UNIVERSITI PUTRA MALAYSIA

FABRICATION AND CHARACTERISATION OF MONO- AND MULTIFILAMENT Ag-SHEATHED Bi1.Pb0.4Sr2Ca2Cu3O10 SUPERCONDUCTOR TAPES VIA POWDER-WIRE-IN-TUBE TECHN IQUE

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By

MASRIANIS BT AHMAD

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

December 2005

DEDICATION

To my husband, Isa Bin Muhzan
&
my mother, Hajjah Aishah Bt Hj. Che Omar
for their love, support and understanding.....

To my family,
for their love and concern......

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Master of Science

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December 2005

Chairman: Professor Abdul Halim bin Shaari, PhD

Faculty : Science

The co-precipitation technique was used in the preparation of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10}$ polycrystalline ceramic superconductor powder to fabricate Ag-sheathed superconductor tapes. Powder prepared via co-precipitation method with ultra-fine grain size was used to enhance the Bi-2223 phase formation. The tapes were prepared using the powder-in-tube and powder-wire-in-tube method. The powder-wire-in-tube (PWIT) method has been developed by packing powder together with composite wires into silver tubes at the second stage of the powder-in-tube (PIT) process. Among the different routes proposed to enhance Bi-2223 phase formation, the PWIT method showed better results. The samples were prepared with different number of filaments (number of filament = 2, 4, 6, 8; PWIT and 0, 20; PIT) and different sintering times (24 hr, 48 hr and 100 hr) heated at 850 °C. Samples heated for longer time showed enhanced 2223 phase formation for pellet and monofilament tape samples from 92 % to 96 % and 84 % to 86 % respectively. The intergranular connectivity can be improved. Sintering temperature 850° enhanced the growth of the superconductor phase, which was much faster in the *ab*-plane than along the *c*-axis.

This made the platelets form well-aligned connections with each other.

in the Bi-2223 XRD peak intensity with sintering time is due to the re-arrangement of the Bi-2212 crystals, which occurs in the larger volume of liquid phase. A slight preferred orientation of the grains in the *c*-axis direction when the samples were rolled into tapes was also observed. By increasing the sintering time the average grain size is increase from 4 μm to 14 μm and by increasing the number of filaments from 2 to 8 filaments inside the tube and core area that developed the non-superconducting phase and the secondary phase were minimized. Small filaments in multifilament tapes are more homogenous than monofilament tapes owing to the better grain orientation along the silver sheath. At 77 K and zero field, the highest transport critical current density (J_C) 11500 ± 300 A/cm^2 was achieved in the tape prepared via PWIT (number of filament = 8) sintered for 100 hours compared to PIT method with transport critical current density (J_C) 7800 ± 300 A/cm^2 at the same sintering duration. Therefore, critical current density increases with the sintering duration and number of filaments.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

FABRIKASI DAN PENCIRIAN MONO DAN MULTIFILAMEN PITA SUPERKONDUKTOR Ag-$Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10}$ SARUNGAN-Ag MELALUI TEKNIK SERBUK-DAWAI-DALAM-TIUB

Oleh

MASRIANIS BT AHMAD

December 2005

Pengerusi : Profesor Abdul Halim bin Shaari, PhD

Faculti : Sains

Teknik pemendakan bersama telah digunakan bagi menyediakan superkonduktor seramik polihablur $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10}$ untuk fabrikasi pita superkonduktor sarungan Ag. Serbuk disediakan dengan kaedah pemendakan bersama untuk menghasilkan serbuk bersaiz ultra-halus bagi meningkatkan pembentukkan fasa Bi-2223. Pita telah disediakan menggunakan teknik serbuk-dalam-tiub dan serbuk-dawai-dalam-tiub. Teknik serbuk-dawai-dalam-tiub (PWIT) dibangunkan dengan memasukkan serbuk bersama-sama dawai ke dalam tiub Ag pada peringkat yang kedua teknik serbuk-dalam-tiub (PIT). Di antara pelbagai kaedah dalam meningkatkan pembentukan fasa Bi-2223, teknik serbuk-dawai-dalam-tiub turut memberikan keputusan yang baik. Sampel disediakan dengan bilangan filamen yang berbeza (bilangan filamen = 2, 4, 6, 8; PWIT dan 0, 20; PIT) dan masa pembakaran yang berlainan (24 jam, 48 jm dn 100 jam) pada suhu 850 °C. Sampel yang dibakar lama menunjukkan pembentukan fasa 2223 meningkat bagi sampel pelet dan pita masing-masing dari 92 % ke 96 % dan 84 % ke 86 %. Keadaan struktur butiran antara butiran diperbaiki. Pembakaran melebihi 850° meningkatkan pertumbuhan

fasa superkonduktor yang mana ia lebih cepat dalam arah *ab* berbanding arah *c*. Ini menunjukkan lapisan-lapisan disusun mudah di antara satu sama lain. Peningkatan puncak Bi-2223 dengan masa pembakaran ialah kerana hablur Bi-2212 menyusun semula menyebabkan fasa cecair bertambah. Didapati bahawa susunan butiran adalah dalan arah *c* bagi sampel yang telah digolek. Dengan meningkat masa pembakaran, purata saiz butiran meningka dari 4 μm ke 14 μm dan dengan menambah bilangan filamen dari 2 ke 8 filamen ke dalam tiub Ag menyebabkan kawasan tengah yang di pelopori oleh fasa bukan superkonduktor dan fasa sekunder dapat di kurangkan. Filamen halus dalam pita multifilamen adalah lebih homogen berbanding monofilamen disamping menghasilkan susunan butiran yang lebih baik di sepanjang permukaan perak. Pada suhu cecair nitrogen, 77 K dan tanpa medan, ketumpatan angkutan arus genting (J_C) ialah 11500 ± 300 A/cm^2 dicapai bagi pita yang disediakan menggunakan teknik serbuk-dawai-dalam-tiub (Mu08100) berbanding sampel pit100 yang menggunakan teknik serbuk-dalam-tiub dengan ketumpatan angkutan arus genting (J_C) ialah 7800 ± 300 A/cm^2. Oleh itu, ketumpatan angkutan arus genting (J_C) meningkat dengan tempoh masa pembakaran dan bilangan filamen.

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ABBREVIATIONS AND KEY WORDS

BCS theory	Bardeen, Cooper and Schrieffer theory
LBCO	La-Ba-Cu-O system
YBCO	Y-Ba-Cu-O system
Y123	Family member in $YBa_2Cu_3O_{7-x}$.
Bi-2201	Family member in $Bi_2Sr_2Ca_nCu_{n+1}O_{6+2n}$, n =0
Bi-2212	Family member in $Bi_2Sr_2Ca_nCu_{n+1}O_{6+2n}$, n =1
Bi-2223	Family member in $Bi_2Sr_2Ca_nCu_{n+1}O_{6+2n}$, n =2
TBCCO	Tl-Ba-Ca-Cu-O system
Tl-2223	Family member in $Tl_2Ba_2Ca_nCu_{n+1}O_{6+2n}$, n =2
HBCCO	Hg-Ba-Ca-Cu-O system
TGA	Thermo Gravimetric Analysis
DTA	Differential Thermal Analysis
Calcination	Heating process where the solid state reaction occur
Sintering	Heating process yielding for more compacting of the sample grains and improve its properties
PIT	Powder-in-tube
PWIT	Powder-wire-in-tube
B , H	Magnetic Field
$B_{\parallel}$	Magnetic field parallel to tape face
$B^{\perp}$	Magnetic field perpendicular to tape face
B_C	Critical magnetic field
B_{C1}	Lower Critical Field
B_{C2}	Upper Critical Field
T	Temperature
T_C	Critical Temperature
$T_{C\text{-onset}}$	Critical Temperature onset
$T_{C\text{-zero}}$	Critical Temperature zero
I	Current
E-I	Electric Field - Current
A	Surface area
V	Voltage