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Exploration of Large Crystal and Single Crystal Niobium Material

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Explore various properties of single crystal niobium to examine its suitability for fabrication of superconductive RF cavities.

I. INTRODUCTION

Large crystal Niobium has been used recently for some SRF cavities. We want to explore single crystal niobium to see if material has a better Critical Field and/or Residual Resistance. In this experiment we will explore the crystal orientation of single crystal niobium and how it is affected by different processes. We will explore the effects of Electron Beam Welding (EBW) on single crystal niobium and how the crystal orientation is affected. We will study the effect of plastic deformation on the crystal orientation of single crystal niobium. Three samples of single crystal niobium are used for this experiment, samples 1, 2, and 3 respectively. Sample 1 is bi-crystal niobium with single crystal on either side of the grain boundary. Sample 2 is single crystal that underwent plastic deformation. Sample 3 is two pieces of single crystal that were cut from the same parent crystal then electron beam welded together.

II. EXPERIMENTS AND RESULTS

A. Single Crystal With Heat Treatment

Sample 1 was BCP 1:1:2 etched for 20 minutes at room temperature. The approximate rate of etching is 2μm per minute. Sample 1 was then studied under an optical microscope. Fig. 1 is a bi-crystal niobium virgin sample with single crystal on both sides of the known grain boundary. The Laue pattern is a typical representation for single crystal niobium material.

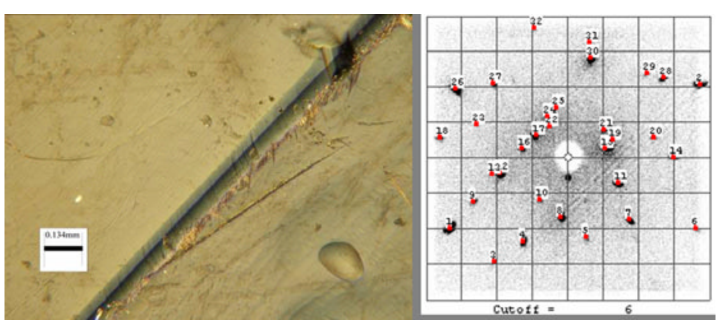

FIG. 1: sample 1 with known grain boundary, back reflection Laue method pattern taken from above grain boundary

In order to study the orientation of the crystals, the back reflection Laue method for x-ray diffraction was used. In the Laue method a beam of white radiation, the continuous spectrum from an x-ray tube, is allowed to fall on a fixed single crystal. The Bragg angle θ is therefore fixed for every set of planes in the crystal, and each set picks out and diffracts that particular wavelength which satisfies the Bragg law for the particular values of d and θ involved. Each diffracted beam thus has a different wavelength. Sample 1 then was heat treated at 900 C in a vacuum for 1 hour. After the heat treatment, sample 1 was re-examined under the optical microscope and the back reflection Laue method was used to determine the crystal orientation. This is represented in Fig. 2. The Laue pattern is very similar to the pattern prior to heat treatment.

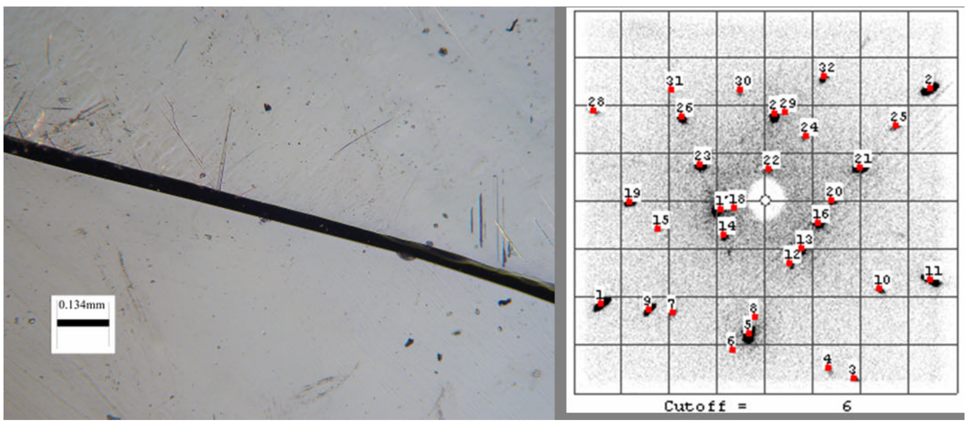

FIG. 2: sample 1 after 900 C heat treatment for 1 hour with known grain boundary, back reflection Laue method pattern taken from above grain boundary

Sample 1 then went through another heat treatment, this time at 1400 C in a vacuum for 2 hours. After the 1400 C heat treatment, sample 1 was examined under the optical microscope again and the back reflection Laue method was used to determine the crystal orientation. Fig. 3 is a sample of bi-crystal niobium that underwent a 1400 C heat treatment for 2 hours in a vacuum. Size of Laue pattern spots are similar in size to virgin sample. Surface of niobium seems to be more pure compared to virgin sample.

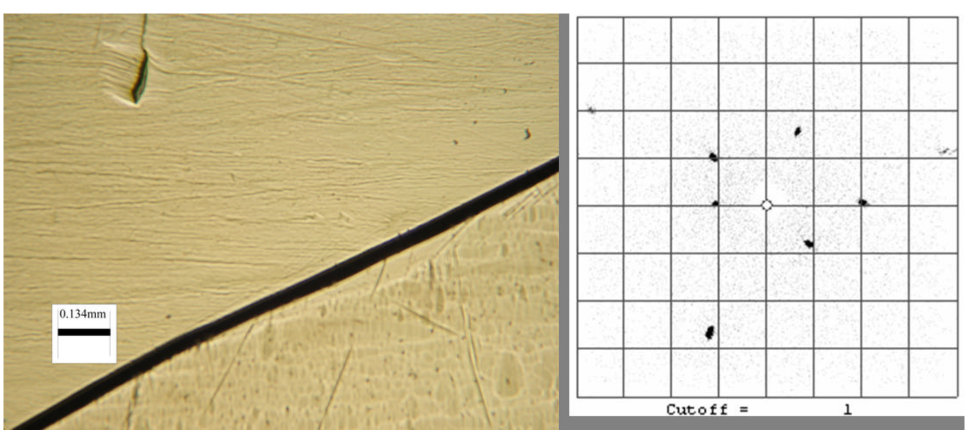

FIG. 3: sample 1 after 1400 C heat treatment for 2 hours with known grain boundary, back reflection Laue method pattern taken from above grain boundary

B. Plastically Deformed Single Crystal With Heat Treatment

Sample 2 underwent plastic deformation to expand the area of the niobium. Used an optical microscope to study the changes to the surface of the single crystal niobium. Then the back reflection Laue method was used to understand the crystal orientation. Fig. 4 is virgin sample 2 after the plastic deformation process. The surface of sample 2 has pits and valleys which are speculated to be caused by airborne particles or deformities in the rollers. The shape of the Laue spots have become larger and more elliptical in appearance. The size of the Laue spots indicates that the single crystal niobium fragmented into micro scopic domains that are different from the original single crystal orientation. A visual representation of this is shown in Fig. 5.

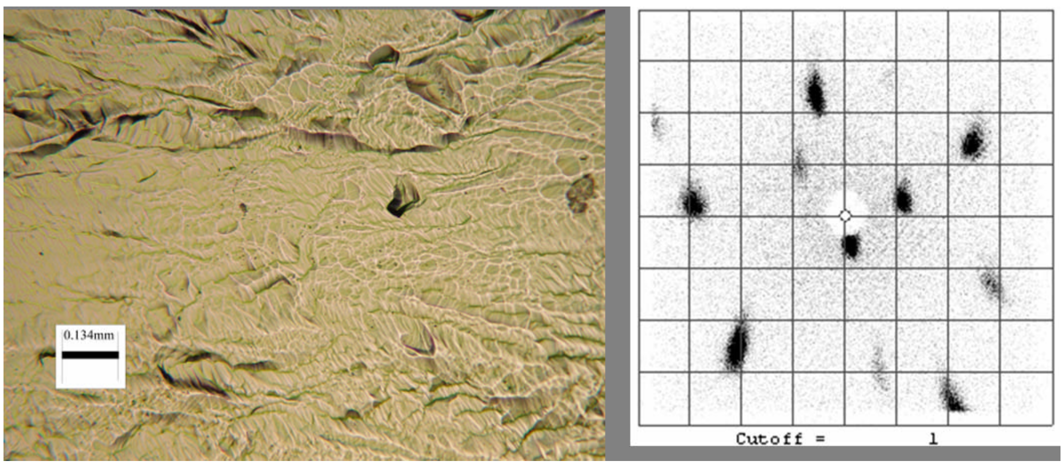

FIG. 4: sample 2 after plastic deformation and back reflection Laue method pattern

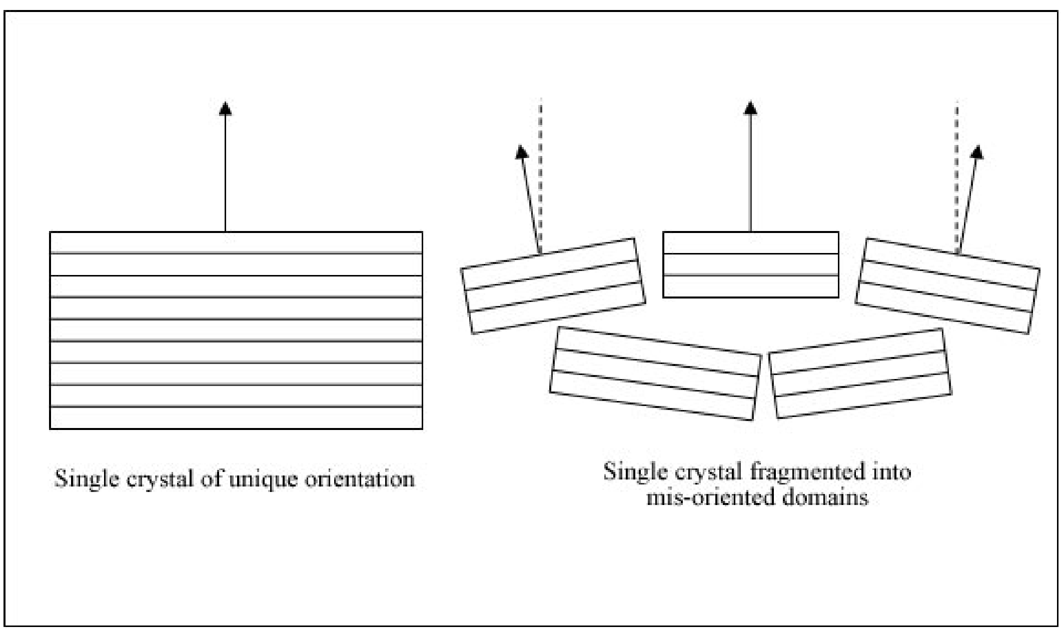

FIG. 5: visual representation of fragmented single crystal niobium

Sample 2 had a 900 C heat treatment for 1 hour in a vacuum. An optical microscope was used to study the surface of sample 2 and the back reflection Laue method was used to determine the crystal orientation. Fig. 6 is a picture of this process. The Laue spots seem to remain the same size and orientation as the virgin sample 2

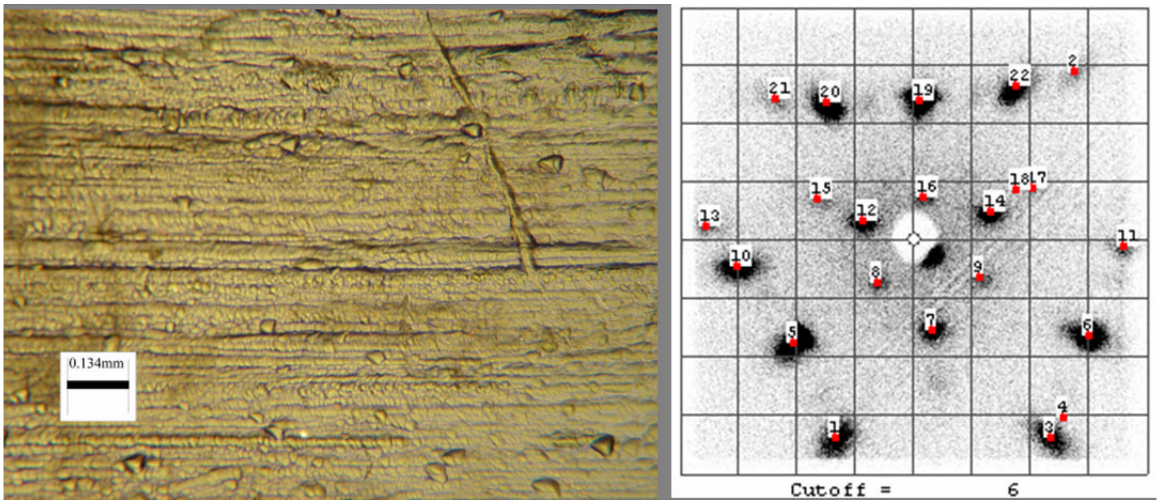

FIG. 6: sample 2 after plastic deformation and 900 C heat treatment for 1 hour and back reflection Laue method pattern

Fig. 7 is sample 2 after a 1400 C heat treatment for 2 hours in a vacuum. After this process, the sample was re-examined under the optical microscope to study the surface. The back reflection Laue method was used again to understand the crystal orientation. Upon visual examination of the Laue spots collected, the crystal orientation appears to have a similar structure as virgin sample 1 in Fig. 3. This would suggest that after a 1400 C heat treatment for 2 hours, plastically deformed single crystal niobium undergoes a crystal re-orientation.

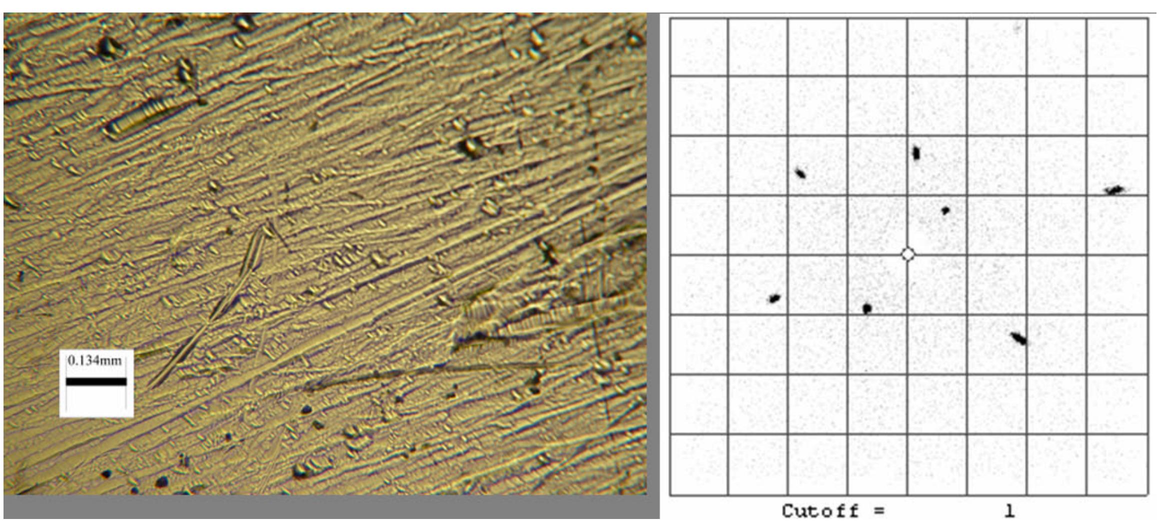

FIG. 7: sample 2 after plastic deformation and 1400 C heat treatment for 2 hours and back reflection Laue method pattern

C. Single Crystal Niobium Joined by Electron Beam Welding

Sample 3 consists of two pieces of single crystal niobium that was electron beam welded together. These two pieces were extracted from the same parent single crystal niobium. Fig. 8 shows two pieces of single crystal niobium that were cut from the same parent crystal then electron beam welded together. The non-heat affected zone, or the regular area, is opposite the welded region. The heat affected zone, near the welded region, shows signs of new grain boundaries that extend outward. The angle at which they extend seems to be proportional to the angle of the ripples in the welded region.

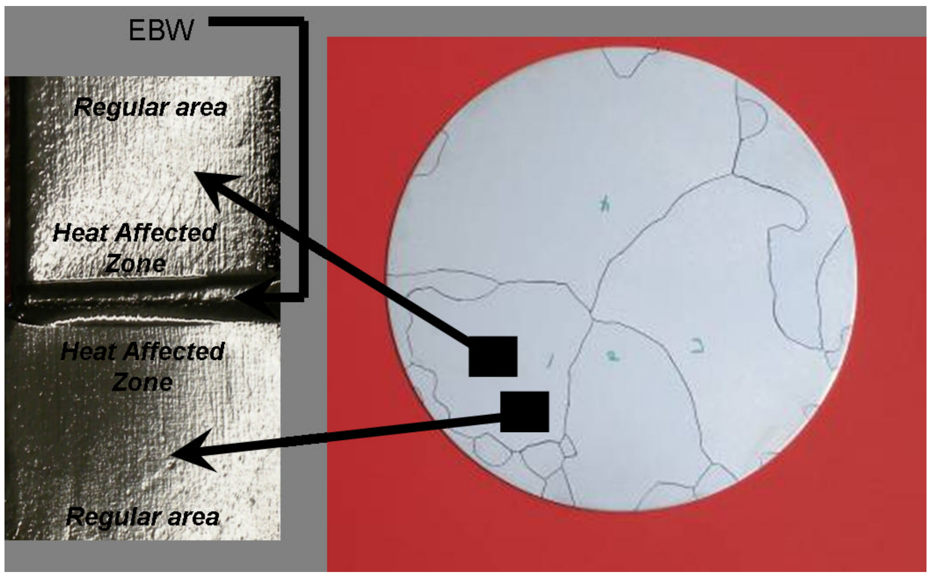

FIG. 8: two pieces of single crystal niobium that were electron beam welded together. These two pieces were extracted from the same parent single crystal niobium

Electron beam welding (EBW) is a fusion welding process in which a beam of high-velocity electrons is applied to the materials being joined. The work pieces melt as the kinetic energy of the electrons is transformed into heat upon impact. The electron beam gun used in EBW both produces the electrons and accelerates them, using an emitter made of tungsten that emits electrons when heated. The electrons are then attracted to an anode inside the tool, where they collect and are then directed with magnetic forces resulting from focusing and deflection coils. These components are all housed in an electron beam gun column. Fig. 9 represents the non-heat affected zone of single crystal niobium after the EBW process. The Laue pattern is consistent with that of a virgin sample of single crystal niobium. Fig. 10 is a picture of the heat affected zone of single crystal niobium after the EBW process. The black lines that extend outward from the welded region were speculated to be new grain boundaries that were formed due to the EBW process in single crystal niobium. Sample 3 was BCP 1:1:2 etched first at approximately 40μm, then another approximate BCP 1:1:2 etching of 40μm was done. A final BCP 1:1:2 etching of approximately 30μm was performed. Between each chemical etchings tests were done to measure the approximate height of these line defects. Upon testing of the heat affected zone using profilometry instruments, the tests revealed that these defects were symetric under the surface of the single crystal niobium. Therefore, what were thought to be new grain boundaries are not, further testing will produce conclusive results as to the nature of these dislocations.

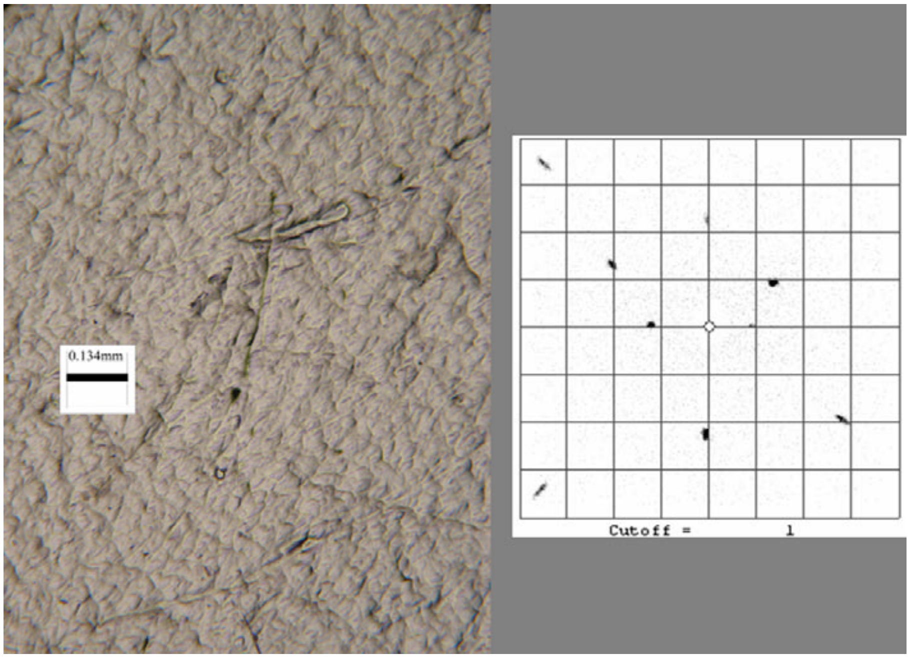

FIG. 9: non heat affected zone after the EBW process with back reflection Laue pattern

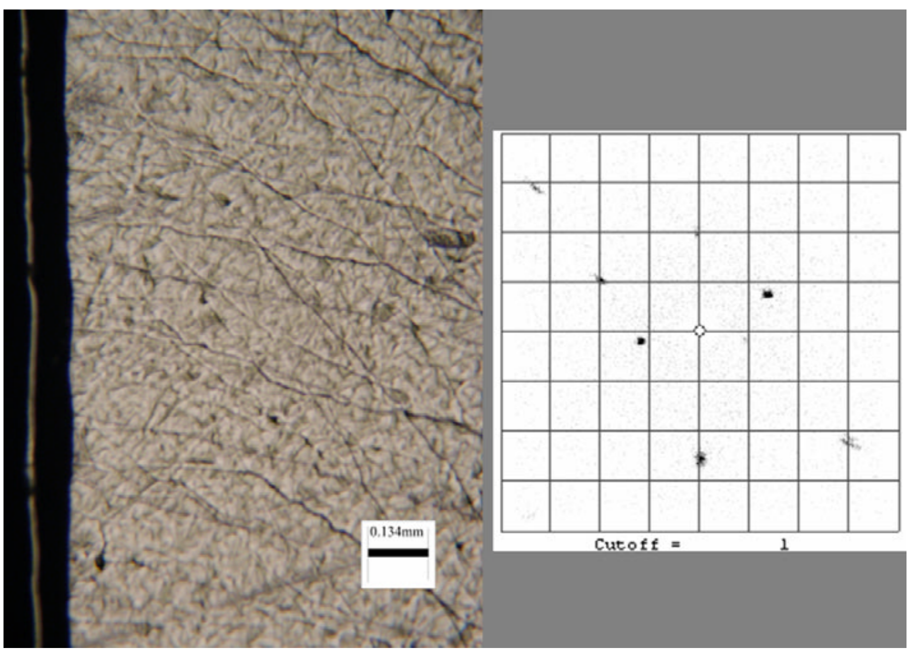

FIG. 10: heat affected zone with defects after EBW process and back reflection Laue pattern

Fig. 11 is the welded region of the two single crystals of niobium from the same parent crystal. In the center of the welded region, their appears to be re-crystallization from single crystal to a poly crystal based on visual inspection under an optical microscope. The Laue pattern from this region is similar to that of a Laue pattern of a known poly crystal shown in Fig. 12.

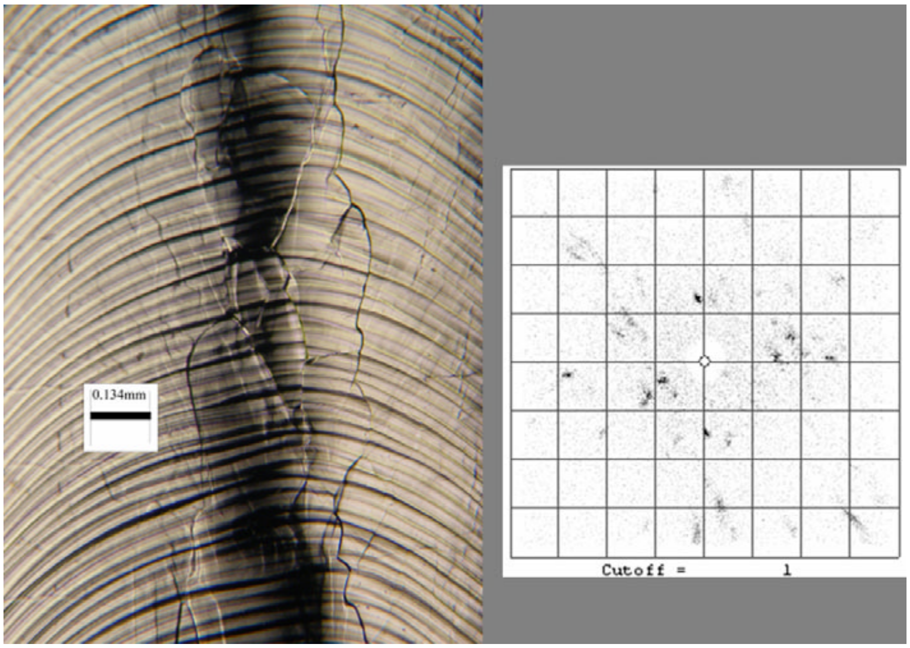

FIG. 11: welded region of two single crystals of niobium with re-crystallization into poly crystal and back reflection Laue pattern

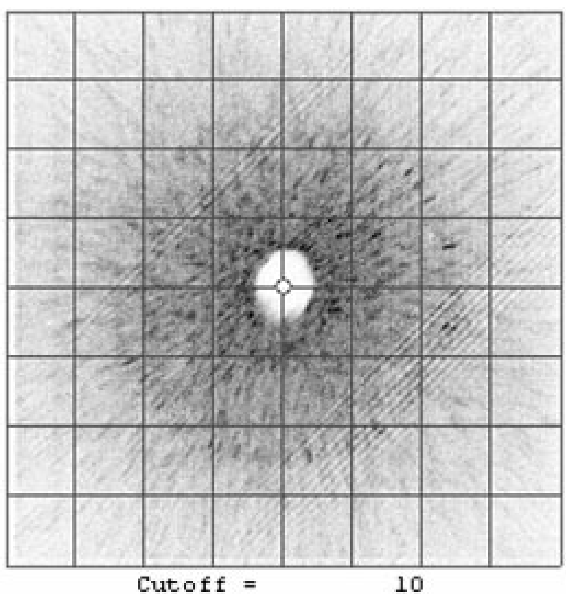

FIG. 12: back reflection Laue pattern of a known poly crystal

D. Summary of Crystal Orientation

TABLE I: chart shows the degree rotation needed to find the 110 crystal orientation for samples 1, 2, and 3.

Process	**X-Axis Rotation for 110** (*in degrees*)	**Y-Axis Rotation for 110** (*in degrees*)
Sample 1		
no treatment	18.4	9.6
900 C heat treatment	18.4	10.2
1400 C heat treatment	18.6	8.2
Sample 2		
no treatment	24.6	30.2
900 C heat treatment	14.3	27.9
1400 C heat treatment	10.5	13.4
Sample 3		
non heat affected zone	16.6	0.6
heat affected zone	18.1	0.0
welded region	20.5	22.7

III. CONCLUSIONS

Sample 1 crystal orientation virtually unchanged throughout heat treatment process. This was not in contrast to what was speculated to happen.

Sample 2 showed mis-orientation in the crystal lattice after plastic deformation then returned to the original crystal orientation of a single crystal above a 1400 C heat treatment. A 900 C heat treatment showed no change in the mis-orientation due to plastic deformation. In order to re-orientate the crystal lattice in single crystal Niobium after plastic deformation, a minimum heat treatment of 1400 C for 2 hours in a vacuum is required.

After electron beam welding two pieces of single crystal niobium from the same large parent crystal, the non heat affected zone kept the same crystal orientation as a non EBW sample of single crystal niobium. The heat affected zone appears to have formed what seems to be new grain boundaries. Further testing of this region will conclusively determine whether new grain boundaries were formed. After the electron beam welding process of two single crystals, the welded region re-crystallized to poly-crystal. This is a finding in contrast to others and should be explored in more detail.

IV. ACKNOWLEDGMENTS

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