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Consolidation of YBaCuO powders by shock loading

N C SONI, RAM PRASAD, ASHOK MOHAN, M D VORA, S BANERJEE, C V TOMY*, S K MALIK*, H S YADAV† and K R K RAO†

Bhabha Atomic Research Centre, Trombay, Bombay 400 085, India
*Tata Institute of Fundamental Research, Bombay 400005, India
†Explosives Research and Development Laboratory, Pune 411 021, India

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Abstract. Superconducting oxide-copper monoliths have been fabricated by shock-wave loading using cylindrical and plane geometries. Bulk densities up to 96% T.D. have been obtained by varying detonation parameters. The superconducting properties, interface bonding and microstructure of the compacts have been evaluated.

Keywords. High temperature superconductors; shock-wave compaction; microstructure.

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1. Introduction

One of the primary problems in the fabrication of $YBa_2Cu_3O_7$ powder into high density high temperature superconducting (HTSC) bodies arises from the fact that the difference in temperatures of calcination and sintering is very small. For treatment, the powder mixture has to be repeatedly heated in the region of 1150 K and reground. On the other hand, sintering cannot be done at temperatures above 1220 K where the material starts reacting with alumina container and partial decomposition of the oxide begins to occur. The difference between the calcination and the sintering temperature is, therefore, restricted to about 70 K.

Apart from the requirement of having high Meissner fraction, high density is important as the porous material tends to degrade rapidly due to atmospheric exposure. Critical current density is also likely to increase in denser samples because of reduction in weak-link effects.

One possible approach to achieving high density could be to prepare active powders by wet chemical processes like sol-gel etc, thereby attaining nearly complete solid solution as well as sintering at comparatively lower temperatures. The other approach, which has been used in the present work is to consolidate the powders by employing a technique like shock compaction (Johnson *et al* 1988; Murr *et al* 1987a, b, 1988).

Shock-wave compaction is a dynamic process in which the powder aggregate is compressed by the travelling shock-wave generated by a suitable explosive. This method has several potential advantages over the conventional cold compaction technique for the processing of superconducting powders. High density oxide-metal monoliths can be obtained which can be used for subsequent extrusion, wire drawing

or cold rolling into usable shapes. The rise in temperature of the bulk during the compaction process is expected to be very limited thus obviating the need for further heat treatments for regeneration of superconductivity. The metal cladding provides protection from possible degradation due to atmospheric exposure. A strong possibility also exists for increase in defect density in the compacted material due to the transient nature of the shock-wave. If one was to draw a parallel from alloy superconductors, a higher defect density could lead to an increase in critical current density of bulk material.

Usually oblique shock-waves are employed for compacting powders (Prümmer 1983). In one of the geometries, shock-wave is produced in the powder by oblique impact of an explosively accelerated plane flyer plate. In another geometry, the shock-wave is generated by inward motion of the wall of a metal tube, containing powder which is accelerated by symmetrical detonation of the explosive placed around it. In the present series of experiments both plane and cylindrical geometries have been used. The density of the compact and its uniformity depend on various parameters (Kostyukov and Kuzmin 1986) like characteristics of the explosive used, its thickness, the geometry of the flyer plate and the nature of the powder.

2. Factors affecting compaction

On ignition of the detonator as shown in figure 1a, an axisymmetrical detonation wave propagates in the explosive along the length of the tube. The pressure P of this detonation wave is given as,

$$P = \frac{\rho_x D^2}{\gamma + 1} \tag{1}$$

ρ_x = density of the explosive, D = detonation velocity and γ = specific heat ratio of detonation products.

The pressure of detonation products moves the wall of copper tube and thereby generates a converging shock-wave in the powder. During propagation, some energy

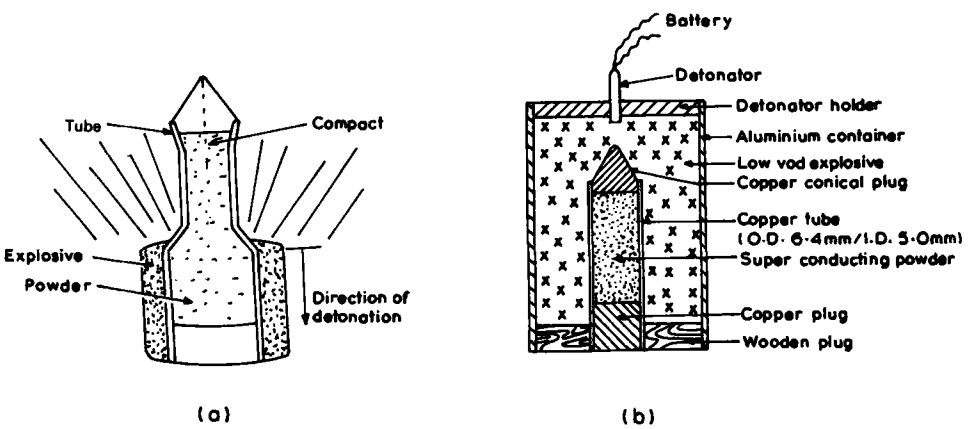

Figure 1. Explosive powder consolidation in a cylindrical configuration. (a) Schematic representation of the process. (b) The assembly before detonation.

of shock-wave is attenuated in crushing and plastically deforming the particles. As the shock-wave proceeds towards the axis of the cylindrical sample, its pressure and particle velocity increase because of convergence. These two processes control the final shape of the shock-wave in the powder. It is reported (Prümmer 1983) that the powder is compacted to uniform density over the cross-section of sample, if the shape of the shock-wave is conical. This occurs when attenuation and convergence of shock-wave are balanced. In plane geometry experiments, however, the impact of flyer plate generates shock-wave with an approximately plane front. Under the conditions of steady state, the state of the powder behind the shock front can be obtained from Rankine-Hugoniot relations which represent the conservation of mass, momentum and energy as

$$\rho_0 U_s = \rho(U_s - U_p) \tag{2}$$

$$P - P_0 = \rho_0 U_s U_p \tag{3}$$

$$E - E_0 = \tfrac{1}{2}(P + P_0)(V_0 - V) \tag{4}$$

where U_s is the velocity of the shock-wave, U_p the particle velocity, ρ_0 the initial density, ρ the density after passage of shock-wave front. V, P and E are the specific volume, pressure and internal energy with subscript '0' referring to the initial state before the passage of the shock-wave front.

3. Experimental

The $YBa_2Cu_3O_{7-y}$ powder was prepared by solid state reaction of component oxides/carbonates. The mixture was repeatedly calcined in the temperature range of 1125–1175 K interspersed with grindings, the final calcination being in flowing O_2 atmosphere. The powder was then dry ground in planetary ball-mill and the average particle size obtained was about 3 μm.

Figure 1b shows the experimental set-up used for compaction of the superconducting powder by axisymmetrical shock-wave loading. It consists of a copper tube of 0·70 mm thickness, 70 mm length and 6·4 mm OD which is filled with HTSC powder and closed at both the ends by copper plugs. It is surrounded by an explosive of low detonation velocity and uniform thickness. In order to achieve axisymmetrical initiation of the explosive, a solid copper cone is kept in contact with the copper plug at the initiating end of the explosive. The explosive column above the solid cone is kept sufficiently long so that the detonation wave gets stabilized before it reaches the powder.

In order to generate a shock-wave in powder by impact of a flyer plate, the experimental set-up shown in figure 2a has been employed. The oxide powder was filled in an annular cavity (ID = 15 mm, OD = 24·9 mm and height = 1·1 mm) or a rectangular cavity (26·25 × 10·2 × 1 mm) in the copper base plate of 197 × 147 × 19·3 mm. The copper plungers used were of 1·3 and 2·85 mm thickness respectively, which in turn were obliquely impacted by an explosively accelerated copper plate of 1·5 mm thickness. The length and breadth of this plate were equal to those of the base plate. Compaction experiments were conducted on all these geometries with varying initial packing density. Velocity of detonation and shock-wave pressure varied by

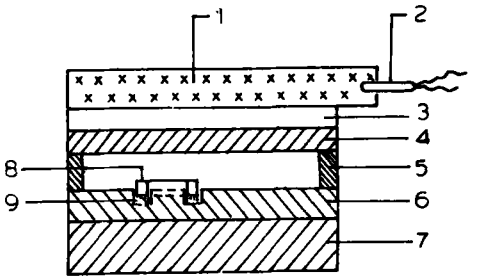

Figure 2a. Plane geometry assembly for explosive compaction of ring shaped specimens. 1. Explosive in tray, 2. Detonator, 3. Rubber buffer, 4. Copper flyer plate, 5. Stand off spacers, 6. Copper parent plate with annular groove, 7. M.S. anvil, 8. Copper annular ring (plunger) and 9. Superconducting powder.

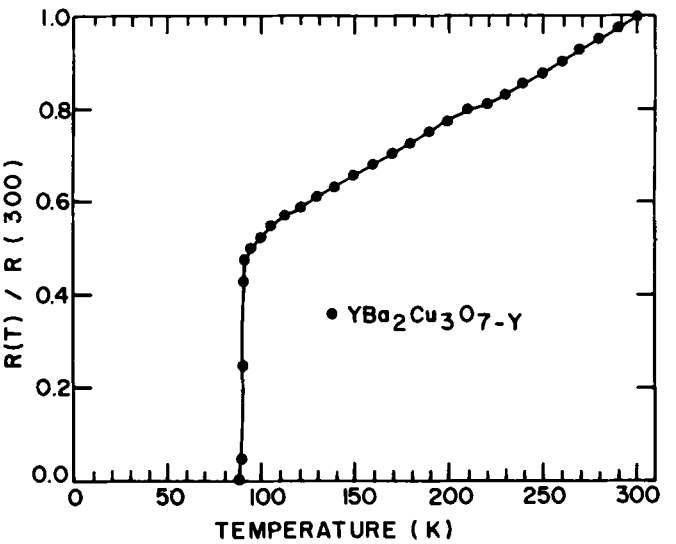

Figure 2b. ρ vs T curve for explosively bonded sample.

changing the nature and the thickness of explosive to obtain different explosive to powder mass ratio (E/M).

4. Results and discussion

Table 1 shows the results obtained with the cylindrical geomety. The densities of the oxide compacts were measured by liquid displacement method after machining out the copper casing. It was observed that good compaction was achieved only with explosive of low velocity of detonation (VOD = 2·3 mm/μs). High VOD explosives (3 mm/μs) were not found to be suitable. A variation in initial packing density from 3·3 to 3·5 gm/cc did not influence the final density attained which was found to be about 96% of the theoretical density (TD). Using an explosive of VOD 2·3 mm/μs several compacted samples with lengths up to 80 mm have been obtained.

Table 1. Parameters for expolsive compaction of superconducting powders.

Initial density of the powder (g/cc)	Copper tube dimensions OD/ID/length (mm)	Explosive VOD (mm/μs)	Final density of the compact (g/cc)	Remarks
3·27	5·6/2·5/60·7	3·0	—	No compaction
3·79	5·6/2·5/60·7	3·0	—	No compaction
3·38	6·4/5·1/80	2·3	6·10	Compacted
3·33	6·4/5·0/70	2·3	6·10	Compacted
3·51	6·4/5·0/70	2·3	6·12	Compacted

In plane geometry experiments, ring and wafer shape specimens were obtained. Being very thin, these samples could not be decladded. They were then sectioned and polished. Their behaviour during polishing gave the impression that the consolidation was not as good as in the cylindrical samples.

Four-probe dc resistivity measurements were carried out on the decladded cylindrical samples. The T_c was found to be between 90–91 K which is similar to that found in the cold pressed and sintered samples prepared from the same starting powder. ρ vs T curve for the explosively bonded sample is shown in figure 2b. Thus no change in T_c has been observed as a result of shock compaction.

Metallographic examination of the cross section of the shock compacted cylindrical sample was carried out to characterize the compacted material with respect to the size, the morphology and the internal structure of the grains, the porosity distribution and the structure between the copper sheath and the oxide core. The results of the microstructural examination can be summarized as follows:

(a) The bonding at the interface between the oxide core and the copper sheath was found to be very good (figure 3). Only at some localized areas along the circumference, a thin interface layer could be detected (figure 4).
(b) The wavy interface characteristic of explosion bonded materials was encountered only in some portions of the circumference (figure 3).

Figure 3. Perfect bonding between the oxide core and the copper tube. The waviness of the interface may be noted.

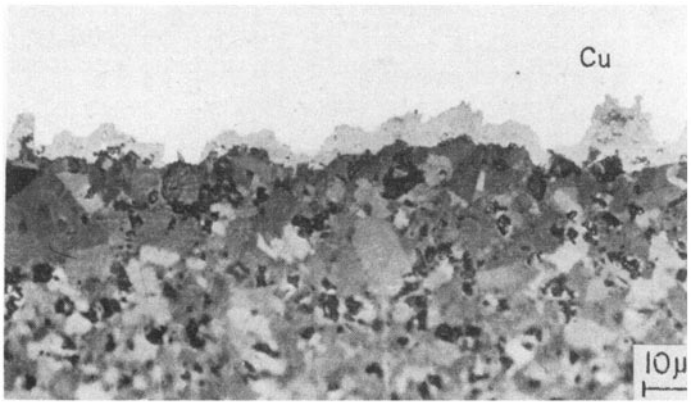

Figure 4. A thin interface layer following the contour of compacted material noticed in some regions.

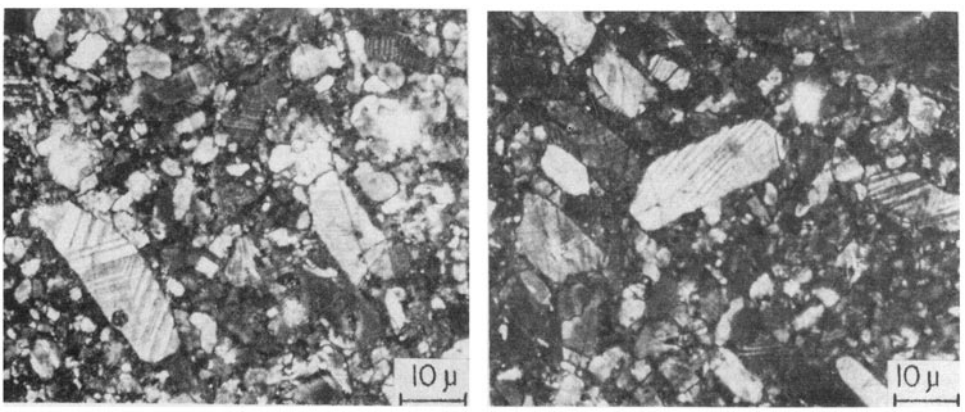

Figure 5. Large grains of HTSC phase in shock compacted material showing a high density of twins.

(c) The most remarkable observation was that some of the grains of the shock compacted HTSC phase were as large as about 30 μm (figure 5), a size which was several times larger than that of the starting powder particles. The comparison between the grain size of the starting powder and the compacted material, as depicted in figure 6 clearly establishes that grain growth has occurred to a significant extent during the explosive compaction process.

(d) The large grains present in the shock compacted material show nearly equiaxed shape with boundaries separating adjacent grains. Evidence of local melting which results in cellular or dendritic structure at the boundaries (Gourdin 1986) between powder particles was not noticed.

(e) Twinning was encountered much more frequently in the grains of the shock compacted material (figure 5) than in the microstructure of the particles of the starting powder.

(f) The porosity distribution in the compacted material was found to be non-uniform,

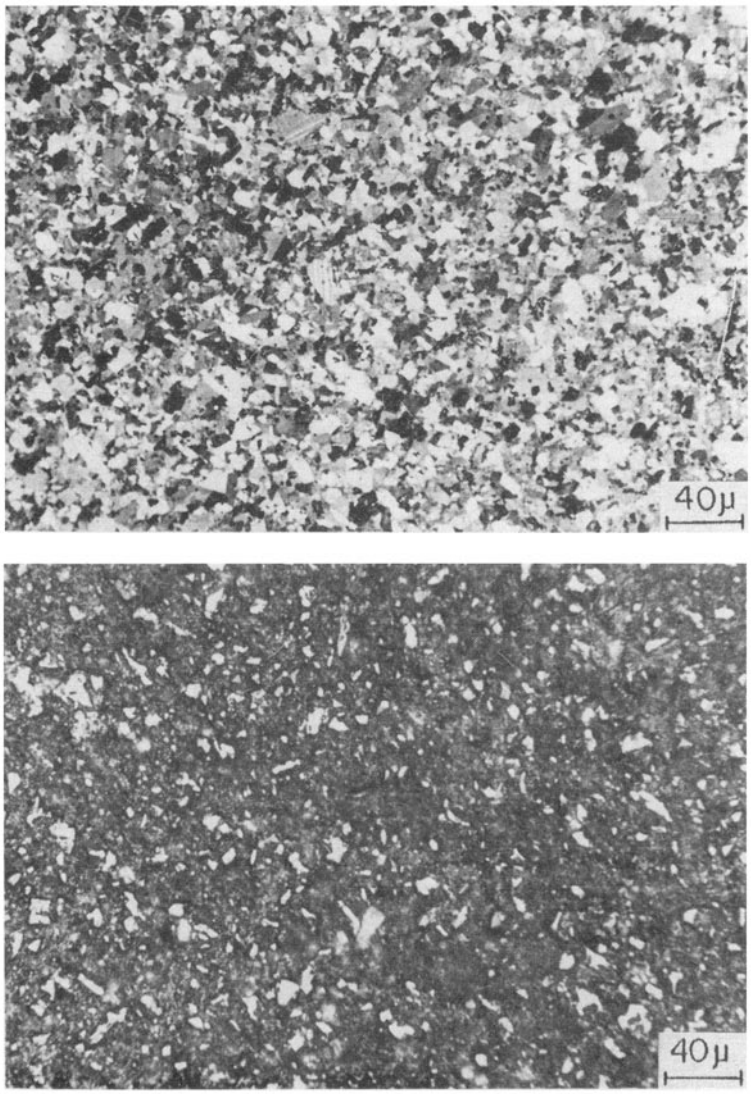

Figure 6. **(a)** The grain structure of the material in the as explosion compacted condition. **(b)** The microstructure of powders are revealed from a section of powder particles embedded in an epoxy matrix. A comparison of the size of grains of the HTSC phase before and after compaction clearly reveals that a significant growth of the grains have occurred spontaneously during the compaction process.

the volume fraction of the porosity decreasing from the periphery to the centre (figure 7).

Metallographic studies on the shock compacted HTSC powder did not show any sign of melting at the inter particle surfaces. However, grain development and grain growth had occurred to such an extent that the morphology of the initial structure was totally obscured. This observation under normal conditions points to the fact that there has been a substantial rise in temperature in localized regions but not to the extent to cause melting.

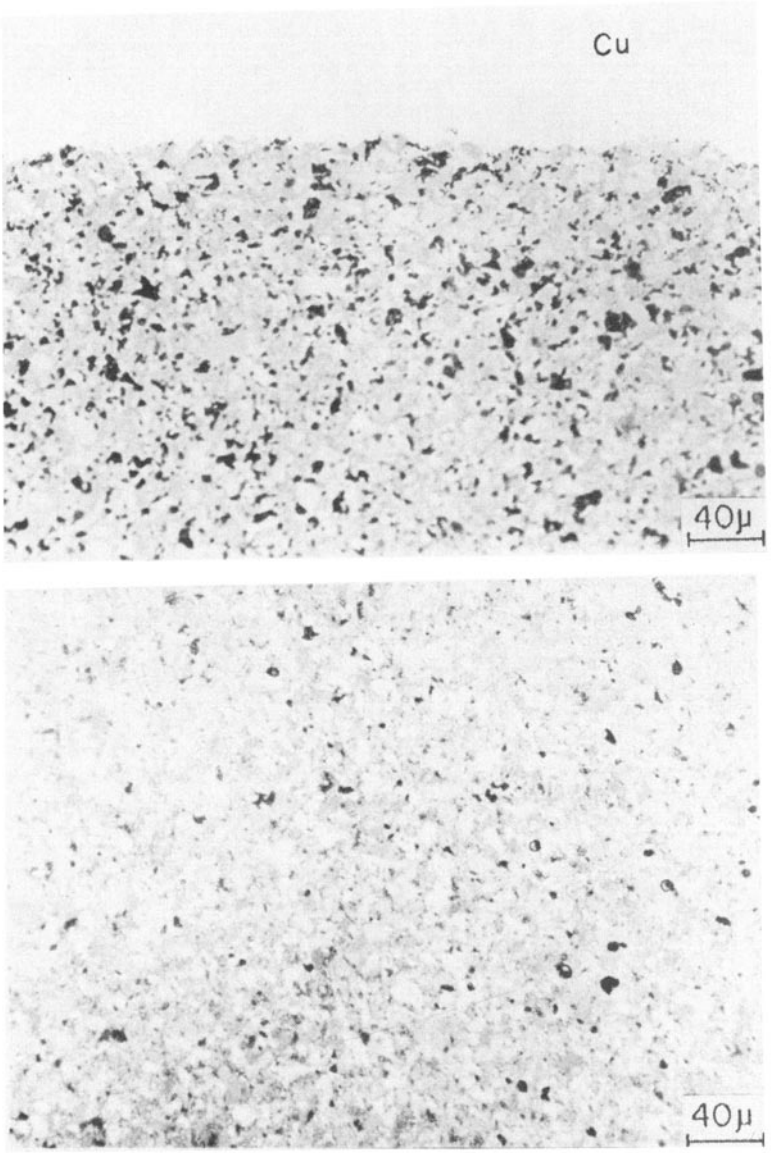

Figure 7. The distribution of porosity (seen in the micrographs as dark patches) in the compacted material near the periphery (**a**) and the centre (**b**) of the sample

It has been reported that when $YBa_2Cu_3O_{7-y}$ is subjected to thermal treatments and mechanical treatments like extrusion and wire drawing, it tends to lose its superconductivity. Very often, a further heat treatment in oxygen can also restore these properties. Similarly, experiments on deposition of thick films on Cu substrate have indicated that $YBa_2Cu_3O_{7-y}$ tends to lose oxygen and becomes non-superconducting. In the shock-wave compaction experiments, no such effects were observed although copper casings were used. No change in T_c was observed as stated

above and no subsequent heat treatment was required. This seems to be an important advantage of the shock-compaction process.

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