A novel processing technique for fabrication of flexible $YBa_2Cu_3O_{7-x}$ wire

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A fabrication technique for producing flexible superconductor wire by extrusion of a composite of Ag_2O-$YBa_2Cu_3O_{7-x}$ is reported. During the course of processing, the superconductive transition of the starting powder is maintained, thus obviating the need for subsequent heat treatment. This nonpoisoning behavior of Ag_2O is of significant technical importance. Scanning electron fractography reveals a mixed mode fracture behavior of the material and the evidence of ductility. Magnetic measurements on the extruded wires show an onset temperature of 91 K. Resistivity measurements indicate that the electrical resistance of the as-extruded wire essentially vanishes at liquid-nitrogen temperature.

The recent discovery[1] of $YBa_2Cu_3O_{7-x}$, a high-temperature ceramic superconductor with zero resistance above liquid-nitrogen temperature (77 K) has attracted increasing attention to explore the possibility of its applications in areas such as computer interconnections, magnets and power transmission, and to develop processing techniques to produce single crystals, thin films, wires, rods, strips, etc. Two current important problems, while not directly addressing the mechanism of superconductivity, should nonetheless be noted. The first, efficient processing of these ceramics into flexible (with strength and toughness) wires, strips, or cables would require that the plasticity (formability) of these materials have to be significantly improved so that the reliable forming methods such as extrusion and drawing or their combinations can be adopted for large scale production. The second question concerns the superconducting properties of these flexible products.

It has been our conjecture that the successful fabrication of flexible wires, rods, and strips will require the development of metal-superconductor composites and the processing methodology of that composite powder, and finally extruding,[2] drawing,[3] or a combination of both, and rolling. The inherent brittleness of ceramic superconductors has posed problems in utilizing conventional metal forming processes. The ductility can be improved, as shown in this communication, when a composite of superconducting ceramic is formed by processing fine grain $YBa_2Cu_3O_{7-x}$ embedded in a metal or metal oxide.[4] Furthermore, the composites with metals [5-10] such as Au, Ag, and Ag_2O, in addition, should exhibit sufficient environmental stability. The unidirectional metal deformation processes such as extrusion and drawing are known to introduce texture, which is reported[11] to have improved the critical current density of bulk superconductors. Since the extruded or drawn wires, rods, and strips would contain Ag and/or Ag_2O, joining and connecting to terminators should be relatively simple.[10]

In this study, composite samples were prepared by thoroughly mixing the $YBa_2Cu_3O_{7-x}$ powder with desired quantities of fine silver oxide (Ag_2O) powder (99.9% of purity), ranging from 50 to 70 vol %. The average particle size of the starting powders as in the 5–10 μm range. The mixture was then warm pressed at 200 °C into 9.2 mm diameter and approximately 19-mm-long compacted billets. Compaction at 200 °C resulted in significantly high green density and strength. The compacted billets were then sintered in air at 880 °C for 2 h, furnace cooled to 500 °C, and were held for 1 h before finally furnace cooling down to room temperature. The sintered billets were wrapped in Ag foil (0.006 in. thick) before placing in the extrusion chamber. Note that this processing technique is fundamentally different from the powder-in-Ag tubing procedure. Here the silver foil is used only to minimize die wear and to improve environmental stability of the finished product. The sintered billet itself has enough plasticity and does not need any extrinsic ductile enclosure to render formability. Subsequently, the billet was extruded at 450 °C into 3.2-mm-diam wire maintaining a single step extrusion ratio of 9:1.

Figure 1 displays a photograph of a representative extruded wire along with the parent sintered billet. The composite wire displays significant flexibility to be bent rather sharply to almost 90° without breaking. Optical microscopy of the extruded product revealed a dense microstructure. Absence of any microcracks indicated that the composite deformed practically like a metal.

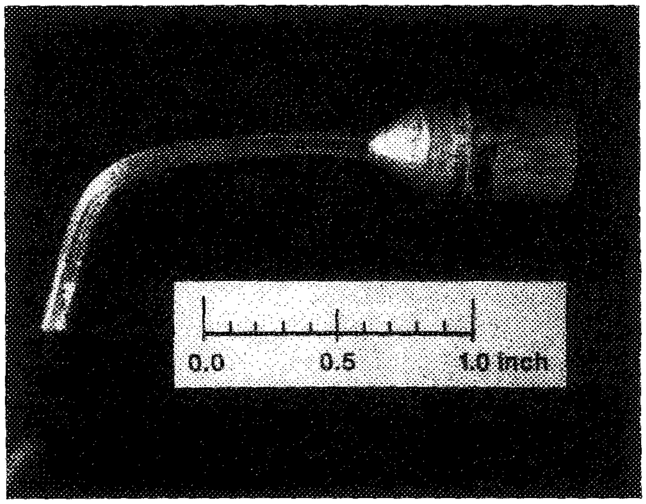

FIG. 1. Photograph of a typical extruded wire along with the sintered billet. Note the bendability of the wire.

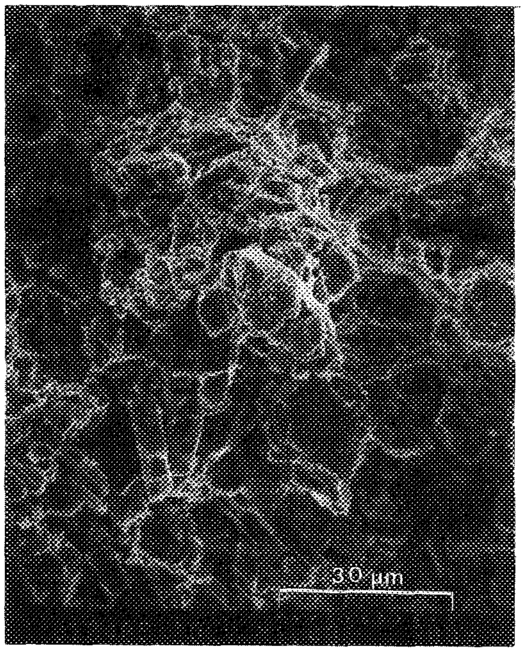

FIG. 2. Scanning electron micrograph of a typical fractured surface observed in the extruded wires.

Figure 2 shows a scanning electron fractograph of the extruded wire of 50:50 composition. This is a clear evidence of mixed mode fracture wherein white regions of metallic, ductile (dimple) fracture surrounds relatively dark regions of planar, faceted brittle (cleavage) fracture. It is understood that the microstructure primarily consists of a brittle (superconducting ceramic) phase dispersed in a metal (Ag) matrix which forms as a result of decomposition of the Ag_2O during the sintering operation. At a higher magnification, Fig. 3 shows how the ductile metallic phase (white) binds the ceramic particles (gray and dark) together and thus imparts toughness to the composite. Note that the interphase interface is very strong and tough and does not tend to fail. It is interesting to find how an incipient microcrack (arrowhead) is blunted and prevented from propagating. On the contrary, numerous intergranular cracks running along the interceramic-particle boundaries testify for a rather poor bonding between the $YBa_2Cu_3O_{x-7}$ grains. The key to developing a tough, formable composite is, therefore, to minimize the poor ceramic-grain contact and ensure a very uniform distribution of these particles in the tough, ductile matrix. Hence, appropriate techniques for uniform mixing of the starting powders are of importance. The addition of Ag_2O actually serves two purposes. First, it simplifies the processing route as *no post-sintering oxygen annealing is required* for the composite to exhibit superconductive transition. It is reasonable to think that *the decomposition of Ag_2O during the sintering operation supplied the oxygen necessary to maintain the proper stoichiometry* of the $YBa_2Cu_3O_{7-x}$ superconducting phase. And second, it eventually helps to provide in the finished product a tough, ductile metallic (Ag) matrix in which grains of the superconducting phase are embedded. It is of immense importance that these two phases are chemically compatible so that they give rise to a strong microcomposite structure (having high interface strength) and not merely to a weakly bonded mechanical aggregate. Our results suggest that by virtue of *in situ* silver generation, the starting ingredient Ag_2O provides an excellent metal host for $YBa_2Cu_3O_{7-x}$ superconducting ceramic for large plastic deformation.

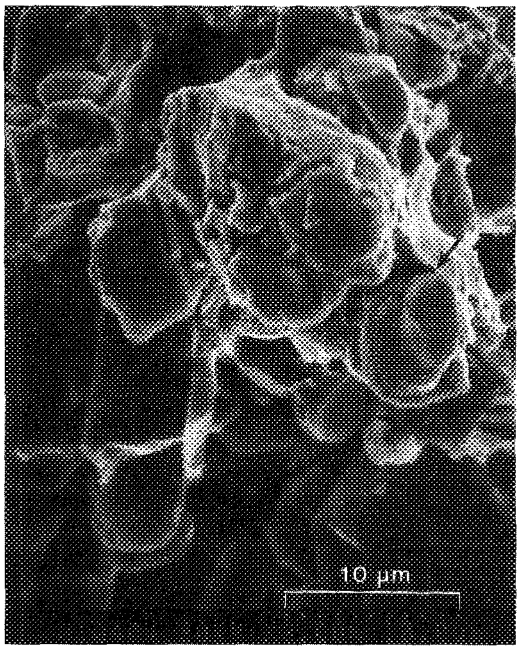

FIG. 3. A region from Fig. 3 at higher magnification.

The superconducting critical temperatures of all specimens were determined from magnetization measurements using a SQUID magnetometer. Figure 4 shows a plot of the magnetic moment as a function of temperature from the results of flux exclusion test conducted on an as-extruded wire sample of 50:50 composition. The onset of superconductivity associated with the Meissner effect is apparent at around 91 K. Clearly, the addition of Ag_2O has a negligible effect on the superconducting transition temperature. It is worth noting that the as-extruded wire has significant environmental

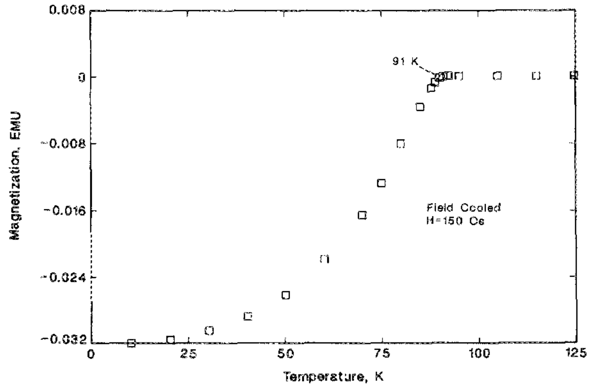

FIG. 4. Magnetic moment as a function of temperature for an extruded wire of 50:50 composition.

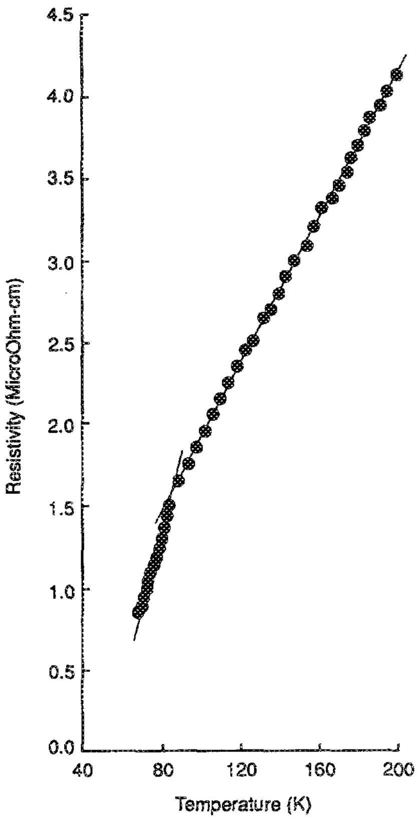

FIG. 5. Resistivity as a function of temperature for an extruded wire of 50:50 composition.

stability and is found to exhibit superconductive transition even after being stored in the ambient for more than 30 days. Standard four probe resistivity measurements were conducted using a 10-mA direct current source in zero field. Specimens of 5-mm^2 cross section and 10 mm length, prepared from the as-extruded wire sample of 50:50 composition, indicate, as expected, a superconductive transition at around 90 K (Fig. 5). The resistivity of the wires essentially tends to vanish below the transition point. However, there is indication of some residual resistivity. The origin of this residual resistivity and the aspect of connected critical current density in the bulk composite product will be reported shortly.[12]

In summary, we have discovered a processing technique for fabricating flexible wires of $YBa_2Cu_3O_{7-x}$ composite at a relatively low temperature (450 °C). This novel technique has already shown[12] potential for fabricating fine (0.75 mm diam) wire, from $YBa_2Cu_3O_{7-x}$ superconducting ceramic, and is considered to be generic in nature since bismuth containing high-temperature superconducting oxide is expected to behave similarly. By using this processing technique it would also be possible to fabricate irregular-shaped bulk superconducting parts with relatively simple process equipment.

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