel to the extrusion axis. The texture can be retained during subsequent annealing, which may effect a reversible tetragonal-to-orthorhombic transformation. This finding suggests that judicious exploitation of such texture could result in mechanically drawn or extruded wires with favorable superconducting current-carrying capability.

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