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SHOCK COMPACTION OF HIGH-T_c SUPERCONDUCTORS

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We present the results of shock compaction experiments on high-T_c superconductors and describe the ways in which shock consolidation addresses critical problems concerning the fabrication of high J_c bulk superconductors. In particular, shock compaction experiments on $YBa_2Cu_3O_7$ show that shock-induced defects can greatly increase intragranular critical current densities. The fabrication of crystallographically aligned $Bi_2Sr_2CaCu_2O_8$ samples by shock-compaction is also described. These experiments demonstrate the potential of the shock consolidation method as a means for fabricating bulk high-T_c superconductors having high critical current densities.

I. INTRODUCTION

The shock-consolidation of high-T_c powders has many features which make it an interesting and promising method for fabricating bulk high-T_c samples having high critical current densities[1]. In the shock-consolidation method, the energy and heating are largely localized to the surfaces of the powder particles, the particle interiors remaining relatively cool and stable[2,3]. Thus, if single-crystallite powder particles are used, shock-consolidation

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processes the grain boundaries in a manner distinct from the grain interiors, and thus enables a degree of control over the bonding process not possible with sintering.

Shock-processing can also enhance the intragranular critical current density of high-T_c superconductors through the introduction of high densities of shock-induced dislocations and other defects which act as flux-pinning sites[4]. Because dislocations perturb the lattice on a length scale comparable to the superconducting coherence lengths of high-T_c superconductors, they are effective at pinning magnetic fluxoids and increasing intragranular critical current densities without reducing the volume of superconducting material.

We describe here the results of our shock experiments on various high-T_c superconductor samples. These experiments were performed with two-stage light-gas guns at Lawrence Livermore National Laboratory (LLNL). Our shock experiments are characterized by small quantity samples (≈ few hundred mg) shocked under carefully controlled conditions, with the starting powder density, the powder particle size and the shock pressure all being precisely measured quantities. The shock-recovered samples were examined by a variety of means including optical microscopy, electrical transport, SEM, TEM, and SQUID magnetometer measurements.

A. *THE SHOCK COMPACTION PROCESS*

The shock waves in our experiments were generated by means of a two-stage light-gas gun which accelerates a 20 mm diameter, 5 g lexan projectile at a copper capsule containing the high-T_c sample. A schematic of the copper sample capsule and the target area is shown in Figure 1. The impact of the lexan projectile onto the copper capsule generates a planar shock wave which compresses the sample. Further details can be found in Ref. 5.

Because the generation of shock waves by means of a gas gun occurs under well-controlled conditions, computer simulations of these shock experiments are fairly straightforward through the use of a two-dimensional axisymmetric finite-element code such as DYNA2D. These simulations are valuable because they yield information on the stress history and stress distribution of the shocked specimens, and thus allow one to better understand the conditions under which the powders were consolidated and the shock-induced defects generated. Standard Mie-Gruneisen equations of state and constitutive models were used for all materials. Figure 2 shows the recovery target in the vicinity of the copper sample capsule as calculated by DYNA2D at 30 μsec after impact of a 1.5 km/sec lexan projectile onto the copper capsule. The plastic projectile is seen to have rebounded off the copper sample capsule and is moving away (downward) from the copper capsule. The copper capsule itself has deformed and extruded outward slightly, trapping it in the steel recovery block. Because the sample thickness is small (≈ 0.5 mm), the sample chamber was not included in these calculations. Figure 3 shows a plot of the calcu-

lated pressure history of point *P* in Figure 2. The peak pressure is 70 kbar and the duration of this pressure is about 2 μsec. This calculation reveals tensile stresses at point *P* at times of 7 and 15 μsec after impact, the magnitude of these stresses being about a kbar.

II. SHOCK COMPACTION OF $YBa_2Cu_3O_{7-\delta}$ + Ag POWDER

Shock compaction experiments on $YBa_2Cu_3O_{7-\delta}$ + Ag powder at a number of shock pressures were performed to determine the behavior of $YBa_2Cu_3O_{7-\delta}$ as a function of shock pressure. Since the amount of heterogeneous heating of the particle surfaces depends on the magnitude of the shock pulse, it was expected that the electrical transport properties of the shocked compacts might be very sensitive to the shock pressure, and so a full study at a wide variety of shock pressures was performed to explore and understand the effects of shock consolidation on $YBa_2Cu_3O_{7-\delta}$.

The samples were prepared using $YBa_2Cu_3O_{7-\delta}$ powder obtained from Argonne National Laboratory (ANL). The $YBa_2Cu_3O_{7-\delta}$ powder was sifted for a uniform 28-45 μm particle size. The sifted $YBa_2Cu_3O_{7-\delta}$ was then mixed with 20-30 μm Ag powder to form a $YBa_2Cu_3O_{7-\delta}$ + 30% vol. Ag mixture, the silver here serving to act as a binder for the $YBa_2Cu_3O_{7-\delta}$ and to increase the ductility of the shocked compact. In a practical application the silver would also function as a current shunt in the event that the superconductive component of the composite were to quench.

Shock recovery experiments were prepared with this powder mixture for shock pressures of 29, 58, 87, and 167 kbar[5]. Approximately 190 mg of the powder mixture was used for each shot. The powder was loaded into the 10 mm diameter disk-shaped sample chamber of the copper shock-recovery capsule diagrammed in Figure 1.

Optical micrographs of the shock-recovered samples revealed that fully dense composites were obtained even at the lowest shock pressure (29 kbar). There was considerable shock-induced fracturing, however, with the grain size decreasing with increasing shock pressure. For the 167 kbar specimen, the average grain size had been reduced to ≈1 μm from the starting grain size of ≈10 μm. Furthermore, twin lamellae were observed throughout the grains at all shock pressures, indicating that the orthorhombic, superconducting phase of $YBa_2Cu_3O_7$ is largely retained in the grain interiors for shock pressures up to at least 167 kbar.

Powder diffraction x-ray spectra on the unshocked starting powder and the 167 kbar shocked compact revealed some amount of transformation from the orthorhombic superconducting phase of $YBa_2Cu_3O_7$ to the tetragonal semiconducting phase. The tetragonal phase is presumably localized on the particle surfaces where heterogeneous shock heating is greatest, since the bulk shock temperature in the 167 kbar shocked sample is esti-

mated to have been below 400 °C. This view is supported by optical micrographs of the 167 kbar shocked specimen, as described in the previous paragraph.

Zero-field-cooled (zfc) and field-cooled (fc) magnetization measurements were made on the shocked composites under a 25 Oe applied magnetic field. The zero-field-cooled magnetic screening signals are shown in Figure 4. First, note that there is no decrease in the bulk critical temperature with shocking. The grain interiors, then, apparently retain their superconducting properties with little loss of oxygen. Secondly, the screening signal decreases with increasing shock pressure, this effect apparently being due to decreasing grain size; as the average grain sizes in the shocked compacts approach the length scale of the superconducting penetration depth, a larger and larger volume fraction of the superconducting material will reside within a penetration depth of a grain surface, and this results in progressively smaller superconducting screening signals with decreasing grain size. Quantitatively, this size effect can be described by the following equation derived by Clem and Kogan[6] for weakly-linked superconducting spheres:

$$\frac{M}{M_o} = -\frac{3}{2} f_o \left(1 - \frac{3\lambda}{R} \right)$$

Here M is the zfc magnetization at T=0, M_o is the ideal Meissner magnetization (- $H/4\pi$), f_o is the volume fraction of superconducting material, λ is the magnetic penetration depth, and R is the radius of the spheres (assuming $\lambda/R \ll 1$). Indeed, taking the average grain size of the unshocked powder to be R=5 μm and using the measured magnetization ratio of M/M_o=0.84, the above equation yields average grain diameters of 2.8, 1.9, 1.4, and 0.8 μm for the 29, 58, 87, and 167 kbar shocks, respectively. These sizes are consistent with grain sizes observed by optical microscopy, which supports the view that the decreasing screening signals observed in Figure 4 can be largely accounted for by grain size reduction.

Transmission Electron Microscopy (TEM) was performed on the $YBa_2Cu_3O_7$ + 30 vol. % Ag shocked composites. The sample foils were prepared by mechanical polishing and then dimpling the shocked specimen to 40-50 μm, followed by argon-ion milling on a liquid-N_2 cooled stage. These studies revealed a remarkably high density of shock-induced dislocations. The $YBa_2Cu_3O_7$ shocked to 167 kbar, for instance, had a dislocation density in the range of 10^{12} cm^{-2}, which is approximately two orders of magnitude higher than dislocation densities generated in $YBa_2Cu_3O_7$ samples which had been subjected to slow mechanical deformation[7].

The shock-induced dislocations had line vectors of <100> and <110> lying in the (001) planes. These dislocations result from the large shear stresses generated across

the (001) planes during shock loading. Indeed, due to the different crystallographic orientations of the individual grains with respect to the shock front, which resulted in a wide range of shear stresses being generated across the (001) planes, the dislocation density was found to vary widely from grain to grain of the polycrystalline sample. According to Schmid's law[8], the shear stresses across the (001) planes and, hence, the dislocation densities should be greatest in those grains oriented with their c-axis at an angle of 45° with respect to the shock direction. A TEM micrograph showing numerous shock-induced dislocations in a single crystal of $YBa_2Cu_3O_7$ shocked to 30 kbar is presented in Fig. 5. As will be seen, these dislocations are an important microstructural feature because they serve as very effective magnetic flux-pinning sites, and result in a considerable increase in the intragranular critical current density. A high density of twin lamellae was also observed in the 167 kbar as-shocked specimen, the average twin spacing being about 0.02 μm. This spacing is several times narrower than what is found in typical as-sintered materials, indicating that some of the shock stress is accommodated by deformation twinning.

The TEM studies of the 167 kbar sample also found evidence of melting around some of the grains due to heterogeneous shock-heating. The melted regions appear to be tetragonal phase material which form a layer ≈1 μm thick around the $YBa_2Cu_3O_7$ grains. Low-angle grain boundaries similar to those found in melt-textured material as well as dendritic growth structures were found in these melted regions. Melted regions were not observed in the specimens shocked to lower pressures.

Interestingly, we found that the 167 kbar shocked $YBa_2Cu_3O_7$ retained a high density of defects even after annealing the shocked material at 890 °C for an extended period of time[4] (24 hrs.). Although few shock-induced dislocations remained after this long anneal, a very high density of extrinsic stacking faults was found, these stacking faults consisting of an intercalation of an extra Cu-O plane by b/2. Because of the large average separation between the partial dislocations which bound each stacking fault (≈0.5 μm), the stacking fault energy is fairly small (≈ 10 erg/cm^2), and this fact may partially explain the stability of the stacking faults even with extensive high-temperature annealing.

Specimens for electrical transport measurements were cut from the shocked compacts and had dimensions of roughly 8mm x 1mm x 0.3 mm. Resistivity measurements were made from room temperature down to 2 K. The resistivities of the as-shocked specimens tended to be linear with temperature, indicating that the current flows mostly through the Ag matrix rather than through the $YBa_2Cu_3O_7$, even below T_c.

To recover superconducting transport, the resistivity specimens were subjected to various annealing runs in so as to heal microcracks and transform the tetragonal, non-superconducting surface layers on the grains back to the orthorhombic, superconducting phase. Anneals were performed for various times and at various temperatures from 450 °C

to 890 °C. Superconducting transport was recovered only after an 890 °C anneal, suggesting that resintering is needed to heal microcracks. The sample shocked to 167 kbar and then annealed at 890 °C exhibited a transport J_c of 320 A/cm^2 at 77 K with zero magnetic field.

III. FLUX-PINNING AND INTRAGRANULAR J_c

The high density of dislocations found in the 167 kbar shocked specimen suggests that the magnetic flux-pinning energy and the intragranular J_c of this sample may be extremely high. In order to determine the flux-pinning energy of this sample, we performed magnetic relaxation experiments with a SQUID magnetometer. The magnetic decay rate of a sample, assuming cylindrical geometry, was calculated by Beasley *et.al.*[9] to be

$$\frac{dM}{d\ln t} = \frac{r J_c}{3} \frac{kT}{U_o} \qquad (1)$$

where r is the cylinder radius, J_c is the intragranular critical current density, and U_o is the flux-pinning energy. Within the approximation that the grains of the sample are cylindrical, the above equation can be applied with r now being the characteristic grain size.

To obtain an averaged value for rJ_c, we make use of Bean's equation[10] which relates rJ_c to $\Delta M(H)$, the width of the magnetic hysteresis loop at H:

$$\Delta M(H) = \frac{2}{3} \frac{r J_c}{c} \qquad (2)$$

Combining Eqns. (1) and (2) yields the following grain-size-independent expression for U_o:

$$U_o(H) = \left[\frac{\Delta M(H)}{[dM(H)/d\ln t]} \right] \frac{kT}{2} \qquad (3)$$

Flux-relaxation experiments were performed at various temperatures after zero-field-cooling to the desired temperature and applying a 10 kOe magnetic field. Magnetic hysteresis runs were also performed to determine $\Delta M(H=10\text{ kOe})$. Using this data and Eqn. 3, the pinning energies shown in Figure 6 were determined for the starting

$YBa_2Cu_3O_7$ + Ag powder mixture, the as-shocked sample, and the shocked + 890 °C annealed sample. Note that the shock process results in a substantial increase in the flux-pinning energy of the sample, apparently because of the large flux-pinning contribution from the high density of shock-induced defects. Also, note that the enhanced pinning is retained to some degree even after an 890 °C anneal. This enhanced pinning is presumably due to flux-pinning by the partial dislocations associated with the extrinsic stacking faults in the annealed material.

The increases in flux-pinning energy shown in Figure 6 have a considerable effect on the intragranular J_c's. Because the characteristic magnetic flux-pinning energies of $YBa_2Cu_3O_7$ and other high-T_c superconductors at 77 K are nearing thermal energies, the intragranular critical current densities of these materials at liquid-N_2 temperature are limited by the process of thermally activated flux-line motion. This results in a strong dependence of the intragranular J_c on the pinning energy U_o, the intragranular J_c having a roughly exponential, Arrhenius-like dependence on U_o (i.e. $J_c \sim \exp(U_o/kT)$) (Ref. 11). Consequently, modest increases in U_o can translate into substantial increases in intragranular J_c. Indeed, by using optical micrographs to estimate the average grain size of the shocked compact and applying the magnetic hysteresis data to Bean's equation (Eqn. 2), we find that the intragranular critical current density of the as-shocked material is approximately 10 times greater than that of the starting powder. A similar measurement of the intragranular J_c of the shocked + 890 °C annealed specimen reveals a factor of 5 increase in intragranular J_c.

IV. SHOCK EXPERIMENTS ON $Bi_2Sr_2CaCu_2O_8$ - CRYSTALLOGRAPHIC ALIGNMENT

Shock recovery experiments were also performed on the compound $Bi_2Sr_2CaCu_2O_8$. This compound is much more ductile than $YBa_2Cu_3O_7$ and shows little tendency to fracture under shock.

The crystallographic grain alignment of high-T_c superconductors is important for forming high-J_c bulk materials because (1) the high-J_c directions of high-T_c materials are in the basal plane and these directions should be aligned with the direction of current flow, and (2) the coherence length of high-T_c materials is greater in the *a-b* plane than in the c-axis direction, so to maximize intergranular coupling it is desirable to align the grains. Additionally, if the grain morphology is platelet-like, as in the case of $Bi_2Sr_2CaCu_2O_8$, alignment of the grains yields a microstructure with large overlap areas between grains, which results in better intergranular coupling simply by virtue of increased intergranular contact areas[12,13].

To form crystallographically aligned $Bi_2Sr_2CaCu_2O_8$ compacts we shock compacted prealigned pressed powders. The starting powder was made by solid state reaction by the DuPont Company. This micacious powder was sifted for the 30-40 μm size. The sifted powder was observed by SEM and found to consist of platelets 10 μm thick, 30-40 μm wide, and 40-100 μm long. A high degree of alignment was then obtained by simply tapping this powder into a specimen holder, with the planes of the platelets aligning perpendicular to the tapping direction. For all compactions, the shock direction was along the tapping direction, or perpendicular to the *a-b* planes of the aligned powder.

Specimens were prepared and shocked at 30, 50, 70, 100, and 140 kbar with 2 μsec duration. Figure 7 shows powder x-ray diffraction patterns taken for a randomly oriented starting powder specimen (Figure 7(a)), the sieved powder after tapping (Fig. 7(b)), and the compact shocked to 100 kbar (Fig. 7(c)). Note the strong (00*l*) reflections in the as-tapped specimen, which indicates a high degree of crystallographic alignment with the c-axes perpendicular to the plane of the compact. This alignment is preserved with shock-consolidation, as can be seen in Fig. 7(c).

Optical microscopy of the shocked specimens reveals a high density (>95% of crystal density), and a high degree of texturing by plastic flow. In contrast to $YBa_2Cu_3O_7$ shock experiments, there is very little shock-induced fracturing of the grains. Also, in contrast to shock experiments in which the shock direction was parallel to some of the *a-b* planes[14], there was relatively little kinking of the *a-b* planes in our shocked samples.

Magnetometer experiments on the shocked compacts revealed no change in the onset T_c from that of the starting powder (≈90 K), although the magnitude of the low-field (20 Oe) diamagnetic screening signal tended to decrease with increasing shock pressure. Since there is little shock-induced fracture in $Bi_2Sr_2CaCu_2O_8$, this decrease in the screening signal is apparently due to a decrease in the superconducting volume fraction because of heterogeneous shock heating and oxygen loss at the particle boundaries. This contrasts with the case of $YBa_2Cu_3O_7$ which showed little decrease in the superconducting volume fraction as a result of shocking but substantial shock fracturing.

An interesting result of the shock compaction of the $Bi_2Sr_2CaCu_2O_8$ powder was the discovery that there was a large increase in the average aspect ratio of the particles due to plastic flow. Low-field magnetic screening measurements were performed both parallel and perpendicular to the c-axis of the shocked compact. Figure 8 is a plot of the magnetic anisotropy for H parallel and perpendicular to the c-axis for various shocked samples as well as for a $Bi_2Sr_2CaCu_2O_8$ pellet pressed and annealed at 800 °C. Note the enhancement of magnetic anisotropy for the shocked samples, indicating high aspect ratios for the grains in these samples. The increase in aspect ratio is apparent from optical micrographs of these samples; the shocked specimens have highly textured microstructures with high aspect ratio grains, while the sintered specimen has low aspect ratio grains and no dis-

cernable texture. Shock consolidation of $Bi_2Sr_2CaCu_2O_8$, then, can produce highly textured and dense bulk high-T_c superconductors with heterogeneously processed grain boundaries. Future work will focus on finding the proper shock conditions and post-shock treatments to maximize intergranular J_c's.

VI. CONCLUSIONS

We have seen that shock-consolidation of high-T_c superconductors addresses both the intragranular and intergranular aspects of the fabrication of bulk superconductors having high critical current densities. The dramatic increase in intragranular J_c with shocking is particularly significant since the relatively low intragranular J_c's in high-T_c superconductors are starting to become a limiting factor in the attainment of high J_c's in bulk materials[15]. Currently, there are a limited number of methods for increasing intragranular J_c's. Although neutron[16] and proton[17,18] irradiation methods have been valuable in demonstrating that intragranular J_c's of $\approx 6 \times 10^5$ A/cm^2 are possible at liquid-N_2 temperatures, there are numerous problems associated with irradiation techniques including the destruction of intergranular links[17], the requirement of long-time irradiation periods, and the production of residual radioactivity in the samples. Because of these problems, the applicability of irradiation methods to the large-scale fabrication of bulk superconductors is questionable. Shock-processing with explosives, on the other hand, can quickly and efficiently process large quantities of shock-defected high-T_c material having greatly enhanced intragranular J_c's.

We have also seen that highly textured bulk specimens of $Bi_2Sr_2CaCu_2O_8$ can be fabricated by shock-consolidation. This technique, then, is a promising one for creating superconducting material with good intergranular links and high critical current densities. Future experiments will include further studies of the properties of shock-consolidated, textured $Bi_2Sr_2CaCu_2O_8$ compacts as a function of pressure and post-shock treatment with the aim of attaining the highest possible transport J_c.

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FIG. 1 *Schematic of the shock-recovery fixture for shocked high-T_c powders.*

FIG. 2 *DYNA2D cross-section calculation of the shock-recovery fixture 30 μsec after lexan projectile impact at 1.5 km/sec. Axisymmetry about a radius=0 axis is assumed.*

FIG. 3 *Calculated pressure history at point P of Fig. 2.*

FIG. 4 *Zero-field-cooled magnetic susceptibilities of the shocked $YBa_2Cu_3O_7$ + 30 vol. % Ag samples relative to the ideal Meissner value, $(4\pi)^{-1}$.*

FIG. 5 *TEM micrograph of a $YBa_2Cu_3O_7$ crystal shocked to 30 kbar. The crystal was oriented so that its c-axis was at an angle of 45° with respect to the shock direction.*

FIG. 6 *Flux-pinning energy U_o (at H=10 kOe) as a function of temperature for shocked and unshocked $YBa_2Cu_3O_7$ + 30 vol. % Ag samples.*

FIG. 7 *Powder diffraction patterns of (a) starting powder with random orientation, (b) as-tapped powder; also, shocked to 100 kbar for (c) as-shocked, (d) shocked and annealed at 800 °C in oxygen, and (e) shocked and annealed at 850 °C in oxygen. Cu Ka line was used for all patterns.*

FIG. 8 *Zero-field-cooled magnetic susceptibility anisotropy for magnetic field parallel and perpendicular to the "c-axes" of the pressed or shocked compacts $(\chi(H||c)/\chi(H\perp c))$.*

Fig. 1

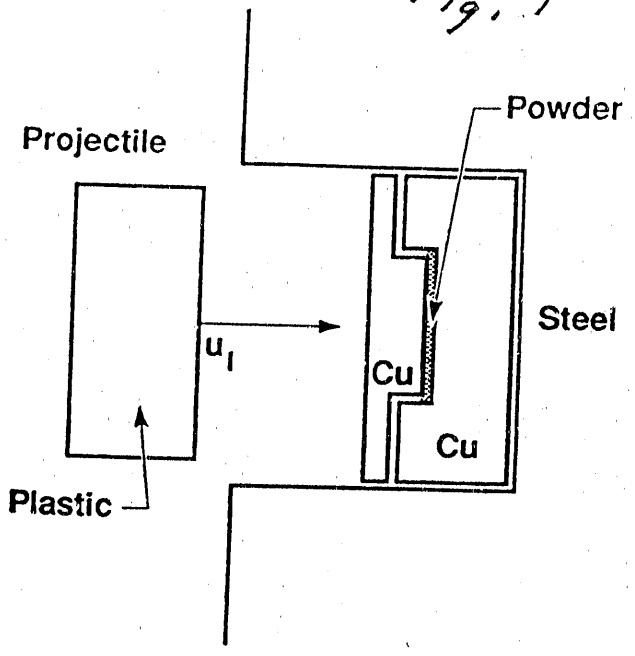


Fig. 2

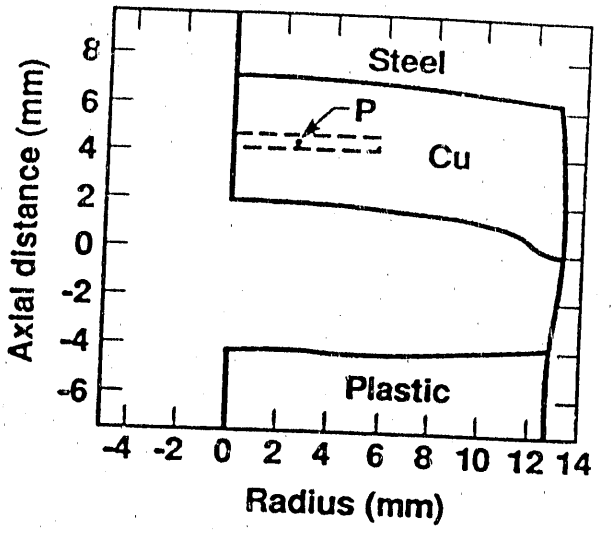


Fig. 3

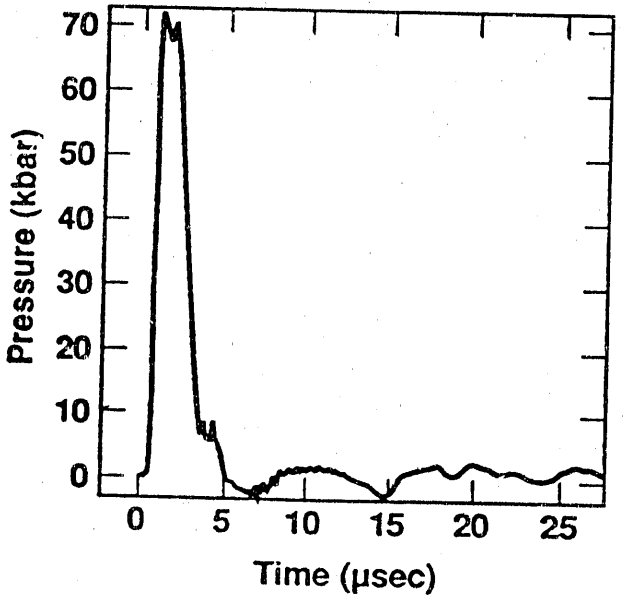


Fig. 4

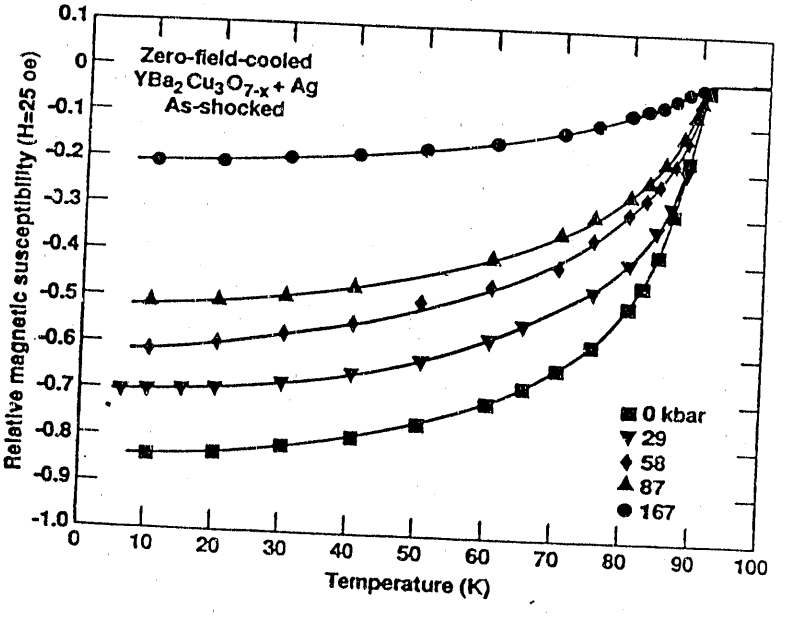


Fig. 5

Fig. 6

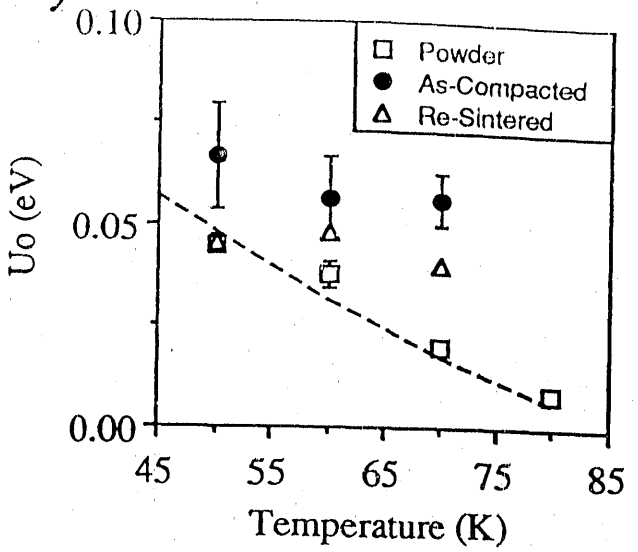


Fig. 7

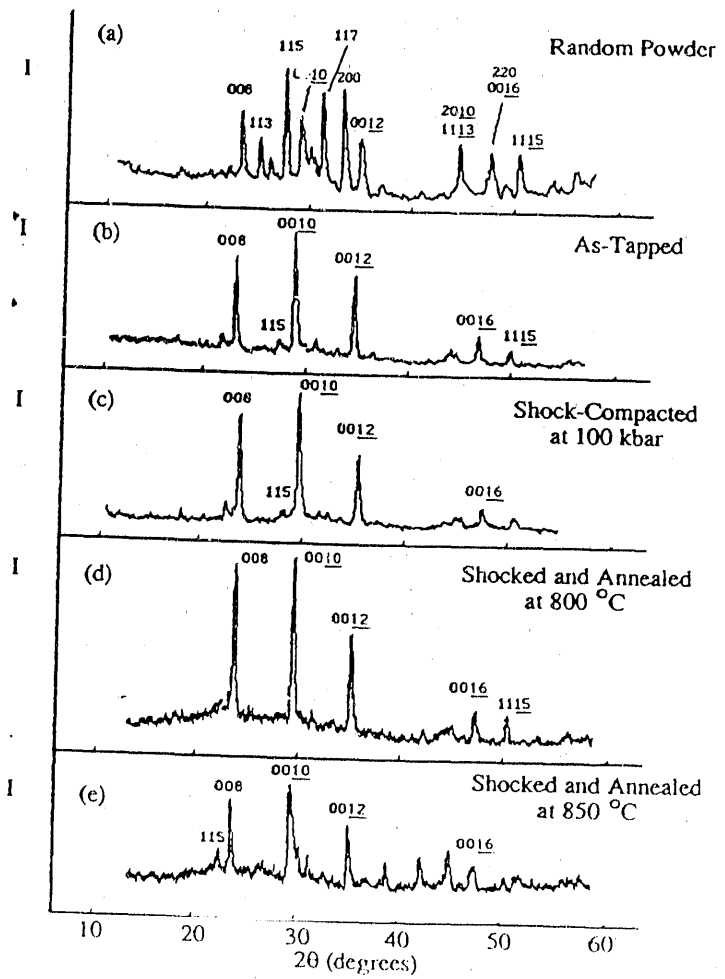
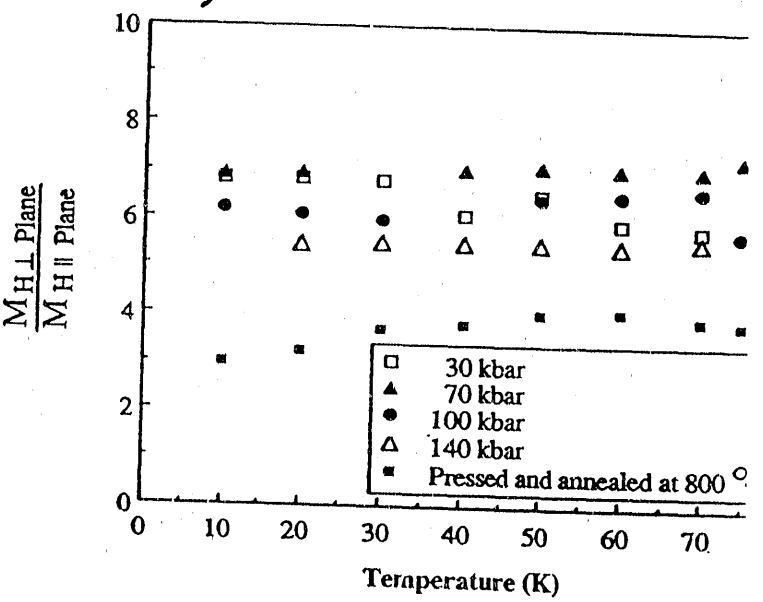


Fig. 8



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