PROJECT TITLE

Time of flight detection on superconducting MgB2 films prepared by HPCVD and RF sputtering methods

INVESTIGATOR(S)

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SCIENTIFIC OBJECTIVES

In this project we will use Time of Flight method to:

1) Investigate the interface diffusion between MgB2 films and copper substrate for MgB2 coated SRF cavities.
2) Further investigate the oxygen content in the MgB2 films obtained from different deposition methods including PLD, HPCVD and RF sputtering.

PROGRESS REPORT and RESEARCH OUTCOMES

The project has been delayed due to a problem in the ANSTO time of flight elastic recoil detection analysizer (ToF-ERDA). However, the proposed research activities has been fully carried out despite the delay.

1. Nine MgB2 thin film and bulk samples were examined by ToF-ERDA at ANSTO. The samples include, MgB2 films on Copper substrate prepared by HPCVD method, MgB2 thin film prepared by the UHV PLD chamber using a new probationary off-axis geometry, and highly dense MgB2 bulks prepared by a new encapsulated two-step sintering method, as listed in Table 1.

Table 1. Sample description

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sample</th>
<th>Sample description</th>
<th>Tc (K)</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>23C</td>
<td>HPCVD MgB2 film on copper</td>
<td>36</td>
</tr>
<tr>
<td>2</td>
<td>22C</td>
<td>HPCVD MgB2 film on copper</td>
<td>36</td>
</tr>
<tr>
<td>3</td>
<td>23C-2</td>
<td>HPCVD MgB2 film on copper</td>
<td>36</td>
</tr>
<tr>
<td>4</td>
<td>22C-2</td>
<td>HPCVD MgB2 film on copper</td>
<td>36</td>
</tr>
<tr>
<td>5</td>
<td>21S-2</td>
<td>HPCVD MgB2 film on Al2O3-C</td>
<td>39</td>
</tr>
<tr>
<td>6</td>
<td>21S</td>
<td>HPCVD MgB2 film on Al2O3-C</td>
<td>39</td>
</tr>
<tr>
<td>7</td>
<td>20080607</td>
<td>PLD MgB2 film on Al2O3-C by rotation off-axis method</td>
<td>Non-sup</td>
</tr>
<tr>
<td>8</td>
<td>R13</td>
<td>Thin sheet of bulk MgB2, Two-step diffusive sintered</td>
<td>39</td>
</tr>
<tr>
<td>9</td>
<td>20080610</td>
<td>PLD MgB2 film on Al2O3-C by rotation off-axis method</td>
<td>15K</td>
</tr>
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</table>
2. We further investigated the role of oxygen impurity in MgB2 films prepared by off-axis PLD methods in conjunction with our 2007 AINSE project. The depth profiles of the serious MgB2 films in our 2007 report were calculated and analyzed. These depth results confirm our previous find out (already published in two conference papers) of the negative influence of oxygen on the Tc behavior of the MgB2 films. The precise element depth profiles also helped us in understanding the oxygen incorporation mechanism in good and bad MgB2 films.

**DATA**

We did ToF-ERDA experiments on 4 MgB2 films prepared by HPCVD on copper substrates. The typical composition profile along the depth is shown in Figure 1. It is found Mg content is relatively constant along the depth but B decrease from the film surface to the substrate interface. There is a significant Mg-Cu metallic compound transition zone between the MgB2 film and the Cu matrix. Oxygen level is much lower than PLD MgB2 films.

![Figure 1 ToF-ERDA depth-profile of content of B, O, Mg, and Cu in HPCVD film on Copper substrate (sample #1).](image)

Figure 2 shows a typical HPCVD MgB2 film of good Tc of 39 K on Al2O3-C wafer. It is found the surface layer of the MgB2 film is significantly Mg deficient. This indicate that during the cooling down process of HPCVD method, MgB2 decomposition reaction overcomes the drop of temperature.
Figure 2 ToF-ERDA depth-profile of content of B, O, Mg, and Al in HPCVD film on Al2O3-C substrate (sample #5).

Figure 3 a) and b) show the ToF-ERDA depth-profile of MgB2 films prepared by the off-axis PLD method in the APX UHV chamber. Mg and B are more evenly distributed in the “bad” MgB2 film (a). It is reckoned that Mg and B were not reacted and MgB2 phase were not formed in (a)-sample #7 due to the low deposition temperature.

Figure 3. ToF-ERDA depth-profile of content of B, O, Mg, and Al in a) “bad” off-axis PLD film (non-superconducting) on Al2O3-C (sample #7), and b) “good” off-axis PLD film (superconducting, with Tc of 15) on Al2O3-C (sample #9).

Figure 4 shows the high-density high-Tc MgB2 sheet sample on Si back plate by a new two-step diffusive sintering method. It is clear that B and Mg are evenly distributed and O content is extremely low comparing to the MgB2 films. On the other hand, Mg content appears slightly high in the middle of the sheet, which may be related to the diffusive sintering processing of the sheet.
Figure 4 ToF-ERDA depth-profile of content of B, O, Mg, and Si in the MgB2 sheet sample prepared by two-step diffusive sintering.

Shown below are four typical element depth profiles we obtained from ToF ERDA analysis of 2007 project samples. Reverse relation ship between Tc and Oxygen content is evident. Also found out is that with deceasing Mg content, the Tc of MgB2 films deceases.

Figure 5. Sample 011206M; MgB2/Al2O3; off axis; Mg cap layer; Tc=26K; A Oxygen=315,137
Figure 6. Sample 021206M; MgB$_2$/Al$_2$O$_3$; off axis; Mg cap layer; Tc=28K; A Oxygen=480,262

Figure 7. Sample 061206M; MgB$_2$/Al$_2$O$_3$; off axis; Mg cap layer; Tc=33K; A Oxygen=421,900
Figure 8. Sample 280806D; MgB$_2$/Al$_2$O$_3$; off axis; Xe sputtered; Tc=34.5K; A Oxygen in film=128,273

Signature of Investigator preparing the report for
After signing this report please fax this page with your signature for our files

Yue Zhao 15/5/2009

PUBLICATIONS / REPORTS arising as a result of your work.


PhD STUDENTS

1 PhD student: Mr Minoru Maeda, Dec 2010, Innovative Sintering method for high-density MgB2 bulk synthesis.