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Shock-wave synthesis of a thallium-based superconductor with a novel defect microstructure

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We report the shock-wave synthesis at a yield $\gtrsim 80\%$ by volume of the single copper layer thallium superconductor of composition $Tl_2Ba_2CuO_6$. The as-synthesized material displays zero resistance near 55 K and a diamagnetic onset to bulk superconductivity at 70 K. Lattice imaging indicates that the superconducting microcrystals consist of a novel defect microstructure involving an intergrowth of two copper-oxygen layers probably interleaved by partial thallium and barium occupancy.

We have been investigating the application of shock waves in the processing and nonequilibrium synthesis of the high-temperature superconductors.[1] The method is unique in that a pulse in the range 10–100 kbar associated with local temperature rise of a few thousand degrees can be achieved in the time frame of a few microseconds, followed by high quench rates (typically 10^4–10^6 K/s). In this manner highly dense, machinable parts of materials such as ceramic superconductors can be fabricated with built-in defect microstructures. Defect microstructures have been obtained by shock-processing $YBa_2Cu_3O_7$ (Y123), which lead to a large increase in flux trapping[2] and a decrease in grain boundary flux creep,[3] as indicated by magnetization and low-field microwave absorption data, respectively.

In this letter we report the shock-induced synthesis and properties of the single copper oxide layer superconductor of ideal composition $Tl_2Ba_2CuO_6$. The material is a bulk superconductor in the as-synthesized state and consists of a novel defect microstructure. Previous attempts at shock synthesizing the oxide superconductors Y123, $La_{2-x}Sr_xCuO_4$ (La214) and $Bi_2Sr_2CaCu_2O_8$ have been made.[2] Only in the case of La214 was the correct structure obtained as a majority fraction, but a post-shock furnace anneal near 1000 °C under oxygen was required to provide the complete superconducting composition.

A well calibrated fixture configuration depicted in Fig. 1 was used for the experiments. The steel flyer plate (monitored by a high-speed camera) is accelerated downwards by the detonation of an explosive charge and impacts the precursor powders in the capsules. Realistic numerical simulations of the shock compression conditions in fixtures of this type have been carried out.[4] The analysis reveals contours of pressure and temperature at various times in microseconds within the powder mass packed at different densities. In our experiments, estimated shock pressures in the 200 kbar range were achieved for a 65% packing density and the residual temperature was a few hundred degrees centigrade. The synthesis experiments were carried out with a matrix material of composition $Ba_2Cu_3O_5$ (obtained by firing $BaCO_3$ and CuO at 930 °C in air for 30 h with one intermediate grinding) mixed with Tl_2O_3 and BaO_2 to give powders in which the Tl:Ba:Cu ratios are 123, 223, and 221.

The products obtained from 123 and 223 precursors were well consolidated at near theoretical density, whereas samples from the 221 precursors showed somewhat poorer consolidation. Representative powder diffraction patterns of 123 and 221 products are shown in Fig. 2. The diffraction lines can be indexed to the patterns for the tetragonal and orthorhombic thallium phase of composition $Tl_2Ba_2CuO_6$ (referred to as the Tl2201 phase).[5] The 123 product corresponds to 80–85% by volume as estimated from the x-ray

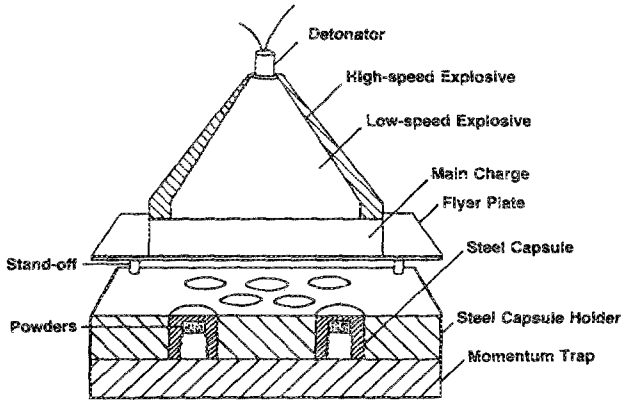

FIG. 1. Fixture used for the shock synthesis experiments.

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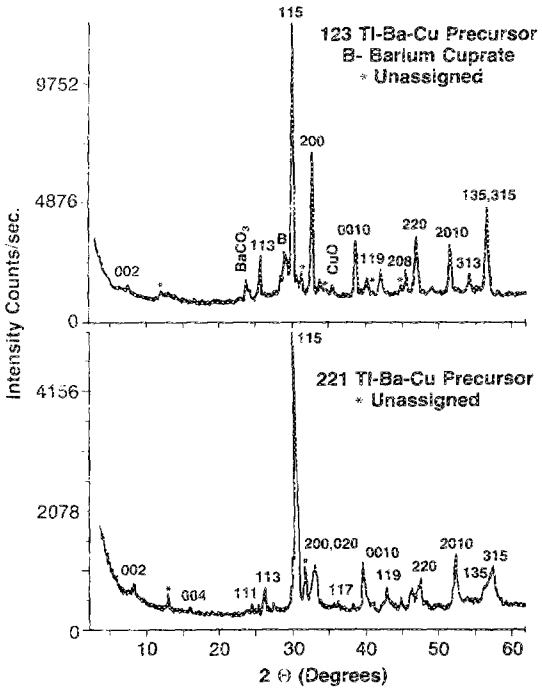

FIG. 2. Powder x-ray diffraction patterns using Cu $K\alpha$ radiation of the products obtained from Tl:Ba:Cu = 1:2:3 and 2:2:1 precursors.

diffraction data, whereas the 221 product corresponds to nearly 95% of the slightly orthorhombic Tl2201 phase. Small amounts of CuO, $BaCuO_2$, and $BaCO_3$ are detected in the 123 product. Both products also contain an unassigned impurity. The x-ray diffraction of the 223 product is similar to that of the 123 product except that there is a substantial fraction of unreacted Tl_2O_3. The heavy metal composition of the 221 product obtained via electron microprobe analyses corresponds closely to a Tl:Ba:Cu ratio of 2:2:1. However, the products from the 123 and 223 precursors consistently gave a ratio of 2:2:2. Since the latter precursors had an excess of copper it is likely that the extra copper in the microcrystals is associated with a thin amorphous layer of CuO on the crystal surfaces.

Lattice imaging of the microcrystals clearly reveals the Tl2201 structure as shown by the images taken in the (100) setting (Fig. 3). In addition, an intergrowth defect in which the sequence is ..Tl–Tl–Ba–Cu–□–Cu–Ba.. is also found. These defects are fairly common in the 123 product and were not observed in the 221 product crystals. Similar defects have not been seen in our investigations and those of others on furnace-synthesized Tl2201 material. Computer simulations indicate that the intervening site between the two copper layers in the defect can be partially occupied by Tl and Ba. In addition to this defect occasional intergrowths of a single Tl layer instead of the usual double Tl layer are seen in the images.

Normalized four-probe resistivity data obtained using a 95 mA current are shown in Fig. 4 for the 123 and 221 products. The 123 product shows nominally zero resistance at 55 K while no transition is seen for the 221 product. This is consistent with observation of superconductivity only in the tetragonal Tl2201 phase in the case of furnace-synthesized material.[6] The 223 product does not show zero resistance but

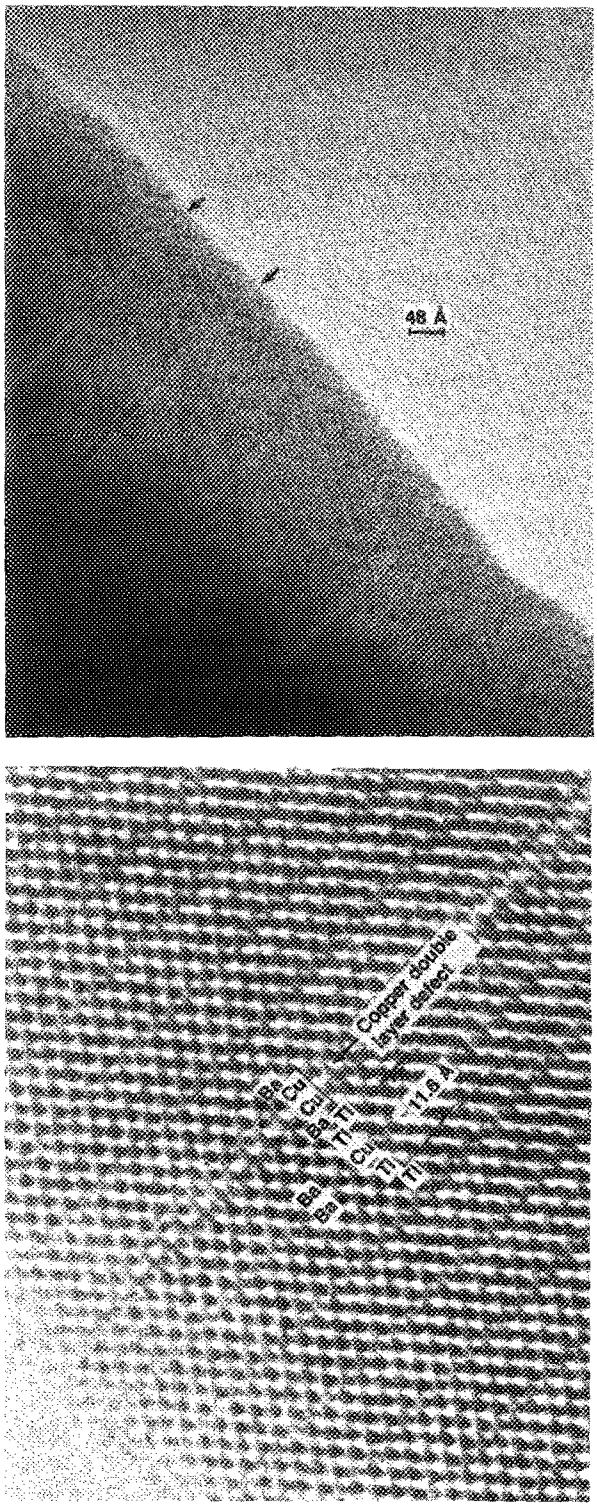

FIG. 3. Top: lattice image of a microcrystal obtained from the shocked product synthesized from a Tl:Ba:Cu = 1:2:3 precursor. Arrows indicate double copper layer defects. Bottom: closer view of the double copper defect.

a large drop in resistance is observed at 40 K. ac susceptibility data shown in Fig. 5 confirm a diamagnetic transition in the 123 product with an onset temperature near 70 K and a shielding value of 80% at 0.3 Oe. dc magnetization data at 30 Oe show a Meissner fraction greater than 10%, indicating bulk superconductivity in the sample. However, the magnet-

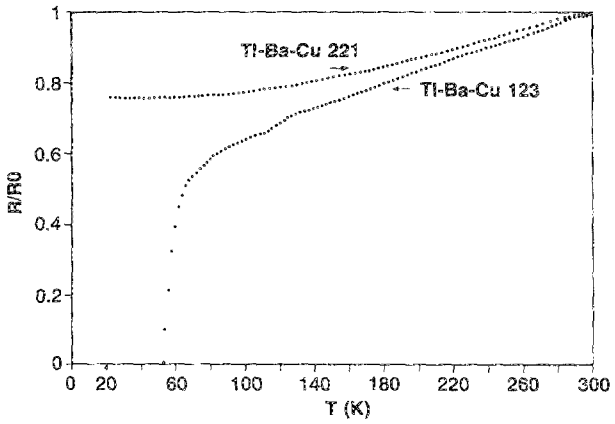

FIG. 4. Normalized dc resistivity vs temperature data obtained using a 95 mA current for products obtained from Tl:Ba:Cu = 1:2:3 and 2:2:1 precursors.

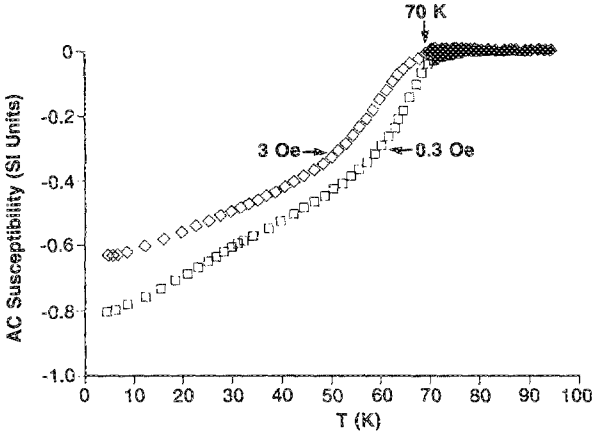

FIG. 5. ac susceptibility vs temperature data obtained in two different fields for the product from a Tl:Ba:Cu = 1:2:3 precursor.

ic transition is wide and appears to occur in two steps which are probably associated with modulations of the tetragonal Tl2201 phase.[6]

Preliminary measurements indicate that the transport critical current density at 20 K for the shock-synthesized Tl2201 material is not enhanced above values obtained for typical furnace-synthesized material probably because high sample densities are not achieved. Low-field microwave absorption hysteresis measurements,[7] however, indicate enhanced flux trapping at the grain boundaries of the shocked product. The double copper layer defect discussed above can provide the necessary pinning sites. Experiments are under way to improve the intergrain connectivity of the shock-synthesized material and additionally to synthesize the $Tl_2Ba_2Ca_2Cu_3O_{10}$ phase with novel microstructures.

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