UNIVERSITI PUTRA MALAYSIA

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

MASRIANIS BT AHMAD

FS 2005 6
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MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA

2005
FABRICATION AND CHARACTERISATION OF MONO- AND MULTIFILAMENT Ag-SHEATHED Bi_{1.6}Pb_{0.4}Sr_{2}Ca_{2}Cu_{3}O_{10} SUPERCONDUCTOR TAPES VIA POWDER-WIRE-IN-TUBE TECHNIQUE

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

and 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

FABRICATION AND CHARACTERISATION OF MONO- AND MULTIFILAMENT Ag-SHEATHED Bi_{1.6}Pb_{0.4}Sr_{2}Ca_{2}Cu_{3}O_{10} SUPERCONDUCTOR TAPES VIA POWDER-WIRE-IN-TUBE TECHNIQUE

By
MASRIANIS BT AHMAD

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_{2}Ca_{2}Cu_{3}O_{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. The increase 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 ($J_C$) $11500 \pm 300$ 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 ($J_C$) $7800 \pm 300$ 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$_2$Ca$_2$Cu$_3$O$_{10}$ 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 polihabtur Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_{10}$ untuk fabrikasi pita superkonduktor sarungan Ag. Serbuk disediakan dengan kaedah pemendakan bersama untuk menghasilkan serbuk bersaiz ultra-halus bagi meningkatkan pembentukan 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 turnt 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 susun butiran adalah dalam arah \( c \) bagi sampel yang telah digolek. Dengan meningkat masa pembakaran, purata saiz butiran meningkat dari 4 \( \mu m \) ke 14 \( \mu m \) 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 susun 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 \( \pm \) 300 A/cm\(^2\) dicapai bagi pita yang disediakan menggunakan teknik serbuk-dawai-dalam-tiub (Mu08100) berbanding sampel pitl00 yang menggunakan teknik serbuk-dalam-tiub dengan ketumpatan angkutan arus genting (\( J_C \)) ialah 7800 \( \pm \) 300 A/cm\(^2\). Oleh itu, ketumpatan angkutan arus genting (\( J_C \)) meningkat dengan tempoh masa pembakaran dan bilangan filamen.
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I certify that an Examination Committee has met on 23rd December 2005 to conduct the final examination of Masrianis Bt Ahmad on her Master of Science thesis entitled “Fabrication And Characterisation of Mono- And Multifilament Ag-Sheathed Bi₁₋₆ Pb₃Sr₂Ca₂Cu₃O₁₀ Superconductor Tapes via Powder-Wire-In-Tube Technique” 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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Associate Professor  
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Date: 27 MAR 2006
This thesis submitted to the Senate of Universiti Putra Malaysia and was accepted as fulfilment of the requirements of the degree of Master of Science. The members of the Supervisory Committee are as follows:

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Date: **13 APR 2006**
DECLARATION

I hereby declare that the 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.

Date: 20/3/2006

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4.28 I-E curve for Ag-sheathed Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_8$ multifilament tape (number of filament = 2; PWIT) sintered at 850°C for 48 hours in zero field at 77 K, 70 K, 60 K and 50 K

4.29 I-E curve for Ag-sheathed Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_8$ multifilament tape (number of filament = 2; PWIT) sintered at 850°C for 100 hours in zero field at 77 K, 70 K, 60 K, 50 K and 40 K

4.30 I-E curve for Ag-sheathed Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_8$ multifilament tape (number of filament = 4; PWIT) sintered at 850°C for 24 hours in zero field at 77 K, 70 K and 60 K

4.31 I-E curve for Ag-sheathed Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_8$ multifilament tape (number of filament = 4; PWIT) sintered at 850°C for 48 hours in zero field at 77 K, 70 K, 60 K, 50 K and 40 K
4.32 $I-E$ curve for Ag-sheathed $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_5$ multifilament tape (number of filament =4; PWIT) sintered for 100 hours at different temperature dependences (B=0)

4.33 $I-E$ curve for Ag-sheathed $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_5$ multifilament tape (number of filament =6; PWIT) sintered at 850 °C for 24 hours in zero field at 77 K, 70 K and 60 K

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4.52 X-ray diffraction patterns for Ag-sheathed Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_{6.5}$ monofilament tape samples at different sintering time

4.53 X-ray diffraction patterns for bulk and monofilament tape samples sintered in air at 850 °C for 24 hours
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### ABBREVIATIONS AND KEY WORDS

<table>
<thead>
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<tr>
<td>BCS theory</td>
<td>Bardeen, Cooper and Schrieffer theory</td>
</tr>
<tr>
<td>LBCO</td>
<td>La-Ba-Cu-O system</td>
</tr>
<tr>
<td>YBCO</td>
<td>Y-Ba-Cu-O system</td>
</tr>
<tr>
<td>Y123</td>
<td>Family member in $\text{YBa}_2\text{Cu}<em>3\text{O}</em>{7-x}$</td>
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<tr>
<td>Bi-2201</td>
<td>Family member in $\text{Bi}_2\text{Sr}<em>2\text{Ca}<em>n\text{Cu}</em>{n+1}\text{O}</em>{6+2n}$, $n=0$</td>
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<td>Bi-2212</td>
<td>Family member in $\text{Bi}_2\text{Sr}<em>2\text{Ca}<em>n\text{Cu}</em>{n+1}\text{O}</em>{6+2n}$, $n=1$</td>
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<td>Bi-2223</td>
<td>Family member in $\text{Bi}_2\text{Sr}<em>2\text{Ca}<em>n\text{Cu}</em>{n+1}\text{O}</em>{6+2n}$, $n=2$</td>
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<td>TBCCO</td>
<td>Tl-Ba-Ca-Cu-O system</td>
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<td>TI-2223</td>
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<tr>
<td>HBCCO</td>
<td>Hg-Ba-Ca-Cu-O system</td>
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<tr>
<td>TGA</td>
<td>Thermo Gravimetric Analysis</td>
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<tr>
<td>DTA</td>
<td>Differential Thermal Analysis</td>
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<tr>
<td>Calcination</td>
<td>Heating process where the solid state reaction occur</td>
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<tr>
<td>Sintering</td>
<td>Heating process yielding for more compacting of the sample grains and improve its properties</td>
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<tr>
<td>PIT</td>
<td>Powder-in-tube</td>
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<tr>
<td>PWIT</td>
<td>Powder-wire-in-tube</td>
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<tr>
<td>B, H</td>
<td>Magnetic Field</td>
</tr>
<tr>
<td>$B_{</td>
<td></td>
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<tr>
<td>$B_{\perp}$</td>
<td>Magnetic field perpendicular to tape face</td>
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<tr>
<td>$B_C$</td>
<td>Critical magnetic field</td>
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<td>Temperature</td>
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<td>Surface area</td>
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