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Flux Pinning in Alloys of Nb44-62wt%Ti having Similar Microstructures

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Abstract

Introduction

Although Nb-Ti alloys are still by far the most widely used superconductors, there is still much that is unknown about them. Development of high current density (J_c) is one of the most active areas of research and this thus focuses attention on the elementary flux pinning force (f_p) which occurs between vortices and the microstructural flux pinning defects. Unfortunately f_p cannot be measured directly in any microstructure of high J_c and f_p must be inferred from measurements of the global or bulk pinning force ($F_p = j_c \times B$) and from measurements of the pin density n. The issue of what summation process correctly connects f_p and n to F_p is always controversial. In the strong and dense pinning limit of practical alloys, a direct summation (i.e., $F_p = n f_p$) is generally accepted.

Identification of the significant pinning mechanism(s) which operate in a given alloy is crucial to further optimizations of Nb-Ti. Although the T_c and H_{c2} values in this system (9-9.5 K and ~ 11 T (4.2 K)) are modest by comparison with those available in the best oxide, Chevrel phase or A15 superconductors, the alloys are very ductile and strong. This confers great advantage in practical use. After a recent period of rapid increase in J_c, J_c now has reached a plateau value of order 3500-3800 A/mm^2 (5 T, 4.2 K). Whether this represents any intrinsic limit or is just a temporary control imposed by current methods of microstructure optimization is not clear. In any case, further increases in J_c of even 25-50% would have major technological impact. Unfortunately

flux pinning predictions are inaccurate at this level of precision.

The standard approach to determining the flux pinning mechanism has been to determine the shape of the flux pinning curve f(b), where $b = B/B_{c2}$ and to observe whether this remains constant (i.e., scales) when the curve is mapped out in reduced units (F_p/F_{pmax}) vs. B/B_{c2}. In earlier studies scaling was generally found. However a more recent study on a very carefully prepared, very high J_c sample showed that temperature scaling did not occur. In this composite, the starting alloy homogeneity was made very high and this led to a rather uniform α-Ti precipitate microstructure. Careful quantitative transmission electron microscopy (TEM) showed that J_c and F_p peaked when the precipitates were drawn out into highly elongated ribbons ~ 1 x ~ 100 nm in transverse section and several μm long parallel to the wire axis. The average separation of the ribbons was 4 nm. These dimensions are all small compared to the characteristic dimensions of the flux line lattice (FLL). The coherence length (ξ (4.2 K)) is 5.4 nm and the FLL lattice spacing a_o is 22 nm at 5 T. In this alloy a very marked change of pinning curve shape occurred as the temperature was raised. Whereas at lower temperatures, e.g., $t (\simeq T/T_c) \leq 0.5$, F_p was proportional to b (1-b), this relationship gradually turned into one of $b^{1/2} (1-b)^{3/2}$ as the temperature was raised towards T_c.

In the standard Ginzburg-Landau expression, the free energy perturbation at a defect is given by:

$$\delta E = \int \mu_o H_c^2 \left(- \frac{\delta H_{c2}}{H_{c2}} \mid \Psi \mid^2 + \frac{1}{2} \frac{\delta \kappa^2}{\kappa^2} \mid \Psi \mid^4 \right) dV \quad . \qquad \textbf{(1)}$$

or

$$E = \int \mu_o H_c^2 \left[- \frac{\delta H_c}{H_c} \mid \Psi \mid^2 - \frac{\delta \kappa}{\kappa} \left(\mid \Psi \mid^2 - \mid \Psi \mid^4 \right) \right] dv \qquad \textbf{(2)}$$

Thus the flux pinning can be attributed to perturbations of either κ or H_c. In the case of the optimized Nb48wt%Ti alloy studied by Meingast and Larbalestier, it was argued that both $\delta\kappa$ and δH_c terms contribute to the pinning at lower temperatures but that the δH_c term increasingly outweighs the $\delta\kappa$ term as T tends towards T_c. By calculating the core pinning interaction (i.e., the δH_c interaction), measuring the precipitate density and assuming full summation, the global pinning force F_p could be derived. Excellent agreement with experiment was found: moreover the expected thickness (t') dependence of f_p (i.e., $f_p \alpha t'$) for precipitates less than 2ξ thick was found. However this agreement was most accurate in the regime where T tends to T_c. Unfortunately, although this agrees very well with the calculation and the postulate that core pinning via the δH_c effect dominates in this region, it does not agree with the field dependence of F_p. As equation 2 shows, the core pinning term contains $|\psi|^2$ which should vary as (1-b). In the limit $T \rightarrow T_c$, F_p in fact varies as $(1\text{-}b)^{3/2}$, thus casting doubt on the completeness of the above conclusion.

Fortunately another possibility of checking the validity of the conclusions of Meingast and Larbalestier appears to be available. Just as the relative strength of $\delta\kappa$ and δH_c change with temperature for given alloy (e.g., the Nb48wt%Ti of the Meingast, Larbalestier study), so too does the relative strength of $\delta\kappa$ and δH_c vary with alloy content at constant reduced temperature. A detailed study of Parek et al. had earlier shown that the pinning curve of Nb62wt%Ti was much closer to a $(1\text{-}b)^2$ than to a (1-b) dependence. A preliminary study by Warnes and Larbalestier showed a gradual change from (1-b) to $(1\text{-}b)^2$ type behavior as the Ti content was increased. Unfortunately, neither study had any microstructural component. Since there is abundant evidence that the microstructure changes significantly over this range, no firm conclusions can really be

drawn from the study. For example Evetts and Plummer and Dew-Hughes have both proposed that the very elongated, laminar precipitates produced in optimized Nb45-50wt%Ti are responsible for the strong core pinning and the (1-b) dependence. This view has been contested by Küpfer and Matsushita since they observed that a (1-b) dependence was observed whether or not the flux lines were parallel to the ribbons or, as is the usual case, orthogonal to them. The purely shape-related origin of the (1-b) dependence also appears incompatible with the results of Meingast and Larbalestier, since only a change of temperature is needed to change the shape of the curve.

Thus to summarize briefly a rather complex story, it is apparent that, in spite of many advances in raising the F_p of Nb-Ti conductors, the basic details of the flux pinning behavior are unclear. The upper limit to J_c is given by the depairing current density (J_d), given by H_c/λ. Thus J_d (4.2 K) for Nb48wt%Ti is (λ (4.2 K) = 240 nm, $\mu_o H_c$ = 24 T). Observed values of J_c (H=0) are of order 2×10^4 A/mm^2, thus showing that optimized Nb-Ti conductors fall about a factor of ??? below their upper limit. By contrast the best oxide superconductor thin films have J_c values of greater than 10% J_d. Potentially therefore there still exists considerable opportunity for raising the J_c in Nb-Ti. Finding out which is the key operating pinning mechanism and how best it can be brought into operation presently seems the key to further advances. This paper describes a study of the flux pinning behavior in Nb-Ti alloys covering the alloy range 44 to 62wt%Ti. The microstructures were also controlled so as to be as uniform as possible from one alloy to another. In this way it was hoped to check the hypothesis that the reason for the change in pinning curve shape with alloy content was due to the increasing role of δH_c pinning rather than to morphological factors.

Experimental Design

(i) Alloy fabrication

The goal was to obtain a set of alloys, which covered the alloy range from high Nb content to high Ti content and whose chemical homogeneity and size was suitable for composite conductor

manufacture. These requirements are rather onerous. A fundamental requirement was to obtain similar microstructures. Careful studies within our group have shown that very high true strains ($\epsilon = 2 \ln (D_0/D)$, where D_0 is the last recrystallization diameter and D the cold-worked diameter) are needed to suppress ω phase and Widmanstätten (i.e., needle-like) α-Ti. For ~ Nb47wt%Ti this pre-strain ϵ_p is ~ 5 but it rises approximately linearly with composition, reaching ~ 12 at NbXXwt%Ti. These strains dictate that the alloys be industrially melted. Given that it is desirable to break up the cast ingot structure by working and extrusion, melting on a scale of at least 150 mm diameter is indicated. Such an ingot can then be extruded to 38-42 mm diameter and then recrystallized to a fine grained structure. Since a strain of 12 is equivalent to a diameter reduction of 400, this produces a filament size of order 100 μm. Heat treatment and subsequent drawing generally requires at least another strain of 5 ($D_0/D \simeq 12$) to be optimum, thus reducing the filament diameter to ~ 8 μm. This is right at the limit where Nb-Ti filaments having no Nb diffusion barrier can work without filament sausaging and such composites are clearly unsuitable for basic flux pinning studies.

These requirements dictated the alloy selection whose details are given in Table I. Alloys of 44, 46.5, 49, 52, 53, 58 and 62Ti were used in the study. All analyzed compositions fell within 0.5wt%Ti of these nominal compositions. Trace element analyses fell within standard tolerances. For example, oxygen varied from 590 to 660 ppm by weight. The alloys came from 3 different batches of Teledyne Wah Chang Albany's production. Two alloys Nb46.5wt%Ti and Nb53wt%Ti were melted on a full industrial scale (diameter > 15") and were of standard chemical homogeneity. The remaining alloys were specially commissioned for this study and were melted at 6" diameter. The first batch (Nb44, 58 and 62Ti) were extruded to 1.5" dia, while the second were extruded to 1.625" dia. Both were recrystallized at this size and drawn down to 0.28" prior to delivery to us. Thus D_0 was 1.5" for all alloys except Nb44 and 52wt%Ti, for which it was 1.625". We defer discussion of the difference between standard and high homogeneity alloy until later.

The alloys were made into 61 filament conductors in our laboratory. Careful stacking and sheathing of the Nb-Ti alloy led to a rather uniform filament array (do we have a figure to put in here?) In order to avoid precipitation of ω or needle-like α-Ti, the first heat treatment was delayed until the strain was ~ 12. This large degree of cold work prior to heat treatment ensures uniform grain boundary triple point precipitation in Nb44wt%Ti to Nb58wt%Ti, based on the previously observed relationship between pre-strain and precipitate morphology.[8] However, it was not expected to be sufficient for the Nb62wt%Ti alloy, a compromise being necessary in order to provide sufficient final strain within the limits of the available strain space.

The goal of the heat treatments was to produce microstructures which were as similar as possible. Having attempted to ensure only grain boundary α-Ti nucleation, we then scaled the heat treatment temperature at fixed time (40 hrs), such that a constant diffusion amount would occur. The input data to the calculation was a summary by Moffat of diffusion data for Nb in Nb-Ti alloys[9] from a wide variety of sources[10-18]:

$$D = D_o e^{\left(\frac{-Q}{RT}\right)} \left(cm^2/sec\right) \quad \textbf{(3)}$$

$$\text{where} \quad D_o = 5.5783 \times 10^{-4} e^{(8.209x)} \left(cm^2/sec\right) \quad \textbf{(4)}$$

$$\wedge \quad Q = 31463 + 65674x \ (cal/mol) \quad \textbf{(5)}$$

where x is the atomic fraction of Nb. The calculation was based on the diffusion of Nb since $D_{Nb} << D_{Ti}$ and the Nb diffusion is thus the rate limiting step.

The baseline treatment that we wished to model was that of 40 hrs at 375°C, which we know produces a rather uniform grain boundary layer of α-Ti which is 2-4 nm thick. This then grows out to produce approximately equiaxed precipitates which are 20-40 nm in diameter in transverse cross-section. The heat treatment temperatures resulting from this calculation are listed

in Table 2 and each of the wires was given a 40 hr heat treatment at this temperature. Further cold drawing strains were administered following the heat treatment. Except for the Nb62wt%Ti alloy, samples were taken at a final strain of $\epsilon_f = 0$, 1, 2, 3, and 4. In the case of the Nb62wt%Ti alloy, the wire was too brittle to be drawn further following heat treatment.

(ii) Microstructural examination

Microstructural measurements were made on the samples using a JEOL 200CX (scanning) transmission electron microscope (STEM/TEM). Samples were prepared for TEM analysis as follows. First the outside diameter of the wire was modified to 3 mm by etching the copper away on the large monofilaments, or by plating copper onto the finer wires. Transverse cross sections of the wire were cut using a diamond saw. These sections were ground down until they were 100 μm thick. Further thinning of the specimens was performed using a jet electropolishing apparatus with a solution of 2vol% HF, 5vol% H_2SO_4 and 93vol% Methanol. The polishing conditions were -40°C, 1.8 mA/mm^2, and 200V. Ion milling was performed on selected specimens as needed.

Quantitative analysis of the TEM micrographs was performed using a high resolution (1024 x 1024 pixels x 256 grey shades) MegaVision image processing system. Using this system micrographs of the same region, but at small tilt variations around the β phase <110> drawing axis were combined (after correction for tilt distortion) to enhance the atomic number contrast between the α-Ti precipitates and the surrounding β phase.[19]

(iii) Critical current measurements

Magnetization techniques were used to measure the critical current density of the samples. A vibrating sample magnetometer (VSM) was used to measure the magnetization of the samples and the critical current density was calculated using the critical state model.[20] For the case of a cylinder in transverse field (using SI units) the critical state model gives

$$J_c = \frac{3\pi}{4} \frac{\Delta M}{d} \quad \textbf{(6)}$$

where d is the diameter of the Nb-Ti filaments and ΔM is the width of the hysteresis loop at a given magnetic field. The pinning force for each measurement was then determined from

$$F_p = J_c \times B \quad \textbf{(7)}$$

The close spacing of the data points, thus obtained, allowed a detailed description of the pinning force curve. The flux pinning curves each contained a minimum of 50 data points and more typically several hundred data points. For this study magnetization loops were run for each sample at $\mu_o H_{c2}$ = 1T, 2.5T, 5T, 7.5T, and 10T. This was accomplished by first testing each sample from 2 K to T_c in 1 K intervals in order to obtain the H_{c2} versus T curve, and then interpolating the values on the curve to obtain the temperature at which H_{c2} equalled 1T, 2.5T, 5T, 7.5T, and 10T.

The VSM was installed in a dewar with a variable temperature insert (VTI) which allowed the temperature of the sample to be varied readily. The temperature of the sample was controlled using germanium and capacitance thermometers which were located just below the vibrating sample in the flow of heated helium gas. The calibration of the system was checked using Nb, Ta, Pb, and V as T_c standards. Using the correction obtained with these standards, the error in the temperature was always less than 0.7%. The upper critical field of each sample was determined from the point at which the hysteresis loop closed or the width of the loop became smaller that the background noise. In a few isolated cases, for instance following heat treatment when the copper is annealed and its resistivity becomes low and near T_c where the signal from the superconductor was small, the eddy currents flowing in the copper formed a loop of constant width beyond H_{c2}. In these cases H_{c2} was determined by drawing a line through the linear eddy current contribution (needs

better explanation). The place where the hysteresis loop deviated from this line was called H_{c2}. The agreement between the up and down ramp using this method was never greater than ± 0.05 T. The determination of H_{c2} with this method is not as well defined as taking the 50% value of a resistivity trace. Rather, some amount of judgement is required to determine where the loop closure occurred. For temperatures where H_{c2} > 1T, an upper limit on the error in determining the value of field where the loop was judged to close was ± 0.06T. For higher temperatures and smaller H_{c2} values, the ramp rate was slowed and the error in determining the loop closure restricted to ± 0.03T.

3. Microstructural Results

The microstructural results in this experiment were much more uniform than any previously observed on these alloys.[8,21,22] However, the microstructures obtained were not identical, across the alloy range from Nb44wt%Ti to Nb58wt%Ti. As discussed previously the total available strain space was limited and uniform precipitation was not expected to occur in Nb62wt%Ti.

The precipitate morphology of UW1719 (Nb44wt%Ti) differs considerably from the rest of the wires in this study. The α-Ti precipitate for UW1719 occurred primarily in the form of a thick (5 nm) grain boundary film. The precipitate morphology for Nb46.5wt%Ti to Nb58wt%Ti wires was primarily Grain boundary triple point with some grain boundary film present, as shown in Figures 2-6. The results of the image analysis for this experiment are listed in Table 3. As shown in Table 3, although the precipitate morphology for Nb46.5wt%Ti to Nb58wt%Ti was similar, the percentage of α-Ti precipitate occurring in these alloys ranged from 7.7% to 15%. We think this range, however, is acceptable for this experiment.

The minimum amount of precipitate for this investigation occurred in Nb44wt%Ti. The maximum amount of precipitate for this investigation occurred in Nb62wt%Ti (Figure 7). As described above the cold work provided in this investigation was not expected to be enough to suppress intragranular precipitation. Although there was no evidence of intragranular precipitation

for UW1819 (Nb62wt%Ti), the precipitation observed was very unusual. The precipitates are large and equiaxed with irregular interfaces with the β-Nb-Ti grains. This microstructural morphology resulted in wire so brittle that it could not even be pulled back through the same die used prior to heat treatment.

Figures 8 and 9 show the effect of giving UW2119 (Nb46.5wt%Ti) and UW2219 (Nb49wt%Ti) a strain of 2 following heat treatment. Following the cold work, the α-Ti precipitates were already very fine (most of the α-Ti was ≈ 2 nm thick, with occasional thicknesses as large as 10 nm) but are not the classic ribbon morphology normally seen in Nb-Ti alloys.[6,23] A total strain of 4 for UW2119 (Nb46.5wt%Ti) reduced all the precipitate to less than 1 nm thick. An interesting feature of these microstructures is the lack of precipitate folding compared to conventionally processed Nb46.5wt%Ti alloys where the large quantity and size of precipitates results in considerable folding transverse to the drawing axis.

4. Flux Pinning Results

The Pinning force results are presented in the form of reduced pinning force curves at the final drawing strains of $\epsilon_f = 0, 2.05, 4.14$. Several major trends can be seen in the reduced pinning force results (Figures 11-17). The first trend was that with increasing cold work following the heat treatment, the peak in the curve shifted to higher reduced fields. This shift was also observed by Küpfer and Matsushita and by Meingast and Larbalestier.[1,2] The second major trend was that as the temperature was increased, the peak in the curve shifted toward lower field. This effect was small for Nb44wt%Ti but very pronounced for Nb62wt%Ti. The final trend is a measurement artifact. As the wire size decreased, the noise level increased. This was due not only to the decreasing volume of sample present but also to the reduction in the filament diameter since the magnetic moment goes as r^3.

4.1 UW1719 - Nb44wt%Ti

Figure 11 shows the reduced pinning force curves for the Nb44wt%Ti alloy. The most noticeable thing about these curves was the lack of scaling for the after heat treatment wire (0.050") and the wire which received a final strain of 2 (0.0179"). The smallest wire ($\epsilon_f = 4$, d = 0.0063") was the closest to exhibiting scaling behavior of any of the wires in this study. The only curve for the 0.0063 in wire which deviated significantly from the others was the high temperature curve. The testing temperature, upper critical field and maximum pinning force for each curve in Figure 11 are listed in Table 4.

4.2 UW2119 - Nb46.5wt%Ti

Figure 12 shows the reduced pinning force curves for Nb46.5wt%Ti. With the small increase in Ti content, the pinning force curves following heat treatment of Nb46.5wt%Ti were already shifted toward lower reduced field relative to those of Nb44wt%Ti following heat treatment. Cold working the wires following heat treatment caused the pinning force curves to gradually move toward higher reduced field. This can be seen readily if the figures are overlaid. Similar to the behavior of the other wires in this experiment, the curves shifted toward lower reduced fields with increasing temperature. This can easily be seen in Figure 12. The shift toward lower field with increasing temperature was similar to the observations of Meingast and Larbalestier.[2] However, the shift was more pronounced for their Nb48wt%Ti alloy than for this Nb46.5wt%Ti alloy. The measurement temperatures, upper critical field and maximum pinning force for Nb46.5wt%Ti are listed in Table 5.

4.3 UW2219 - Nb49wt%Ti

The high temperature reduced pinning force curve for Nb49wt%Ti following the heat treatment had its peak at extremely low reduced field ($b_p \approx .12$) (see Figure 13). Following a strain

of 4, the high temperature peak shifted up considerably in reduced field ($b_p \approx .26$). The cold work following heat treatment also affected the low temperature measurements taking them from non-scaling following heat treatment to nearly indistinguishable curve shapes at a strain of 4. This scaling was limited to low temperatures and breaks down as the temperature is increased. The measurement temperatures, upper critical field and maximum pinning force for Nb46.5wt%Ti are listed in Table 6.

4.4 UW3419 - Nb52wt%Ti

The reduced pinning force curves for UW3419 are shown in Figure 14. The trends typical of the rest of the alloys can be seen in these curves. Following the heat treatment the reduced pinning force curves were shifted toward lower field. Cold work caused the curves to shift toward higher field. None of the wires showed any indication of scaling behavior, the trend was a shift toward lower field with increasing temperature. The measurement temperatures, upper critical field and maximum pinning force for Nb52wt%Ti are listed in Table 7.

4.5 UW2319 - Nb53wt%Ti

The flux pinning behavior of the Nb53wt%Ti alloy (Figure 15) was very similar to that observed for the Nb52wt%Ti alloy. Following the heat treatment the F_p curves for Nb53wt%Ti were shifted to slightly lower field than those of the Nb52wt%Ti alloy (the actual composition difference, listed in Table 1, is 0.1%). However, following cold work the F_p curves of the Nb53wt%Ti alloy are higher in reduced field than those of the Nb52wt%Ti alloy. The measurement temperatures, upper critical field and maximum pinning force for Nb53wt%Ti are listed in Table 8.

4.6 UW2419 - Nb58wt%Ti

Figure 16 shows the reduced pinning force curves for the Nb58wt%Ti alloy. Although the

general trend of a shift in b_p toward higher field with increasing cold work was observed in this alloy, it was not as pronounced as in the rest of the alloys. The high temperature curve ($H_{c2}(T) \approx 1T$) remained at low reduced field throughout all the wire drawing. The measurement temperatures, upper critical field and maximum pinning force for Nb58wt%Ti are listed in Table 9.

4.7 UW1819 - Nb62wt%Ti

As discussed above, this wire was too brittle to draw following heat treatment. Thus reduced pinning force data was available only following heat treatment (see Figure 17). For Nb62wt%Ti both the curves for $H_{c2} \approx 1T$ and $H_{c2} \approx 2.5T$ peak at low field. In the lower Ti content alloys only the $H_{c2} \approx 1T$ curve was shifted to very low reduced fields. Reducing the temperatures caused the curves to shift to higher field but the peak in the $H_{c2} \approx 10T$ curve for Nb62wt%Ti was substantially lower in reduced field ($b_p = .35$) than Nb58wt%Ti ($b_p = .46$). The measurement temperatures, upper critical field and maximum pinning force for Nb62wt%Ti are listed in Table 10.

5. Discussion

The purpose of this experiment was to answer the question as to whether the observed shift in the flux pinning behavior with increasing Ti content was due to the compositional change as hypothesized by Meingast and Larbalestier[2] or was due to the observed increase in the volume fraction of α-Ti as the Ti content was increased.[8] In order to answer this question it was necessary to produce composite wires with similar microstructures. The microstructural results of this experiment showed that most of the samples produced had similar precipitation morphology with the α-Ti precipitate occurring in the grain boundary triple points and some grain boundary film. As described above, the Nb44wt%Ti and the Nb62wt%Ti had different precipitate morphologies. The rest of the wires had fairly constant quantities of precipitate with an average amount of ≈ 11%. Although there is still some variation in the microstructures, they are much more similar than any previously produced across this range of Nb-Ti alloys. These microstructures are

sufficiently similar to address Meingast and Larbalestier's hypothesis.

There are two main differences between the samples used by Meingast et al. and the samples from this study.[2,6] The first difference in the two investigations is that a much wider range of Ti compositions was used in this study. The second difference is the microstructures observed in the two investigations. Following the last precipitation heat treatment, Meingast et al. found 19% of α-Ti precipitate in their samples with an average precipitate area of 49300 $(nm)^2$. In this study the wires from Nb46.5wt%Ti to Nb58wt%Ti had an average of ~ 11% α-Ti and an average precipitate area of 462 $(nm)^2$. The consequence of the fine microstructure in this study is that following heat treatment the density of α-Ti precipitates (or pinning centers) is ≈ 2 x 10^{14} ppts/m^2 (for UW2119, the other wires are of comparable size) while for Meingast and Larbalestier's wire the density of α-Ti precipitates was ≈ 3 x 10^{12} ppts/m^2. Due to the large difference in the density of pins following the heat treatment and the difference in the volume fraction of α-Ti between the two studies, the pinning force curves for this study are shifted to higher reduced field than those of Meingast and Larbalestier.

The effect of differences in the percentage of α-Ti precipitate can be seen best by examining Figures 14 and 15. As noted above the reduced pinning force curves of the Nb53wt%Ti alloy following a heat treatment and further cold work were shifted to higher reduced field than those of the Nb52wt%Ti alloy. The main difference between these two composites is the volume fraction of α-Ti precipitate, measured at 15vol% for the nominal 52wt%Ti alloy and 8.9vol% for the nominal 53wt%Ti alloy, whereas the measured alloy composition (see Table 1) differed by only 0.1wt%Ti. Thus increased percentages of α-Ti precipitate tend to shift the pinning force toward lower reduced fields.

Considering the large size difference and percentage of precipitate of the as heat treated microstructure of the wires from Meingast and Larbalestier's study and the as heat treated wires from this study, it is not surprising that the microstructures obtained with subsequent cold work

were also different. In Meingast and Larbalestier's wires, cold work following heat treatment caused the formation of α-Ti ribbon clusters. The ribbon cluster morphology is commonly observed in high J_c Nb-Ti wires[23,24] and results from the folding of the α-Ti precipitates and the β matrix. The reduced quantity and size of precipitate in this experiment has greatly reduced the amount of folding. Although the precipitates deform into ribbons they are relatively short and isolated. The lower temperature of heat treatment used in this experiment also increase the portion of precipitate that occurs as thin grain boundary film.[25] With the presence of the dense ribbon morphology Meingast and Larbalestier observed a gradual shift toward lower reduced field with increasing temperature. In this study, however, the finest Nb46.5wt%Ti wire (UW2119 0.0063") does not show a large shift toward lower field with increasing temperature (Figure 12). This suggests that the shift toward lower field observed by Meingast and Larbalestier was in part due to the ribbon clusters which would act as local regions of higher average Ti content.

The dependence of the flux pinning on the alloy content can be seen best by comparing Figures 12 (Nb46.5wt%Ti) and 16 (Nb58wt%Ti). The 1T H_{c2} curves for the Nb46.5wt%Ti wires show increasing b_p with increasing ϵ_f. However, for Nb58wt%Ti the 1T H_{c2} curves remain shifted to low field through the entire cold working process. Since the curves remain at low field, ΔH_c pinning must be dominating the high temperature pinning behavior for Nb58wt%Ti.

Meingast and Larbalestier's prediction of a shift to low field in the peak of the pinning force curve with increasing Ti content due to an increase in the ΔH_c contribution to the pinning force was based on plotting the normalized values of κ and H_c as a function of composition. The tendency for ΔH_c pinning to dominate with increasing Ti content and temperature can be viewed by making a plot similar to Meingast and Larbalestier's plot for the alloys of this study. Figure 18 shows normalized ΔH_c and $\Delta\kappa$ values for the alloys of this study.

The curves in Figure 18 were calculated as follows. dB_{c2}/dT was determined by doing second order least squares fits to the B_{c2} vs T_c data. These slopes were then used in the expression

$$-\frac{dH_{c2}}{dT} = 4.48 \times 10^4 \gamma \rho \ (Oe/K) \qquad \textbf{(8)}$$

where γ is the electronic specific heat in (erg)/(cm^3 K^2) and ρ is the resistivity in Ωcm.[26] The resistivity was then determined using second order least squares fits to Muller's specific heat data.[27] The resulting resistivity values were then used in

$$\kappa = 7.49 \times 10^3 \gamma^{1/2} \rho \qquad \textbf{(9)}$$

(where γ is the electronic specific heat in (erg) / (cm^3 K^2) and ρ is the resistivity in Ωcm[26])
with the fit to Muller's specific heat data to obtain κ_{GL}. The temperature dependence of κ was then taken into account by assuming a straight line between the limiting cases in Maki's calculations which gives[28]

$$\kappa_1 = \kappa_{GL}\left[1.2 - .2\left(\frac{T}{T_c}\right)\right]. \qquad \textbf{(10)}$$

The resulting temperature dependent κ values were then used, together with the second order fits to B_{c2} versus T, to determine the temperature dependent H_c values. The ratios of the κ_1 values and H_c values are shown in Figure 18. There is negligible composition dependence for κ at 3 K or 8 K and for H_c at 3 K. The H_c ratio at 8 K (much closer to T_c) falls sharply for Ti contents above 49wt%Ti.

Overall, the reduced flux pinning curves from this study showed three major trends. First, as the temperature was increased, b_p became smaller. Second, cold working the wire following heat treatment caused b_p to become larger. Finally, increasing the Ti content caused b_p to become

smaller. The dependence of b_p on the overall Ti content of the alloy supports the hypothesis of Meingast and Larbalestier.

6. Conclusions

Similar microstructures were produced in a range of Nb-Ti alloys having compositions from Nb46.5wt%Ti to Nb58wt%Ti. The microstructure obtained in these samples was controlled by using a high degree of initial cold work to control the precipitate morphology and varying precipitation heat treatment temperatures to control the percentage of precipitate. The pinning behavior of the samples with similar microstructures showed a distinct dependance on the overall Ti content of the alloy. As the Ti content was increased, the peak in the reduced pinning force curve shifted toward lower reduced field. Thus ΔH_c is the dominant pinning mechanism in the high Ti alloys which confirms Meingast and Larbalestier's hypothesis.[2]

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Table 1: The Chemistry, Grade, Density, and Billet identification for the Alloys used in this study. Compositions are in wt%Ti, densities (ρ) are in g/cm^3, the recrystallization anneal size is in inches, and impurities are in ppm.

Nominal Composition	Nb44Ti	Nb46.5Ti	Nb49Ti	Nb52Ti	Nb53Ti	Nb58Ti	Nb62Ti
Wet Chemistry	43.5	46.5	49.3	52.4	52.5	57.8	62.3
Last Full Anneal	1.625"	1.5"	1.5"	1.620"	1.5"	1.5"	1.5"
ρ (g/cc)	6.06	6.02	5.96	5.77	5.85	5.67	5.52
Grade*	HiHo	Std	HiHo	HiHo	Std	HiHo	HiHo

Al	< 25	< 25	< 25	< 25	< 25	< 25	30
C	40	30	40	34	40	50	60
Cr	< 50	25	< 50	< 50	< 50	< 50	< 50
Cu	12	< 10	11	< 10	11	45	12
Fe	< 50	58	< 50	< 50	< 50	74	92
H	16	32	20	< 5	28	21	48
N	23	42	8	30	16	20	8
Ni	< 25	< 25	< 25	< 25	< 25	< 25	< 25
O	690	660	590	608	630	680	620
Si	< 100	< 100	< 100	< 100	< 100	< 100	< 100
Sn	< 40	< 40	< 40	< 40	< 40	< 40	< 40
Ta	1310	320	680	982	550	540	520
Sample ID	UW1719	UW2119	UW2219	UW3419	UW2319	UW2419	UW1819

*HiHo grade Nb-Ti alloy is certified free of Ti rich "freckles" whereas standard grade is not.

Table 2: Heat treatment temperatures based on keeping the diffusion rate of Nb constant in Nb-Ti for the nominal Nb-Ti compositions shown. The baseline for this calculation was Nb46.5wt%Ti at 375°C.

wt% Ti	Sample ID	Nb in Nb-Ti[19]
44	UW1719	390°C
46.5	UW2119	375°C
49	UW2219	360°C
52	UW3419	342°C
53	UW2319	337°C
58	UW2419	309°C
62	UW1819	288°C

Table 3: Percentage of α-Ti precipitate present following the last heat treatment for the isomorphology experiment.

Wire ID	Nominal Alloy wt% Ti	Measured Alloy Composition wt%	Heat Treatment	%α-Ti	Average Area $(nm)^2$	Calculated Residual β-Nb-Ti Composition after Heat Treatment
UW1719	44	43.5	40/390	3.6	219	42.1
UW2119	46.5	46.5	40/375	7.7	408	43.5
UW2219	49	49.3	40/360	11	721	45.1
UW3419	52	52.4	40/342	15	255	46.9
UW2319	53	52.5	40/337	8.9	439	49.4
UW2419	58	57.8	40/309	12	488	54.0
UW1819	62	62.3	40/288	6	1775	57.6

Table 4: The measuring temperature, upper critical field and maximum pinning force for the Nb44wt%Ti wires (UW1719).

Size (in)	Temp	H_{c2}(T)	F_{pmax} (N/m^3)
0.0063	4.75 K	10.15	5.10×10^9
0.0063	6.11 K	7.40	2.48×10^9
0.0063	7.35 K	4.90	9.88×10^8
0.0063	8.34 K	2.52	2.48×10^8
0.0063	8.90 K	1.07	4.70×10^7
0.0179	4.78 K	10.10	5.19×10^9
0.0179	6.10 K	7.43	2.66×10^9
0.0179	7.27 K	5.16	1.17×10^9
0.0179	8.36 K	2.48	3.08×10^8
0.0179	8.93 K	2.04	6.69×10^7
0.050 AHT	4.30 K	10.39	4.72×10^9
0.050 AHT	5.88 K	7.42	2.53×10^9
0.050 AHT	7.33 K	4.79	1.07×10^9
0.050 AHT	8.37 K	2.32	3.24×10^8
0.050 AHT	8.94 K	1.00	7.19×10^7

Table 5: The measuring temperature, upper critical field and maximum pinning force for the Nb46.5wt%Ti wires (UW2119).

Size (in)	Temp	H_{c2}(T)	F_{pmax} (N/m^3)
0.0063	4.66 K	10.09	5.06×10^9
0.0063	6.03 K	7.50	2.48×10^9
0.0063	7.21 K	4.98	1.00×10^8
0.0063	8.20 K	2.51	2.39×10^8
0.0063	8.75 K	1.01	4.38×10^7
0.0179	4.86 K	9.70	4.76×10^9
0.0179	6.10 K	7.25	2.51×10^9
0.0179	7.23 K	4.94	1.14×10^9
0.0179	8.23 K	2.58	3.34×10^8
0.0179	8.77 K	1.00	6.29×10^7
0.050 AHT	4.41 K	10.14	4.76×10^9
0.050 AHT	5.76 K	7.44	2.45×10^9
0.050 AHT	6.96 K	5.31	1.24×10^9
0.050 AHT	8.20 K	2.60	3.39×10^8
0.050 AHT	8.80 K	0.97	5.98×10^7

Table 6: The measuring temperature, upper critical field and maximum pinning force for the Nb49wt%Ti wires (UW2219).

Size (in)	Temp	H_{c2}(T)	F_{pmax} (N/m^3)

0.0063	4.37 K	10.15	5.69×10^9
0.0063	5.70 K	7.58	2.88×10^9
0.0063	6.93 K	4.71	1.19×10^8
0.0063	7.95 K	2.32	2.72×10^8
0.0063	8.56 K	1.13	3.35×10^7
0.0179	4.34 K	10.24	6.00×10^9
0.0179	5.68 K	7.46	3.10×10^9
0.0179	6.95 K	5.07	1.43×10^9
0.0179	8.02 K	2.46	3.90×10^8
0.0179	8.66 K	0.98	4.28×10^7
0.050 AHT	4.27 K	10.14	4.92×10^9
0.050 AHT	5.73 K	7.49	2.65×10^9
0.050 AHT	7.05 K	4.84	1.17×10^9
0.050 AHT	8.18 K	2.33	2.75×10^8
0.050 AHT	8.78 K	1.04	1.54×10^7

Table 7: The measuring temperature, upper critical field and maximum pinning force for the Nb52wt%Ti wires (UW3419).

Size (in)	Temp	H_{c2}(T)	F_{pmax} (N/m^3)
0.0063	4.06 K	10.25	5.26×10^9
0.0063	5.39 K	7.33	2.84×10^9
0.0063	6.55 K	4.83	1.07×10^8
0.0063	7.58 K	2.41	2.37×10^8
0.0063	8.13 K	0.92	2.68×10^7
0.0179	4.12 K	10.11	6.79×10^9
0.0179	5.50 K	7.66	3.56×10^9
0.0179	6.77 K	5.00	1.40×10^9
0.0179	7.73 K	2.44	3.26×10^8
0.0179	8.36 K	1.00	1.71×10^7
0.050 AHT	3.99 K	10.18	6.37×10^9
0.050 AHT	5.44 K	7.82	3.33×10^9
0.050 AHT	6.67 K	4.79	1.34×10^9
0.050 AHT	7.69 K	2.41	3.42×10^8
0.050 AHT	8.38 K	0.80	1.40×10^7

Table 8: The measuring temperature, upper critical field and maximum pinning force for the Nb53wt%Ti wires (UW2319).

Size (in)	Temp	H_{c2}(T)	F_{pmax} (N/m^3)
0.0063	4.15 K	10.01	4.49×10^9
0.0063	5.47 K	7.53	2.39×10^9
0.0063	6.71 K	5.07	8.75×10^8
0.0063	7.60 K	2.65	2.50×10^8
0.0063	8.13 K	1.00	4.49×10^7

0.0179	4.16 K	9.89	6.20×10^9
0.0179	5.40 K	7.53	3.50×10^9
0.0179	6.68 K	5.01	1.43×10^9
0.0179	7.76 K	2.51	3.24×10^8
0.0179	8.43 K	1.00	1.51×10^7
0.050 AHT	3.75 K	10.27	5.92×10^9
0.050 AHT	5.26 K	7.77	3.22×10^9
0.050 AHT	6.66 K	4.70	1.20×10^9
0.050 AHT	7.74 K	2.42	2.98×10^8
0.050 AHT	8.39 K	1.18	1.26×10^7

Table 9: The measuring temperature, upper critical field and maximum pinning force for the Nb58wt%Ti wires (UW2419).

Size (in)	Temp	H_{c2}(T)	F_{pmax} (N/m^3)
0.0063	2.91 K	10.21	4.13×10^9
0.0063	4.46 K	7.66	2.29×10^9
0.0063	5.73 K	5.19	9.32×10^8
0.0063	6.84 K	2.53	1.93×10^8
0.0063	7.47 K	1.21	1.40×10^7
0.0179	3.18 K	10.02	5.84×10^9
0.0179	4.69 K	7.26	3.06×10^9
0.0179	5.89 K	5.16	1.16×10^9
0.0179	7.09 K	2.24	1.75×10^8
0.0179	7.58 K	0.98	1.51×10^7
0.050 AHT	3.46 K	10.05	8.12×10^9
0.050 AHT	5.01 K	7.84	3.68×10^9
0.050 AHT	6.31 K	4.86	1.09×10^9
0.050 AHT	7.33 K	2.43	1.52×10^8
0.050 AHT	7.88 K	1.19	4.25×10^6

Table 10: The measuring temperature, upper critical field and maximum pinning force for the Nb62wt%Ti wires (UW1819).

Size (in)	Temp.	H_{c2}(T)	F_{pmax} (N/m^3)
0.050 AHT	3.18 K	9.96	6.47×10^9
0.050 AHT	4.70 K	7.72	3.57×10^9
0.050 AHT	5.99 K	5.07	1.40×10^9
0.050 AHT	7.13 K	2.61	2.31×10^8
0.050 AHT	7.75 K	1.07	8.78×10^6

Figure 1: TEM micrograph of a Nb44wt%Ti (UW1719 0.050") transverse cross section (approx 5° from the β_{110} drawing axis) after one heat treatment of 40h at 390°C, $\epsilon_p = 11.8$.

Figure 2: TEM micrograph of a Nb46.5wt%Ti (UW2119 0.050") transverse cross section (approx 5° from the β_{110} drawing axis) after one heat treatment of 40h at 375°C, $\epsilon_p = 11.8$.Figure 3: TEM micrograph of a Nb49wt%Ti (UW2219 0.050") transverse cross section (approx 5° from the β_{110} drawing axis) after one heat treatment of 40h at 360°C, $\epsilon_p = 11.8$.

Figure 4: TEM micrograph of a Nb52wt%Ti (UW3419 0.050") transverse cross section (approx 5° from the β_{110} drawing axis) after one heat treatment of 40h at 342°C, $\epsilon_p = 11.8$.

Figure 5: TEM micrograph of a Nb53wt%Ti (UW2319 0.050") transverse cross section (approx 5° from the β_{110} drawing axis) after one heat treatment of 40h at 337°C, $\epsilon_p = 11.8$.

Figure 6: TEM micrograph of a Nb58wt%Ti (UW2419 0.050") transverse cross section (approx 5° from the β_{110} drawing axis) after one heat treatment of 40h at 309°C, $\epsilon_p = 11.8$.

Figure 7: TEM micrograph of a Nb62wt%Ti (UW1819 0.050") transverse cross section (approx 5° from the β_{110} drawing axis) after one heat treatment of 40h at 288°C, $\epsilon_p = 11.8$.

Figure 8: TEM micrograph of a Nb46.5wt%Ti (UW2119 0.050") transverse cross section (approx 5° from the β_{110} drawing axis) after one heat treatment of 40h at 375°C, $\epsilon_p = 11.8$, $\epsilon_f = 2.05$.

Figure 9: TEM micrograph of a Nb49wt%Ti (UW2219 0.050") transverse cross section (approx 5° from the β_{110} drawing axis) after one heat treatment of 40h at 360°C, $\epsilon_p = 11.8$, $\epsilon_f = 2.05$.

Figure 10: TEM micrograph of a Nb46.5wt%Ti (UW2119 0.050") transverse cross section (approx 5° from the β_{110} drawing axis) after one heat treatment of 40h at 375°C, $\epsilon_p = 11.8$, $\epsilon_f = 2.05$.

Figure 11: Reduced pinning force versus reduced field for Nb44wt%Ti (UW1719) with one heat treatment: a) 0.050 in, $\epsilon_f = 0.0$; b) 0.0179 in, $\epsilon_f = 2.05$; $\epsilon_f = 4.14$.Figure 12: Reduced pinning force versus reduced field for Nb46.5wt%Ti (UW2119) with one heat treatment: a) 0.050 in, $\epsilon_f = 0.0$; b) 0.0179 in, $\epsilon_f = 2.05$; $\epsilon_f = 4.14$.

Figure 13: Reduced pinning force versus reduced field for Nb49wt%Ti (UW2219) with one heat treatment: a) 0.050 in, $\epsilon_f = 0.0$; b) 0.0179 in, $\epsilon_f = 2.05$; $\epsilon_f = 4.14$.

Figure 14: Reduced pinning force versus reduced field for Nb52wt%Ti (UW3419) with one heat treatment: a) 0.050 in, $\epsilon_f = 0.0$; b) 0.0179 in, $\epsilon_f = 2.05$; $\epsilon_f = 4.14$.

Figure 15: Reduced pinning force versus reduced field for Nb53wt%Ti (UW2319) with one heat treatment: a) 0.050 in, $\epsilon_f = 0.0$; b) 0.0179 in, $\epsilon_f = 2.05$; $\epsilon_f = 4.14$.

Figure 16: Reduced pinning force versus reduced field for Nb58wt%Ti (UW2419) with one heat treatment: a) 0.050 in, $\epsilon_f = 0.0$; b) 0.0179 in, $\epsilon_f = 2.05$; $\epsilon_f = 4.14$.

Figure 17: Reduced pinning force versus reduced field for UW1819 0.050" (Nb62wt%Ti) after heat treatment.

Figure 18: Values of κ and H_c calculated using κ_1 from [16] and normalized to Nb49wt%Ti.