Surface Studies of Niobium
Chemically Polished Under
Conditions for SRF Cavity Production

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- Reproducibility Studies about Surface Roughness & Composition
- Effect of BCP Solution Flow Rate
- Polycrystal vs. Single Crystal
Experimental Work

- Fresh BCP (1:1:2) etch, 6 samples/batch, room temperature.

- Varied surface speed to represent flow rate difference in cavity processing: static $\rightarrow$ 2.0 inches/sec.

- Lab XPS analysis, $0^\circ$ take off angle, 6 samples/run.

- Stylus profilometry ("Dektak").

- Synchrotron based XPS analysis $\rightarrow$ NSLS, X1B.
  - $h\nu = 300$ eV, 550 eV, 930 eV, 1150 eV.
  - Take off angle $= 0^\circ$, 41°, 60°.
  - Spot size $< 250$ µm with enough intensity
Experimental Work

- XPS analysis: Nb 3d & O 1s spectra
Reproducibility Studies

*What differences are due to experimental scattering?*

- **Sample-to-Sample variation**

<table>
<thead>
<tr>
<th>No. (samples)</th>
<th>Roughness</th>
<th>Chemistry</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\text{Ra(\mu m)}$</td>
<td>$\sigma(\mu m)$</td>
</tr>
<tr>
<td>Sheet-7</td>
<td>30</td>
<td>1.66</td>
</tr>
<tr>
<td>Sheet-11</td>
<td>10</td>
<td>1.61</td>
</tr>
</tbody>
</table>

- Batch-to-batch variation is comparable to sample-to-sample. Position-to-position variation within samples and sheet-to-sheet variation are less.

- Roughness values and variation all exceed the few-nm escape depth of photoelectrons.

- The average intensity ratios of $\text{O}_{\text{total}}/\text{Nb}_{\text{total}}$ for each sample are much smaller than $2.5$ ($\text{Nb}_2\text{O}_5$).
Flow Rate Dependence of Surface Topography

No significant effect of BCP flow rate on surface roughness.
Flow Rate Dependence of Surface Chemistry

Effect of flow rate on niobium speciation is significant

Flow Rate Dependence of Surface Chemistry

Effect of flow rate on niobium speciation is significant
Different solution flow rate causes different surface chemistry, the surface roughness shows no significant change. 

- Nb$_2$O$_5$ is thicker on the high-flow sample. 
- Effect of solution flow rate is being studied.
Angle Resolved \textit{vs.} Variable Photon Energy XPS - Depth Profiling

<table>
<thead>
<tr>
<th>Photon energy (eV)</th>
<th>Sampling depth (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>1.764</td>
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<tr>
<td>550</td>
<td>3.309</td>
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<tr>
<td>930</td>
<td>5.340</td>
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<tr>
<td>1150</td>
<td>6.495</td>
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<tr>
<td>1254</td>
<td>7.014</td>
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</tbody>
</table>
Surface Chemistry (BCP 1:1:2, static)

Single Crystal vs. Polycrystal

Increasing $h\nu$, "see though" the surface $\text{Nb}_2\text{O}_5$

$h\nu$ variation is more surface sensitive than angle variation
Surface Chemistry II (BCP 1:1:2, static)

**Single crystal vs. Polycrystal**

- The intensity ratio of $\text{Nb}^{5+}/\text{Nb}_{\text{total}}$ of polycrystal is larger than that of single crystal.

- Detailed deconvolution is in progress.

- The single crystal surface is smoother ($Ra = 0.6 \, \mu m$) than the polycrystalline surfaces ($Ra = 1.66 \, \mu m$).
Conclusions

- Reproducibility studies with 40 samples provide first known assessment of XPS measurement variation significance on Nb; Scattering in the \( \text{Nb}^{5+}/\text{Nb}_{\text{total}} \) is about 5% of ratio; variation of intensity ratio of \( \text{O}_{\text{total}}/\text{Nb}_{\text{total}} \) is smaller than variation of surface roughness.

- Different solution flow rate causes different surface chemistry, the surface roughness shows no significant change.

- Variable photon energy XPS is more surface sensitive than angle resolved XPS.

- Single crystal is smoother than polycrystal, \( \text{Nb}_2\text{O}_5 \) of single crystal appears thinner than that of polycrystal.
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