PREPARATION AND CHARACTERIZATION

OF

SOME SELECTED CERAMIC

SUPERCONDUCTORS

S.H.S.P. Samarappuli

Ph.D

1996

University of Peradeniya
Sri Lanka

Preparation and Characterization of some selected Ceramic Superconductors

Abstract

Some selected ceramic cuprate superconductors and related materials were prepared by several methods, and various cation substitution effects on structural, electrical and magnetic properties investigated.

Y-Ba-Cu-O samples were prepared by standard solid state reaction and precursor methods, and their structural, electrical and magnetic properties are discussed.

Bi-Sr-Ca-Cu-O samples with several starting compositions were prepared by solid state reaction and citrate pyrolysis methods and their structural, electrical and magnetic properties are discussed. The oxygen content of some samples was determined using the iodometric titration technique.

A new superconducting phase has been found in a mixed system prepared from the $YBa_2Cu_3O_{7-\delta}$ and $Bi_2CaSr_2Cu_2O_{9-\delta}$ high T_c materials. The new compound shows an onset of resistivity transition at ~100 K and zero resistivity below ~50 K. The powder x-ray diffraction pattern of the new compound differs completely from those of $YBa_2Cu_3O_{7-\delta}$ and $Bi_2CaSr_2Cu_2O_{9-\delta}$.The material prepared using a mixture of the oxides and carbonates with identical cation stoichiometric ratios, on the other hand does not exhibit a similar phase.

Properties of a series of superconducting compounds of nominal composition $(Bi_{0.7}Pb_{0.3})SrCa(Cu_{1.5-x}Ni_x)O_\delta$ where x ranges from 0 to 1.1 have been investigated using electrical resistivity, AC magnetic susceptibility and x-ray powder diffraction. From $x = 0$ up to $x = 0.2$, T_c drops to 60 K rather rapidly. From $x = 0.2$ to 0.95 the material remains superconducting with T_c around ~50 K. For $x \geq 1.0$ the material becomes semiconducting. The fact that even the compound with Ni : Cu = 0.95 :0.55 is superconducting with T_c around ~50 K may suggest that the magnetic disorder introduced by Ni is smaller in this material than in the Ni-doped $YBa_2Cu_3O_{7-\delta}$ material, where samples with Ni : Cu = 0.75 : 2.25 lose their superconducting properties.

Superconducting properties of phosphorus added $(Bi_{0.7}Pb_{0.3})SrCaCu_{1.5}O_y$ system using two mixing ratios, $Ca_3(PO_4)_2$: $CaCO_3$ = 1 : 4, and 1 : 1 have been investigated using electrical resistivity, AC magnetic susceptibility and x-ray

diffraction. The results show that phosphorus substitution for bismuth using phosphates in a solid state reaction is unlikely to be realised easily. This methods results in the formation of a multiphase material with a low transition temperature.

Superconducting properties of samples with nominal composition $(Bi_{0.7}Pb_{0.3})SrCa(Cu_{1.4}Pd_{0.1})O_y$ prepared by solid state reaction technique have been investigated using electrical resistivity, AC magnetic susceptibility and x-ray powder diffraction. The material shows a zero resistivity transition at ~70 K. Resistivity, susceptibility and x-ray diffraction data suggests that the formation of the low temperature phase (2212) of the pure material has been stabilized due to Pd doping and that Pd is actually substituted for copper in this phase.

Electrical resistance, magnetic susceptibility and powder x-ray diffraction of $(Bi_{0.7}Pb_{0.3})SrCaCu_{1.5}O_y$ pellet samples subjected to an extra annealing cycle have been investigated. Zero resistivity transitions of 120 K have been attained in these samples. The rise in T_c is attributed to the change in microstructure of the samples due to the extra thermal annealing.

Samples of $Bi_2Sr_2(RE_{1-x}Th_x)_2Cu_2O_{10-\delta}$ (RE = Pr, Nd, Sm, Eu, Gd, Tb, Dy, and Ho) have been synthesized. Their structure is identical to that of the layered copper oxides $Bi_2Sr_2(RE_{1-x}Ce_x)_2Cu_2O_{10-\delta}$ the so-called 2-2-2-2 phases. Due to the larger ionic radius of Th^{4+} compared to that of Ce^{4+}, the lattice constants for $x = 0.18$ are larger than the cell parameters of the corresponding Ce compound. The as-sintered samples are not superconducting above 1.9 K.

Results from AC susceptibility $\chi(H,T)$ and resistivity $\rho(H,T)$ measurements on $YBa_2Cu_3O_7$ single crystals, in the mixed state by applying magnetic fields H parallel to the c-axis, strongly suggest that the occurrence of the dissipation peak in the imaginary part of the complex susceptibility $\chi''(\omega,T)$ is due to the skin effect known for electrical transport in metals in the normal state. At a fixed field H, the peak temperature $T_p(\omega,H)$ increases with increasing frequency of the superimposed AC probing field, and does not coincide with the irreversibility temperature $T^*(H) < T_p$ for magnetization obtained in the static limit $\omega \rightarrow 0$. A phase diagram is presented which includes the DC irreversibility line $H^*(T)$, the peak positions $H_p(\omega,T)$, the experimentally resolved zero-resistivity fields $H_{\rho=0}(T)$, and the crossover fields from activated to diffuse resistive behaviour, $H_k(T)$, all measured on the same $YBa_2Cu_3O_7$ single crystal.