UNIVERSITI PUTRA MALAYSIA

THE EFFECT OF CD AND NB ON BI-1212 SYSTEM

AZMAN AWANG TEH.

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By

AZMAN AWANG TEH

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

June 2004
SPECIALY DEDICATED
TO
MY WIFE
AND
FAMILY
Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Master of Science

THE EFFECT OF Cd AND Nb ON Bi-1212 SYSTEM

By

AZMAN AWANG TEH

June 2004

Chairman: Professor Abdul Halim Shaari, Ph.D.

Faculty: Science and Environmental Studies

The 1212 phase of Bi-based superconductor by theory has good properties in terms of structure and transition temperature, as it is an adaptation of the Y123 system and the Bi2212 system. The new Bi1212 system with stoichiometries \((Bi_{0.2}Pb_{0.6}M_x)Sr_2(Y_{0.3}Ca_{0.7})Cu_{2.05}O_\delta\) and \((Bi_{0.4}Pb_{0.35}M_x)Sr_2(Y_{0.3}Ca_{0.7})Cu_{2.05}O_\delta\) where \(M = \text{Cd and Nb}, x = 0, 0.05, 0.1, 0.15, 0.2, 0.25\) has been successfully synthesized and characterized by means of X-Ray (powder) diffraction, scanning electron microscope, AC susceptibility and resistivity measurements. For the composition of \((Bi_{0.2}Pb_{0.6}M_x)Sr_2(Y_{0.3}Ca_{0.7})Cu_{2.05}O_\delta\), the above stated systematic examinations clarified the effect of Cd and Nb on Bi-1212 phase, both as a substitution \((0 < x \leq 0.2)\) and as an addition \((x = 0.25)\). All samples were prepared using solid-state reaction. These samples underwent sintering process at two different temperatures, which were 960°C and 980°C, for a time duration of 2 hours and 10 hours. Then all the samples underwent heat treatment in flowing argon at 750°C for 10
hours. All the samples were orthorhombic with the space group Pmmm except the compounds with composition (Bi$_{0.2}$Pb$_{0.6}$Cd$_{0.05}$)Sr$_2$(Y$_{0.3}$Ca$_{0.7}$)Cu$_{2.05}$O$_{5\delta}$ and (Bi$_{0.4}$Pb$_{0.35}$Cd$_{0.05}$)Sr$_2$(Y$_{0.3}$Ca$_{0.7}$)Cu$_{2.05}$O$_{5\delta}$, which were sintered at 960°C for 2 hours were tetragonal with space group P4mmm. The X-ray diffraction pattern showed that all samples were in mixed phase (1212 and 2212) with 1212 acting as a dominant phase. All as-prepared samples showed non-superconducting properties below 20 K, but after heat treatment in flowing argon, some of them showed superconducting properties with the highest transition temperature of $T_{c,zero}=64$ K and $T_{c, onset}=80$ K obtained from Nb-doping sample with $x=0.2$. The ac susceptibility studies showed that most of the superconducting material exhibited weak grain conductivity. The surface morphology of most of the samples observed from scanning electron microscope showed a layered-slab texture.
Abstrak tesis yang dikenakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

**KESAN-KESAN PENDOPAN ELEMEN KE ATAS SISTEM Bi-1212**

Oleh

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Fasa 1212 bagi superkonduktor berasaskan Bi secara teorinya mempunyai sifat yang baik daripada segi suhu peralihan dan sifat pepejal sampel disebabkan oleh pengabungan antara dua asas iaitu sistem Y123 dan sistem Bi2212. Sistem Bi1212 yang baru melibat komposisi 

\[(\text{Bi}_{0.2}\text{Pb}_{0.6}M_x)\text{Sr}_2(\text{Y}_{0.3}\text{Ca}_{0.7})\text{Cu}_{2.05}\text{O}_6\]

dan

\[(\text{Bi}_{0.4}\text{Pb}_{0.35}M_x)\text{Sr}_2(\text{Y}_{0.3}\text{Ca}_{0.7})\text{Cu}_{2.05}\text{O}_6\]

dengan M ialah Cd dan Nb, dengan nilai x=0, 0.05, 0.1, 0.15, 0.2, 0.25. Semua sampel disediakan dengan menggunakan kaedah tindakbalas keadaan pepejal. Sampel-sampel ini disinter pada dua suhu berlainan iaitu 960°C dan 980°C untuk jangkamasa 2 jam dan 10 jam. Sifat angkutan sampel-sampel ini ditentukan dengan menggunakan kaedah penduga empat titik, sifat magnet menggunakan kerentanan au, mikrostruktur oleh Mikroskop Imbasan Elektron (SEM) dan struktur serta fasa kimia oleh teknik pembelauan sinar-X (XRD). Bagi komposisi \((\text{Bi}_{0.2}\text{Pb}_{0.6}M_x)\text{Sr}_2(\text{Y}_{0.3}\text{Ca}_{0.7})\text{Cu}_{2.05}\text{O}_6\), pemeriksaan-pemeriksaan yang dinyatakan di atas akan dapat menerangkan kesan Cd dan Nb ke atas fasa Bi1212 sama
ada sebagai pendopan \((0 < x \leq 0.2)\) atau penambahan \((x = 0.25)\). Terdapat sampel yang bukan superconductor dan sampel yang superkonduktor dengan suhu peralihan yang tertinggi diperolehi adalah \(T_{c,\text{site}}=64\ \text{K}\) and \(T_{c,\text{mul}}=80\ \text{K}\) daripada sampel pendopan dengan Nb bagi \(x=0.2\). Struktur permukaan semua sampel yang diperolehi daripada ujian SEM menunjukkan permukaan yang berlapis-lapis. Daripada ujian XRD pula didapati struktur bagi semua sampel adalah ortorombik dengan kumpulan Pmmm kecuali sampel dengan komposisi \((\text{Bi}_{0.2}\text{Pb}_{0.6}\text{Cd}_{0.05})\text{Sr}_2(\text{Y}_{0.3}\text{Ca}_{0.7})\text{Cu}_{2.05}\text{O}_\delta\)

\((\text{Bi}_{0.4}\text{Pb}_{0.35}\text{Cd}_{0.05})\text{Sr}_2(\text{Y}_{0.3}\text{Ca}_{0.7})\text{Cu}_{2.05}\text{O}_\delta\)

yang disinter pada suhu \(960^\circ\text{C}\) selama 2 jam adalah tetragonal dengan kumpulan P4mmm. Semua sampel menunjukkan terdapatnya campuran fasa \((1212\ \text{dan}\ 2212)\) yang didominasi oleh fasa \(1212\). Kajian kerentanan au ke atas sampel-sampel menunjukkan pengaliran arus di antara butiran-butiran adalah lemah.
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I certify that an Examination Committee met on 4th June 2004 to conduct the final examination of Azman Awang Teh on his Master of Science thesis entitled “The Effect of Cd and Nb on Bi-1212 System” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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Date: 10 SEP 2004
DECLARATION

I hereby declare that this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

AZMAN AWANG TEH

Date: 23 August 2004
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5.4.7(a-b) AC susceptibility component (a) $\chi''$ and (b) $\chi'$ of 
$(Bi_{0.2}Pb_{0.6}Cd_x)Sr_2(Y_{0.3}Ca_{0.7})Cu_{2.05}O_{6}$, $x=0.15$ sintered at 980°C for 10 hours and annealed.

5.4.8(a-b) AC susceptibility component (a) $\chi''$ and (b) $\chi'$ of 
$(Bi_{0.2}Pb_{0.6}Cd_x)Sr_2(Y_{0.3}Ca_{0.7})Cu_{2.05}O_{6}$, $x=0.25$ sintered at 980°C for 10 hours and annealed.

5.4.9(a-b) AC susceptibility component (a) $\chi''$ and (b) $\chi'$ of 
$(Bi_{0.2}Pb_{0.6}Nb_x)Sr_2(Y_{0.3}Ca_{0.7})Cu_{2.05}O_{6}$, $x=0.2$ sintered at 960°C for 2 hours and annealed.