STUDY OF SLIP IN HIGH PURITY SINGLE CRYSTAL NB FOR ACCELERATOR CAVITIES*

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Abstract

SRF cavities can be formed by deep drawing slices from Nb ingots with large grains. Crystal orientation dependent slip system activities affect the shape change of ingot slices during deep drawing, and form a dislocation substructure that affects subsequent recrystallization and ultimately, cavity performance. Two groups of single crystal tensile specimens with different orientations were extracted from a large grain ingot slice. The first group was deformed monotonically to 40% engineering strain. Analysis suggests that slip on {112} planes controlled the work hardening behavior. The second group was heat treated at 800 ºC for two hours, and then 4 of them were deformed incrementally to 40% engineering strain using an in situ tensile stage. Crystal orientations and surface images were recorded at each increment of deformation. The results indicate that the heat treated group had lower yield strengths, and the rotations of crystals differed from corresponding specimens in the first group, indicating that different slip systems were active. Active slip systems were investigated and compared to the first group.

INTRODUCTION

Fundamental study of deformation in Nb dates back to almost 50 years ago, when several researchers used single crystal Nb to investigate active slip systems under various experimental conditions [1, 2]. However, due to the lack of industrial applications of Nb and the complexity of slip in body-centered cubic (BCC) systems, aside from the continuous efforts by Seeger et al. on slip in ultra-high purity Nb and other BCC metals [3], little in-depth attention has been given to Nb since then. The trend has changed in the past couple decades though, when Nb has become the material to fabricate superconducting radiofrequency (SRF) cavities for particle accelerators. Currently there are a number of research groups in the SRF community focusing on the material properties of Nb that are related to cavity performance.

One of the ultimate goals in cavity design is to establish a model that integrates the grain orientations of an ingot slice, slip activities during deep drawing, and dislocation substructure evolution due to slip system interactions, which governs recrystallization from heat treatment and welding. Then it would be possible to simulate/predict the final microstructure of a cavity (hence its performance) based upon the initial grain orientations [4].

In order to understand the slip system behavior during deep drawing of large grain Nb, it is first necessary to know how slip occurs in the uniaxial tensile deformation of single crystal Nb. Baars et al. has made initial investigation in this direction [5]. Some of these findings are highlighted in the results and discussion section, as a foundation for this work, which compares slip behavior of Nb in samples taken from an ingot slice, before and after an 800 ºC anneal.

MATERIALS AND METHODS

To investigate slip systems activities in Nb, 12 sets of single crystal specimens were extracted from a large grain Nb ingot slice (Fig. 1). Each set contained three parallel specimens from the same grain, so the three have similar crystal orientations. The orientations were chosen to initially favor either a single slip system or a particular combination of slip systems. A letter from O to Z is designated to each set of specimens, and for the three specimens in a set, number 1 denotes the specimen marked with a black dot, and numbers 2 and 3 follow downwards. Specimens from groups 2 and 3, O2 through Z2, and O3 through Z3, have been used for tensile tests of annealed and as-received conditions, respectively. The dimensions for the specimens are shown on the left of Fig. 1, which are a shrunk version of ASTM E8-04 standard for sub-size specimens.

Group 3 specimens were electropolished after being extracted from the ingot slice. Nine of them were deformed monotonically to 40% engineering strain using a tensile tester (Instron 4302), with a loading rate of 1 mm/min. Group 2 were also electropolished, and then heat treated at 800 ºC for two hours to remove preexisting dislocations formed during ingot production. Four specimens from group 2 have been deformed in situ inside a scanning electron microscope using an Ernest Fullam stage, with a strain increment of ~10% followed by characterization. Slip traces and grain orientations were recorded at each stage of deformation, at 3 locations along the gauge length. The engineering stress-strain curves are compared to those from group 3, to identify the effects of heat treatment on the deformation behavior.

RESULTS AND DISCUSSION

The engineering stress-strain curves for group 3 (without heat treatment) are shown in Fig. 2. The majority of specimens exhibited a stage of easy glide that is characteristic of single crystals, while specimen U3 had a stress-strain behavior that hardened more than a typical
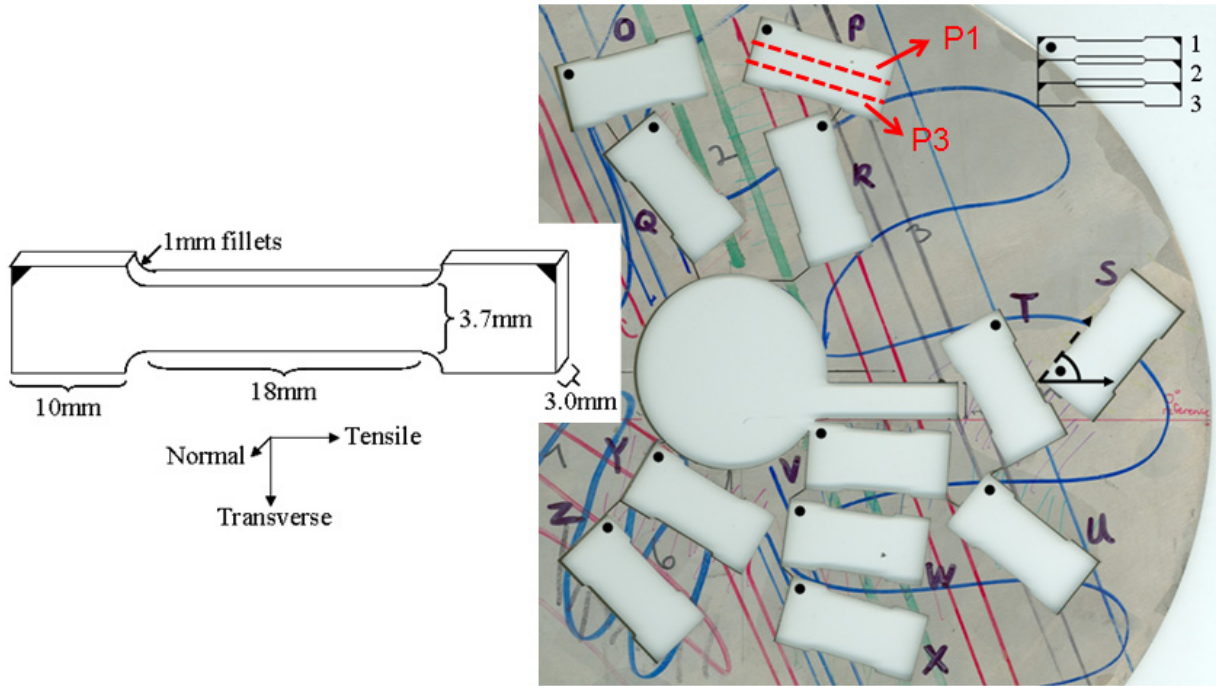
Figure 1: Layout of the tensile specimens from an ingot slice. Dimensions are shown on the left [5].
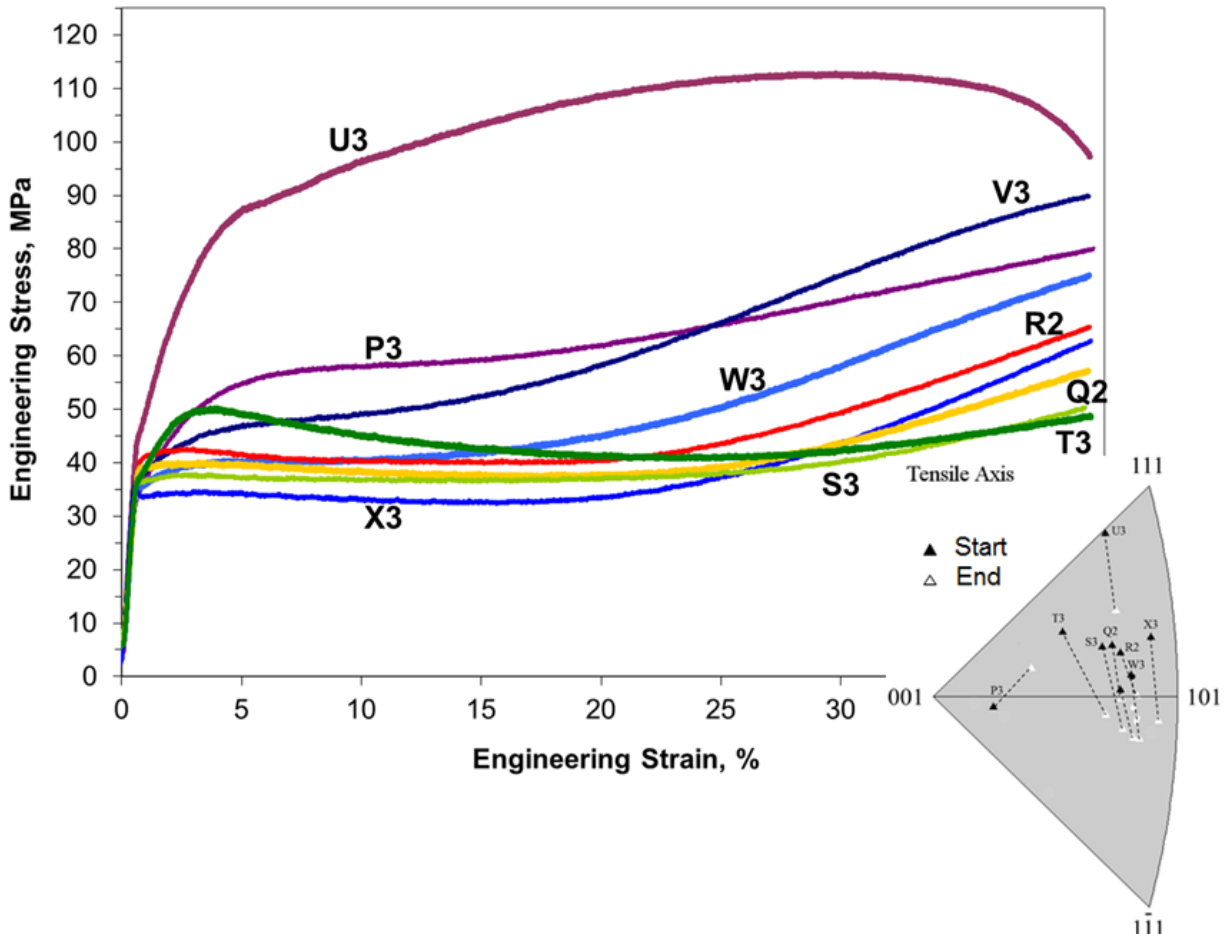

Figure 2: Engineering stress-strain curves for group 3 specimens (without heat treatment). The inverse pole figures on the lower right corner indicate starting and ending orientations for each specimen [5].

poly-crystal. The inverse pole figure on the lower right corner shows the starting (black) and ending (white) locations of tensile axes for each specimen.

One noteworthy specimen is T3 (green curve). It was oriented to initially favor a single [1-11](110) slip, so easy glide was expected. However, the fact that hardening started immediately after yielding contradicts the assumption of single slip. One possible explanation is that the two {112} slip systems with slightly smaller Schmid factors and different <111> slip directions were activated at the beginning instead of the (110) system. The interaction between dislocations slipping in different directions may account for the hardening because the different slip directions intersect with each other. In fact, the initial portion of the stress-strain curve of T3 resembles that of P3, which had an orientation that favors two {112} slip systems with different slip directions, suggesting that the two specimens had the same hardening mechanism. This suggests that {112} slip has an advantage over {110} slip in T3.

An analysis of the relationship between {110} and {112} systems revealed that the initial hardening rate for most of the 9 specimens is correlated with the relative Schmid factor values on the two most highly stressed {112} slip systems with different slip directions. Figure 3 shows that specimens with large differences between the initial resolved shear stresses on intersecting {112} slip systems tended to have low initial hardening, while specimens with small differences tended to have high initial hardening. The other two slip system interactions ({110} with {110}, {110} with {112}) were also investigated, but neither was correlated with the initial hardening rate. This further supports the argument that {112} slip was favored over {110} and dominated the deformation behavior.

In situ tensile tests were performed on heat treated specimens Q3, R3, T2 and W2. Figure 4 shows the engineering stress-strain curves for all of them, in comparison with their corresponding specimens without heat treatment. The noisy nature of the curves is due to a different load cell system with a lower resolution. After heat treatment, the yield and flow strengths decreased significantly for all 4 specimens, and there was no longer a slight drop of flow stress right after yield that is present in the specimens without heat treatment. This implies that the dislocations initially present in the ingot play an important role in deformation, and that the heat treatment removed most of these dislocations effectively.

Figure 5 shows the evolution of the tensile axes in the inverse pole figures for the 4 heat treated specimens during deformation. A comparison with the overlaid as-received specimens (initial and final orientations in black and white triangles) reveals that the deformation paths have a different rotation direction, even though the specimens have nearly identical starting orientations. This implies that preexisting dislocations affect which slip systems were activated during deformation, although the small differences in initial orientations might have also contributed to the divergence after 40% strain.

Figure 6 illustrates the orientation evolution with deformation for a heat treated specimen (R3) and its corresponding as-received specimen (R2). They started off with a small difference in orientations (a misorientation of 1.83°), but the as-received R2 had a much greater crystal rotation after 40% strain than R3. As a result, their ending orientations differ from each other by about 14°.
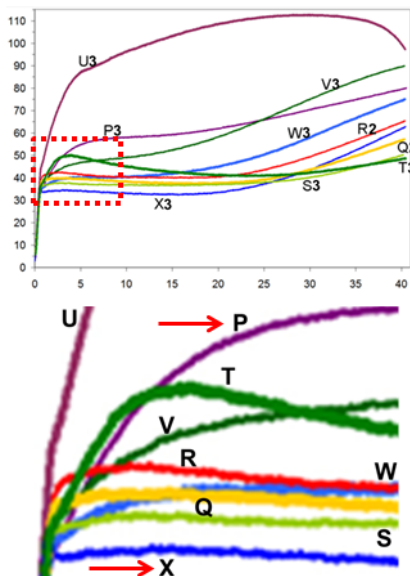
Specimen	Difference between highest resolved shear stress on primary and secondary {112} slip systems (MPa), and ratio of the two		Initial hardening rate
P3	0.1	1.042	High
V3	0.4	1.022	Moderate-High
U3	0.6	1.027	Very High
T3	1.3	1.073	High
W3	1.4	1.091	Moderate-Low
R2	3.5	1.200	Slight
S3	3.5	1.237	Barely
Q2	4	1.263	Barely
X3	4	1.316	Barely

Figure 3: The table on the right lists specimens in the order of decreasing differences between primary and secondary {112} slip systems, which corresponds to increasing initial hardening rates as shown in the zoomed image on the bottom left [5].
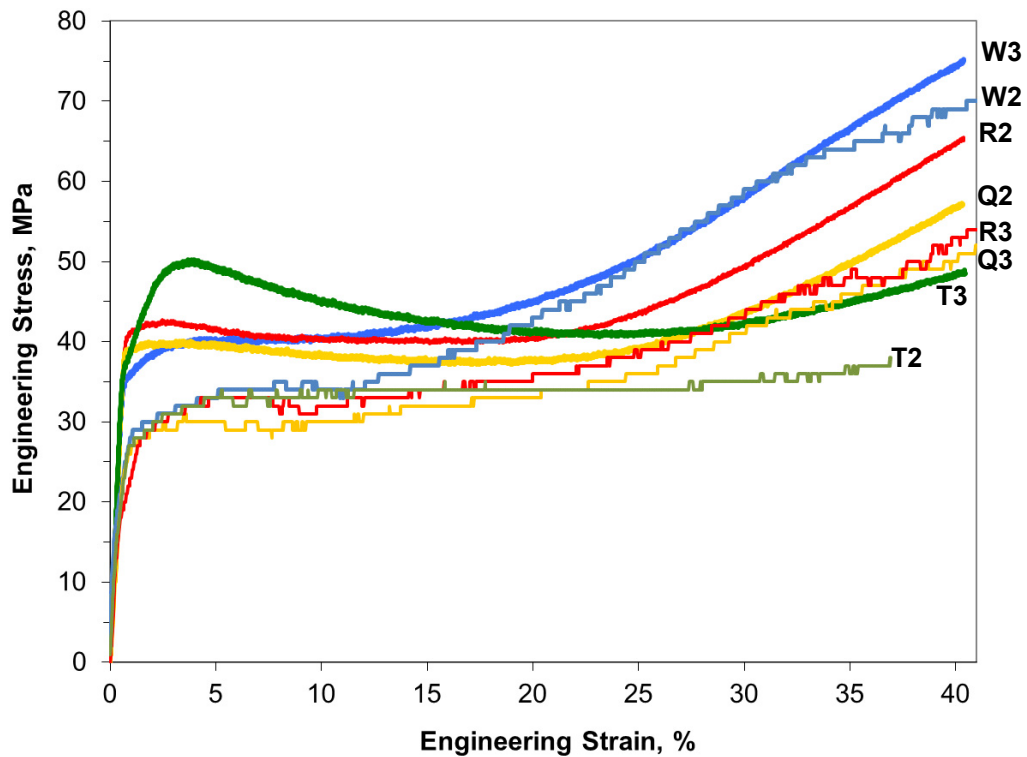
Figure 4: Engineering stress-strain curves for the 4 heat treated specimens deformed in situ ("noisy" curves), in comparison with corresponding specimens without heat treatment.
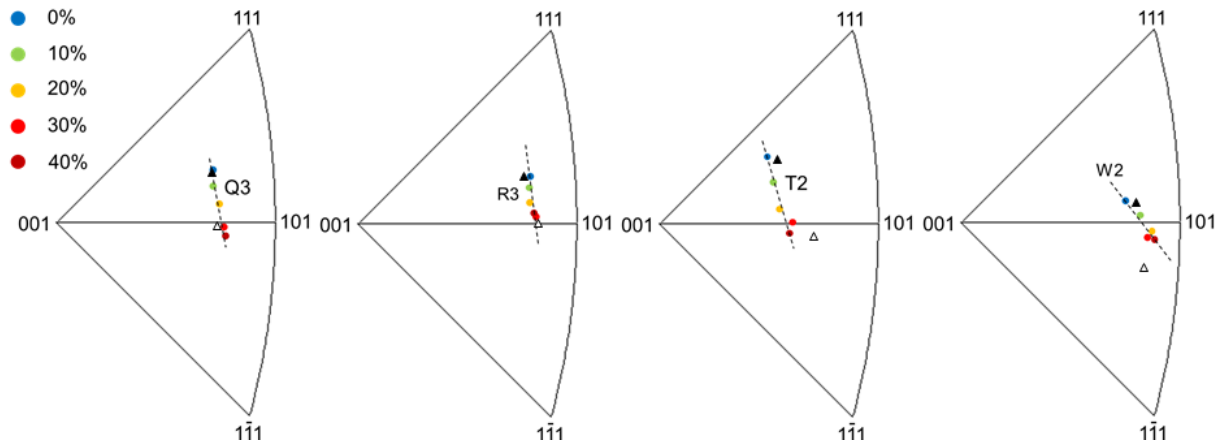

Figure 5: Inverse pole figures showing the evolution of tensile axes with deformation. The tensile axes for corresponding as-received specimens are overlaid with black (initial) and white (final) triangles for a comparison.
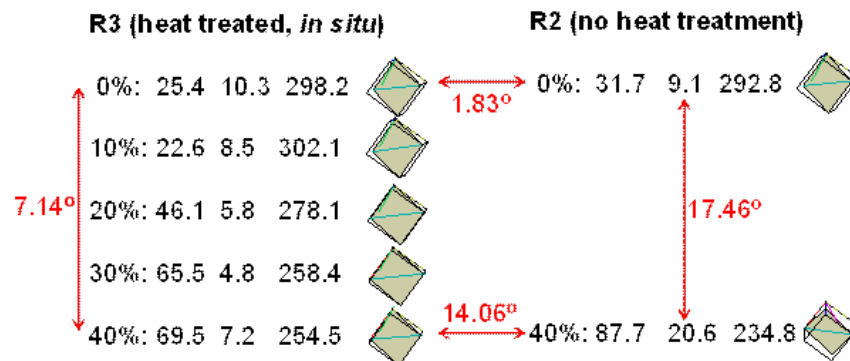

Figure 6: An illustration of orientation evolution with deformation for heat treated (R3) and as-received (R2) specimens. The orientation at each stage is presented in Bunge Euler angles ($\varphi1$ Φ $\varphi2$).

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Slip trace analyses were performed on the 4 heat treated specimens, an example of which is shown in Figure 7. Typically no slip traces were visible on the specimens until about 20% strain, suggesting that "homogenous" slip (where dislocations move through the crystal smoothly and exit the crystal freely) accounts for the lack of traces at low strains. Also, only {110} traces were observed on the 4 specimens, although more tests are needed to determine if this is a trend. The very linear traces differ from traces observed in the as-received specimens, which had a range of orientations that suggested bursts of slip in the same direction, but on different planes. These differences may be related to a greater amount of core relaxation in the as-received specimens, or the effects of internal strains that altered the core structure to favor {112} slip [6-9].
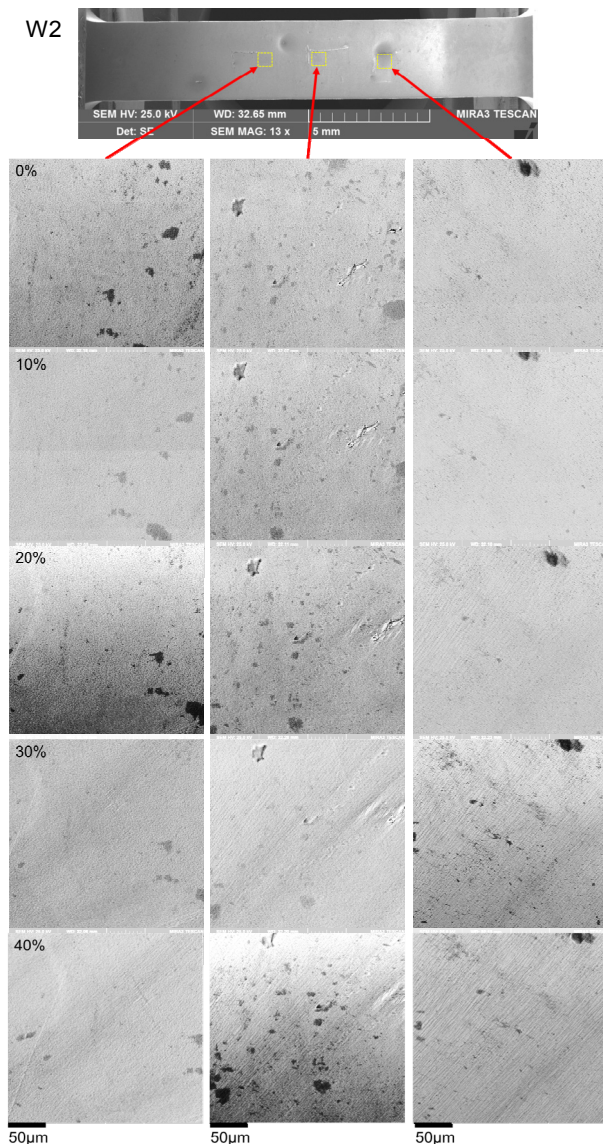

Figure 7: an example of slip trace evolution with deformation of 3 locations on heat treated W2.

CONCLUSIONS

Recent study of slip in as-received specimens sliced from an ingot has demonstrated that slip on {112} planes is favored over {110} under the uniaxial test conditions. A heat treatment alters the deformation behavior of single crystal Nb significantly. Homogeneous slip on {110} planes may account for the lack of observable slip traces at early stages of deformation. This implies that the prior strain history (and hence, the dislocation content) has a large effect on which slip systems are favored to operate.

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