Synthesis of Bi$_2$Sr$_2$CaCu$_2$O$_x$ oxide precursor from nano-oxides and its relationship with multifilamentary wire transport properties

Yun Zhang$^1$, Stephen Johnson$^2$, Joey Stieha$^2$, Manasi Chaubal$^2$, Ganesh Venugopal$^2$, Andrew T. Hunt$^2$, Justin Schwartz$^1$

$^1$Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC, 27695-7907

$^2$nGimat, LLC, 2436 Over Dr, Lexington, KY, 40511
Bi2212/Ag wire before partial-melt processing

\[ T_1 = 819^\circ C, \ 2\text{Hrs} \]

\[ T_p, \ 0.2\text{ hrs} \]

\[ T_{\text{anneal}} = 832^\circ C, \ 48\text{Hrs} \]

\[ T_{p-10} \]

\[ 2.5^\circ C/\text{Hr} \]

\[ 10^\circ C/\text{H} \]

\[ 80^\circ C/\text{Hr} \]
Bi2212/Ag Wire after partial-melt processing

- Dense and connected Bi2212 grains
  - Capable of carrying high currents

- Non-superconducting Secondary phases

- Interfilamentary bridges

- Porosity
Challenges on Bi2212 precursors

- Stoichiometry
- Chemical homogeneity
- Grain size
- Carbon content
- Agglomeration (type, size)
- Other trace elements content
- Tap density
- Melting behavior
- Secondary phases distribution (size, content, type)
- Porosity
- Wire fabrication
- Grain alignment/texture
- Large-scale production
Technical Approach

NanoSpray Combustion™ (nGimat, LLC) + Solid-state calcination
Starting materials: Nanosize oxides via NanoSpray Combustion™

Average surface areas range from 9-14 \( \text{m}^2/\text{g} \);
Particle size: 67-104 nm

Multiple oxides
HAADF-STEM/EDS to reveal nm-scale chemical homogeneity

Mass transport diffusion length on 10s of nm scale → ensure homogeneous and synergetic reaction to form Bi2212;
Powder calcination: phase transformation from nano-oxides to Bi2212

Only Bi2212 peaks are detected: >95wt% pure

Fast phase transformation; no need for repeated pulverization and calcination;
Bi2212 precursor powder after full 72-hrs calcination

Soft agglomerations of Bi2212 single grains
Grain size: 3-8 micron;
Carbon dioxide release during full 72-hrs calcination

High surface area of starting nanosize oxides
\(\rightarrow\) CO\(_2\) release starts at very low temperature, 300°C lower than conventional method;
\(\rightarrow\) CO\(_2\) release completes before reaching calcination temperature;
\(\rightarrow\) <100 ppm carbon content in the final Bi2212 precursor;
Relationships between Bi2212 precursor properties and wire transport properties
Three precursor batches and wires
Bi2212/Ag/Ag-0.1wt%Al

**Stoichiometry**

- Bi$_{2.26}$Sr$_{1.90}$Ca$_{0.90}$Cu$_{1.98}$
- Bi$_{2.15}$Sr$_{1.89}$Ca$_{0.93}$Cu$_{1.95}$
- Bi$_{2.26}$Sr$_{1.89}$Ca$_{0.86}$Cu$_{1.99}$

**Wire configuration**

- LXA127A, 0.81 mm, FF=15%, 37 x 7
- LXA127B, 0.81 mm, FF=15%, 37 x 7
- LXA147, 0.81 mm, FF=12%, 91 x 7

*Stoichiometry measured by XRF, with a tolerance of 0.1 mol%*
Transport $J_c$ (4.2 K, 5 T) vs PMP peak temperature (1 bar processing)

50 bar overpressure on LXA147:
$J_c = 3960 \text{ A/mm}^2$ (4.2 K, 5 T)
Data courtesy of J. Jiang, FSU--NHMFL
Phase and carbon content of three precursor batches

Only Bi2212 peaks are detected

Carbon content:

- **LXA 147**: 50 ppm
- **LXA 127A**: 60 ppm
- **LXA 127B**: 90 ppm
Minor impurity phases in precursor - Image analysis on pellets by SEM/EDS

Only minor impurity: 
(Ca, Sr)$_2$CuO$_x$(AEC)

AEC Vol% significantly lower than previous studies; AEC size < filament size;

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>LXA127A</th>
<th>LXA127B</th>
<th>LXA147</th>
</tr>
</thead>
<tbody>
<tr>
<td>AEC vol%</td>
<td>0.29</td>
<td>0.03</td>
<td>0.86</td>
</tr>
<tr>
<td>AEC particle size (μm)</td>
<td>2-11</td>
<td>3-5</td>
<td>4-9</td>
</tr>
</tbody>
</table>
Melting behaviors of three green wires

-Thermal analysis

Precursor powder
Stoichiometry Std%: Composition variation

Shallow and wide melting peak $\Rightarrow$ high composition variation $\Rightarrow$ more phase segregations $\Rightarrow$ bad wire performance
Summary

• A novel method combining NanoSpray Combustion™ and solid-state calcination is used to synthesize Bi2212 oxide precursor.

• >99.1 vol% of Bi2212 single crystals with <0.5 mol% composition variation are synthesized.

• Small particle size, high surface area and short diffusion length of the starting materials → rapid and homogeneous phase transformation to Bi2212 + an early and rapid carbon release.

• Carbon content < 60 ppm is required.

• Precursor with Bi$_{2.26}$Sr$_{1.89}$Ca$_{0.86}$Cu$_{1.99}$ (LXA147) and 1.51 mol% composition variation → State-of-art wire transport current density: 2520 A/mm$^2$ (4.2 K, 5 T, 1 bar) and 3960 A/mm$^2$ (4.2 K, 5 T, 50 bar)
Acknowledgements

• This study is funded through DOE-STTR (DE-SC0009705).
• The authors are grateful to Leszek Motowidlo and Supramagnetics Inc for manufacturing the multifilamentary wires for this study.
• The authors acknowledge the use of the Analytical Instrumentation Facility (AIF) at North Carolina State University, which is supported by the State of North Carolina and the National Science Foundation.
• The authors also want to thank Jenna Pilato and Kyle Malone for assistance with this study.
THANK YOU!

YUN ZHANG