Crystal orientations near welds in high RRR Niobium with very large grains

D. Baars\textsuperscript{3}, H. Jiang\textsuperscript{3}, T. Bieler\textsuperscript{3}, C. Compton\textsuperscript{1}, P. Bauer\textsuperscript{2}, T. Grimm\textsuperscript{1}

Abstract—Superconducting radio frequency (SRF) cavities made of single crystal niobium are under development for use in charged particle accelerators. Use of single crystals may simplify manufacturing and reduce cost, as well as improve properties over the currently used fine grain niobium material. However, the processes of forming by deep drawing, and subsequent welding of the formed parts to assemble the cavity, might lead to recrystallization in regions of high strain or curvature near the weld. Orientation imaging microscopy (OIM) was used to assess these possibilities in some preliminary experiments.

A sample of single crystal niobium strip was arbitrarily bent and electron beam (EB) heated across one end to simulate welding. The bent sample had no more than 14% strain, and it did not exhibit definitive recrystallization near or away from the EB heated area. Another sample was prepared by halving a large grain niobium bicrystal across the boundary of two grains, flipping one half and EB welding the halves back together, such that the weld had three different grain misorientations along its length, including two triple points. There was no formation of new orientations along the weld where it joined two crystal orientations. However, some new orientations solidified where the weld encountered three different grain orientations.

This preliminary data is encouraging, suggesting that minimal generation of new grain orientations during EB welding may be practical. However, more work with different orientations and strains is needed to determine how tolerant Nb is for maintaining a flat solidification interface and resisting recrystallization.

Index Terms—Accelerator Cavities, Electron Beam Weld, Niobium, Recrystallization

I. INTRODUCTION

Superconducting radio frequency (SRF) cavities made of very large grain niobium and of single crystal niobium are under development for use in charged particle accelerators [1]. Use of single crystals may simplify manufacturing and reduce cost, as well as improve properties over the currently used fine grain high RRR niobium. However, the processes of forming by deep drawing, and subsequent welding of the formed parts to assemble the cavity, might lead to recrystallization in regions of high strain and curvature near the weld, as well as stray grain formation within the weld itself. The experiments described provide a preliminary assessment of how this may occur in RRR Niobium joined using electron beam welding.

II. MATERIALS AND METHODS

Fig. 1 shows a schematic of a slice from a Niobium ingot that had only a few grain orientations present (CBMM Brazil, minimum RRR 250). The majority of this slice was used to make prototype cavity halves, and the center disc and the outer ring were used for the experiments described in this paper. A single crystal piece from the ring was arbitrarily bent by hand around a pipe and electron beam (EB) heated across one end to imitate welding (the other end was clamped, and served as a heat sink, Fig. 1). The EB power was 2.5 kW with a duration of about one minute. The sample reached yellow-white hot (about 1400°C, 0.6T\textsubscript{m}), about 2.5 cm from the weld in the region of highest curvature. Cold rolled high purity Nb can recrystallize at 700°C (0.4T\textsubscript{m}) [2]. In a study of recrystallization of a 90% cold rolled low carbon steel, complete recrystallization occurred in less than 10s, so it is reasonable to expect that recrystallization is possible in Nb electron beam welds [3]. Strains were estimated using digital

---

1 National Superconducting Cyclotron Laboratory, Michigan State University, East Lansing, MI, 48824 USA.
2 Fermi National Accelerator Laboratory, Batavia, IL, USA.
3 Chemical Engineering and Materials Science, Michigan State University College of Engineering.

D. Baars (phone: 586-850-5066 e-mail: baarsder@msu.edu).
photos by directly measuring the length of the inside and outside edges of the bent sample and comparing them to the undeformed material.

Another sample was prepared by halving the circular disk from the center of the large grain niobium slice, which had two grain orientations indicated in Fig. 2 across the boundary of the two grains. One half was flipped and EB welded back together, in the direction from left to right, such that the weld had three different grain misorientations along its length, including two triple points (Fig. 2).

Both samples were mechanically polished, ending with 0.03μm colloidal silica OP-S Suspension (Struers, Denmark) solution. The samples were then electropolished for 15min; initial scans revealed effects of many scratches from mechanical polishing still present, so the samples were electropolished for another 15min. Both specimens were examined with a Link Systems (TexSEM Laboratory) orientation imaging microscopy (OIM) system (version 4.2) installed on a CAMSCAN 44FE.

III. RESULTS

A. Bent Sample

The crystal orientation of the sample had <111> roughly parallel to the axis of bending (perpendicular to the examined surface), an orientation that provides six opportunities for [110] slip along a direction 55° from the plane of the sheet, or a multi-slip condition. Next to the weld, the curvature of the bent sample is the same as the initial portion of the large ring used for the experiment, implying that there was essentially no strain in this region.

Fig. 3 shows orientation maps of the surface in the areas indicated in Fig. 1 (after the second electropolishing procedure). Adjacent to the weld (left side), the color is uniform, but the effects of deep subsurface shear from scratches imposed by the grinding process caused unindexable or rotated lattice locations that make the surface appear scratched, even though the surface was very smooth. The apparent scratches were more severe after the first electropolish (not shown). In the region of highest curvature, where the temperature reached at least 700°C, there was no evidence of a change in crystal orientation (~16% strain along the inside of the curve, and ~14% strain along the outside). There was a smooth orientation gradient from the inside to outside edge of the bent region (right side), but no measurable boundaries (other than effects of scratches that occurred after the heating). The color shows a gradual change due to smooth orientation gradients. Plots in Fig. 4 show the cumulative misorientation from the inner edge to the outer edge in both scanned areas; next to the weld there is little change (other than bumps resulting from scratches), while there is a systematic but smooth change in crystal orientation in the bent region. At the bottom of the EB heated area very close to the weld, there were some small grains with a different orientation along the outside radius (these were visible and had the same orientations after both the first and second electropolishing steps, see Fig. 3 and right end of Fig. 4 top).

B. Weld Sample

OIM maps of the weld are shown in several sections in Fig. 3 of low angle boundaries. All scans in this figure are 20μm step size.

Figure 3. Left: OIM scan adjacent to the EB heating, no orientation gradient from inner to outer edge (top of image to bottom, 5.9mm). Note small grains along outside edge. Right: OIM scans of bent region showing smooth orientation gradient from inside to outside edge (5.9mm left to right), and no recrystallization and lack of low angle boundaries. All scans in this figure are 20μm step size.

Figure 4. Top: Cumulative misorientation from inner to outer edge is minimal near weld. Bottom: Cumulative misorientation from inner to outer edge varies smoothly at bend. Both: Spikes are due to subsurface damage from scratching during polishing. Euler angles are Bunge notation.
For much of the weld, the join line was straight and maintained the misorientation of the two crystal orientations. This implies that epitaxial growth of each parent grain occurred to form a straight grain boundary.

When the weld reached triple point locations, new orientations crystallized within the fusion zone. In the first triple point reached by the weld, only one new orientation was observed. When the weld reached the second triple point, many new orientations were observed. The orientations of the new grains were random; the $<100>$ easy-growth direction was not observed perpendicular to the solidification pool edge. Most of the boundaries were high-angle ($>20^\circ$), and not of a special type such as coincident site lattice. The orientations of the parent and new orientations, along with angle/axis misorientations with the parent grains, are given in Table 1.

IV. DISCUSSION

A. Bent Sample

No evidence of recrystallization was found in the bent sample, except for the very small region at the outer edge next to the weld, which was undeformed. Two interpretations for these small grains are possible: The small grains may not have been recrystallized grains, since they are in an undeformed region (no strain), and there was no recrystallization even in the region of much more deformation. The same orientations were present after a second electropolish, suggesting that these are not artifacts of specimen preparation. If they were not recrystallized then they may have been pre-existing. However, because the slice from the ingot showed very large grains (Fig. 1), it is unlikely that such small grains were pre-existing. A second interpretation is that the outer corner may have been nicked or dented during handling, but the lack of growth into the rest of the specimen indicates a lack of driving force for continued growth, implying that recrystallization was limited to only the small highly deformed area. This suggests that recrystallization may be possible, but that insufficient strain was present in this specimen to cause nucleation.

While the strain of the bent sample was $\sim$14%, more than 40% is required to expand the circumference of this iris during deep drawing (initial radius of $\sim$2.76cm is stretched to $\sim$3.94cm), so much larger strains are likely in cavity fabrication. Furthermore, the effect of the initial crystal orientation on deformation processes is unknown, as to whether dislocations accumulated within the material or if most of them escaped from the free surface. Consequently, further deformation experiments with different crystal orientations, and with bicrystals are underway, and the results will be reported in a future paper.

B. Weld Sample

Where there are only two grains along the weld, epitaxial growth to form a straight grain boundary was observed. However, at the triple points, heterogeneous crystallization took place. The size of the weld fusion zone is superposed on the second triple point, indicating that three orientations nucleated at about the same time, and grew until they ran into epitaxially growing parent orientations. The new grains in the weld bulge into the parent grains, suggesting they formed at the solidification interface and grew into the parent grains, then competed for growth with each other and the parent orientations as solidification progressed. The new grains at the triple points may have formed from detached dendrites from the parent grains that flowed within weld pool, and became attached at the solidification interface and then grew as the area cooled.

Single crystal weld literature on nickel super-alloys indicates that, in general, low power and high weld speed reduce stray grain formation. While local grain orientation does affect stray grain formation, over the length of a weld it is not significant. Using the methods described by Vitek et al., it may be possible to determine if the same generalizations are
true for niobium, and hence, determine the best orientations and welding parameters for niobium. [4,5].

V. CONCLUSION
This preliminary data is encouraging, suggesting that minimal generation of new grain orientations during EB welding may be practical. However, more work with different orientations is needed to determine how tolerant Nb is for maintaining a flat solidification interface or resisting recrystallization, particularly as a function of initial strain and crystal orientation. Less than 0.14 strain did not introduce significant recrystallization during the EB welding of single crystal cationic activity at bicrystal boundaries, resulting in a straight grain boundary. A solidification microstructure with random crystal orientations can develop in the fusion zone during the welding as a result of heterogeneous nucleation near a tri-crystal boundary. The random grains in the weld don’t follow special boundary orientation relationships with the base metal grains they are contact with.

ACKNOWLEDGMENT
This work has been supported by the Superconducting Radio Frequency Cavity Development Program at the National Superconducting Cyclotron Laboratory at Michigan State University. TRB appreciates sabbatical support from Michigan State University and the Max Planck Institute für Eisenforschung in Düsseldorf, Germany.

REFERENCES

Table 1. Bunge euler angles of measured grain orientations and angle-axis misorientations between adjacent grains in electron beam welds

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Euler Angles</th>
<th>B-purle</th>
<th>A'-lavender</th>
<th>B'-bluegray</th>
<th>A-turquoise</th>
<th>other</th>
</tr>
</thead>
<tbody>
<tr>
<td>A'-lavender</td>
<td>58 35 29</td>
<td>12.9@</td>
<td>27.8@</td>
<td>82 48 24</td>
<td></td>
<td></td>
</tr>
<tr>
<td>B'-bluegray</td>
<td>84 49 26</td>
<td>33.2@</td>
<td>20.9@</td>
<td>47.3@</td>
<td>26.5@</td>
<td></td>
</tr>
<tr>
<td>1-pink</td>
<td>68 32 27</td>
<td>13.7@</td>
<td>10.1@</td>
<td>27.8@</td>
<td>9.42@</td>
<td>4.73@</td>
</tr>
<tr>
<td>2-light blue</td>
<td>63 37 31</td>
<td>11.9</td>
<td>1.0</td>
<td>26.5@</td>
<td>12.5</td>
<td>1.45</td>
</tr>
<tr>
<td>3-pink</td>
<td>45 29 26</td>
<td>11.0</td>
<td>1.0</td>
<td>3.4</td>
<td>1.26</td>
<td>2.2</td>
</tr>
<tr>
<td>4-red</td>
<td>66 65 53</td>
<td>65.1@</td>
<td>11.4</td>
<td>6.4</td>
<td>11.4</td>
<td>6.4</td>
</tr>
<tr>
<td>5-salmon</td>
<td>37 74 14</td>
<td>46.1@</td>
<td>58.8@</td>
<td>-1 5.8</td>
<td>3.4</td>
<td>6-magenta</td>
</tr>
<tr>
<td>6-magenta</td>
<td>52 75 69</td>
<td>44.3@</td>
<td>4.73</td>
<td>1.38</td>
<td>3.2</td>
<td>4'-red</td>
</tr>
<tr>
<td>7-orange</td>
<td>68 15 1</td>
<td>56.1@</td>
<td>4.73</td>
<td>30.2</td>
<td>-1 26.2</td>
<td>6-magenta</td>
</tr>
<tr>
<td>8-yellow</td>
<td>22 61 9</td>
<td>64.7@</td>
<td>1.39</td>
<td>13.9</td>
<td>15.1</td>
<td>7-orange</td>
</tr>
</tbody>
</table>