PROGRESS IN THE UNDERSTANDING AND MANIPULATION OF MICROSTRUCTURE IN HIGH J_C NB-TI ALLOY COMPOSITES

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Abstract - The development of high J_C microstructures in Nb-46.5wt.%Ti under standard processing techniques is reviewed. Microstructures of alloys of higher Ti content given the same processing are shown to be significantly different. The effect of composition on precipitate morphology has recently been found to be quantifiable, predictable and controllable. The implications of these results in terms of low field applications is discussed.

Introduction

Nb-Ti alloys in the composition range of Nb-46.5wt.%Ti to 50wt.%Ti have become the dominant commercial superconductors. The development of high critical current density, J_c, microstructures in Nb-46.5wt.%Ti has only recently been fully characterized and quantified.[1,2,3] The driving force behind these extensive studies of the Nb-46.5wt.%Ti alloy has been the desire for increased J_c wire for high energy physics applications where the flexibility and high Jc of this alloy has made this the standard material for magnet designs in the 5T to 8T range. The excellent high field performance of Nb-46.5wt.%Ti is related to the high H_{C2} of this composition. The principal commercial application of superconducting wire, however, is MRI where the magnetic fields are relatively low (0.35-4T). There is also a very large possible future market for Nb-Ti for superconducting magnetic energy storage, SMES, where low to mid magnetic fields will probably be required (3-5T) with cooling by liquid He II (1.8 K). Under these conditions where high H_{C2} is less important, an early study by McInturff and Chase[4] indicated that superior J_Cs could be obtained using higher Ti content alloys. Since that early study, improved processing techniques have resulted in a two-fold increase in J_C for the Nb-46.5Ti alloy throughout the field range 2-8T.[5] Using these new techniques in a recent study, these high Ti content alloys have again been shown to exhibit superior low field current carrying capability.[6] The microstructures generated in these alloys, however, are inferior in homogeneity to those developed in Nb-46.5wt.%Ti and have increased filament hardness.[6,7] If these microstructural problems can be solved, then the low field J_C capabilities of Nb-Ti alloy should be further enhanced.

The paper reviews the essential elements of high J_C Nb-Ti microstructures and examines the latest attempts to control the microstructure of high Ti alloys.

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Nb-46.5wt.%Ti Final Wire Microstructures

The microstructural development of high J_C Nb-46.5wt.%Ti wires has been extensively studied by TEM.[1,2,3] The highly anisotropic nature of these heavily cold worked wires necessitates examination in both transverse and longitudinal cross-section. The results of these studies are summarized in the schematic diagram in Figure 1, which is a three dimensional representation of the α-Ti ribbon morphology in a high J_C wire. Quantification of the microstructure indicates that the mean ribbon thickness is approximately 1nm with an average of 4nm of β-Nb-Ti between each ribbon.[3,7] The ribbons rarely exceed 3nm in thickness in an optimized wire. The α-Ti precipitates extend for more than 2μm parallel to the drawing axis of the wire. The microstructures of such wires are very uniform across the wire cross-section. α-Ti precipitates make up 20-26% of the filament. Only the α-Ti and β-Nb-Ti phases are observed within the filaments. If diffusion barriers are not used the remnants of brittle reaction products between the Cu stabilizer and Nb-Ti filaments can also be found at the Cu-Nb-Ti interface.

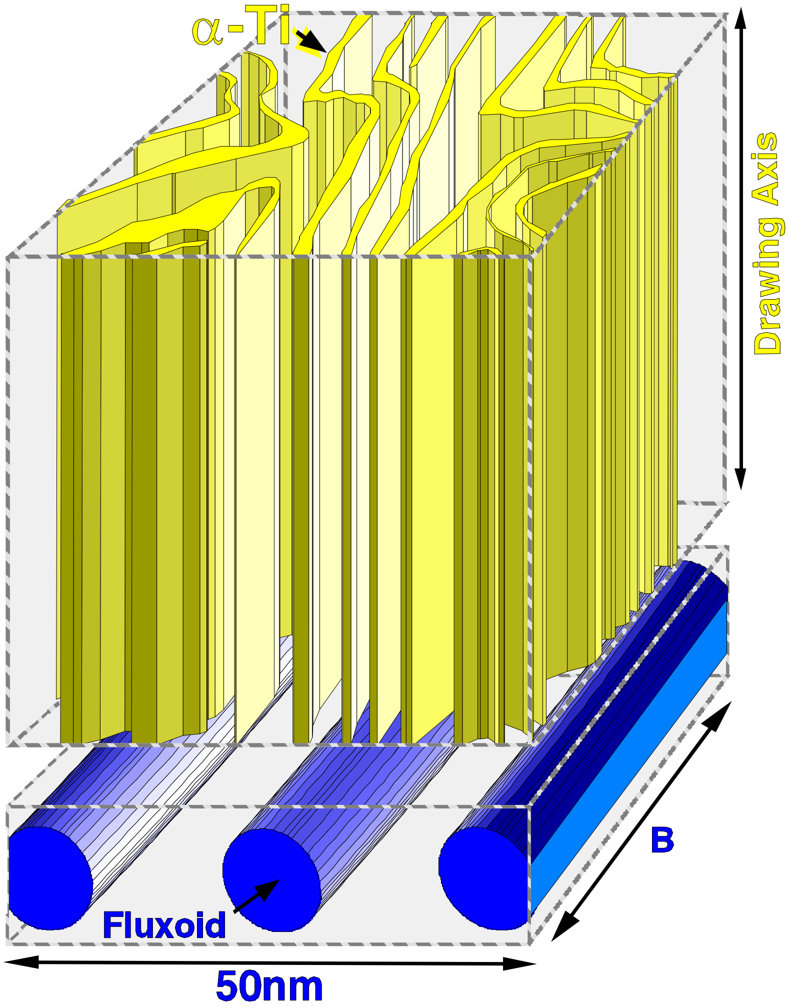

Figure 1 Schematic illustration of α-Ti ribbon morphology in a high J_c Nb-46.5wt.%Ti filament. The equilibrium fluxoid spacing at 5T, 4.2 K is compared below.

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Development of High J_c Microstructure in Nb-46.5wt.%Ti

In order to obtain the uniform microstructure found in the optimized wires the initial Nb-Ti rod must be chemically homogeneous. A schematic illustration of the processing sequence is shown in Figure 2. The initial precipitation is given when the Nb-Ti has received a cold work drawing strain of at least 5.[9] The microstructure after this heat treatment is illustrated in Figure 2b, and consists of α-Ti precipitates at β-Nb-Ti grain boundary triple points and a thin (less than 4nm) grain boundary film of α-Ti. The heat treatment is typically for 10-80 hours in duration and at between 370°C and 420°C. The effect of temperature and duration of heat treatment is small at this stage. Lower temperatures favor the film precipitation and at 300°C only grain-boundary film is produced.[8] The distribution and quantity of triple-point α-Ti precipitates is controlled by the initial β-Nb-Ti grain size. The precipitate size after initial heat treatment can vary significantly after initial heat treatment, depending on grain size but the total volume of

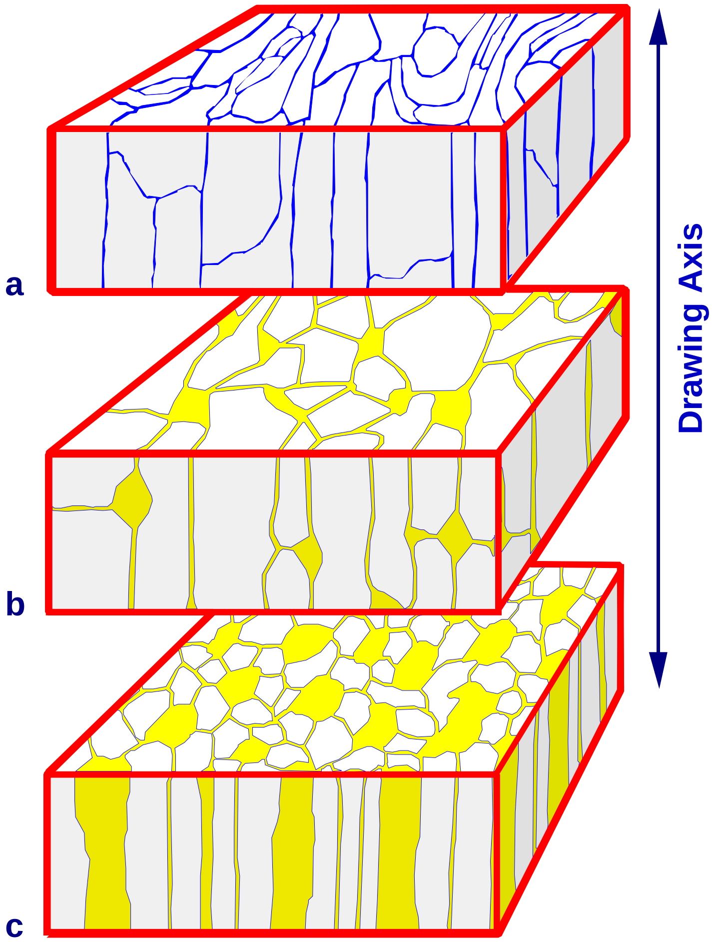

Figure 2. A schematic illustration of the development of microstructure in a conventionally processed Nb-46.5wt.%Ti high J_C superconductor, a) β-Nb-Ti before initial heat treatment, b) precipitation of α-Ti (shaded areas) at grain boundaries and grain boundary triple points produced by initial heat treatment, and c) increase in volume of precipitate and refinement of microstructure by subsequent drawing and heat treatment cycles prior to final drawing strain.

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precipitate does not exceed 11% of the filament. Further heat treatments are required to produce the 20-26 volume % of precipitate found in high J_C material.[3,8] Additional heat treatments are applied after additional cold work, typically a cold work strain of 1.15. In general the longer, the more frequent, and the higher the temperature of heat treatments, the higher is the ultimate J_C of the final wire.[10] These conditions correspond to an increased volume of precipitate and increased transverse cross-sectional area.[8] The microstructure after final heat treatment is illustrated in Figure 2c. The additional heat treatments have resulted in a uniform distribution of α-Ti precipitates which are similar in dimension to the β-Nb-Ti grain size. In order to obtain the high J_c microstructure illustrated in Figure 1 from the final heat treatment microstructure in Figure 2c an additional large cold work strain is required. The folded ribbon microstructure is a result of the incompatible deformation characteristics of the hexagonal α-Ti and the BCC β-Nb-Ti. The folding of the ribbons results in a large increase in the density of interfacial surface. Final drawing strains required to produce optimized wire range from 4-6 (98.2-99.8% area reduction), the larger the size of precipitate at final heat treatment, the larger is the strain required for optimization. Despite this considerable cold work strain, both phases exhibit remarkable ductility and we have not so far observed any evidence of precipitate fracture during normal drawing.

High Titanium Alloys

Applying the heat treatments described above to higher titanium content alloys results in precipitation that is neither homogeneous nor conducive to good ductility.[6] For the high Ti alloys, triple point α-Ti competes with string of pearls α-Ti along grain boundaries, Widmanstätten α-Ti needles in the β-Nb-Ti grains and the metastable ω-phase. All these forms of precipitation can be found in the electron micrograph shown in Figure 3, which is a transverse cross-section of a Nb-53wt.%Ti alloy after initial heat treatment (at a prestrain of 5) of 40 hours at 420°C. The ω and Widmanstätten α-Ti considerably increase the hardness of the Nb-Ti and reduce the drawability of the composite.[6] The precipitation of α-Ti in the string of pearls morphology results in a very uneven distribution of precipitation. Increasing the temperature of heat treatment suppresses ω-precipitation. The higher the Ti content the greater the density of Widmanstätten α-Ti precipitation.[7] Despite these drawbacks good critical current densities can be obtained from higher Ti content alloys provided that they can be drawn. Additional heat treatments result in a ripening process that favors the larger triple-point α-Ti precipitates eliminating most of the needle-like or Ti and blunting those that remain resulting in reduced hardness. By using three heat treatments (80hr/420°C, prestrain of 5) on a Nb-58wt.% alloy, a J_C in excess of 7400 A/mm^2 at 2T has been obtained which is well in excess of that obtainable with Nb-46.5wt.%Ti.[6] Unlike high J_C Nb-46.5wt.%Ti wire which has a strong optimization peak in the J_C versus final drawing strain curve, the high Ti alloys often exhibit a behavior almost independent of strain. This result would be expected of a microstructure that contains a large variation in precipitate size. It suggests that the performance of these alloys can be further enhanced by creating a more uniform microstructure.

If Nb-46.5wt.%Ti is given its first heat treatment at a prestrain of 2 rather than 5, both acicular α-Ti and ω will also be formed.[9] Thus increasing pre-strain favors triple-point α-Ti precipitation. By applying this observation to high Ti content alloys, triple-point α-Ti only microstructures have recently been obtained in alloys of up to 58wt.%Ti.[11] The relationship

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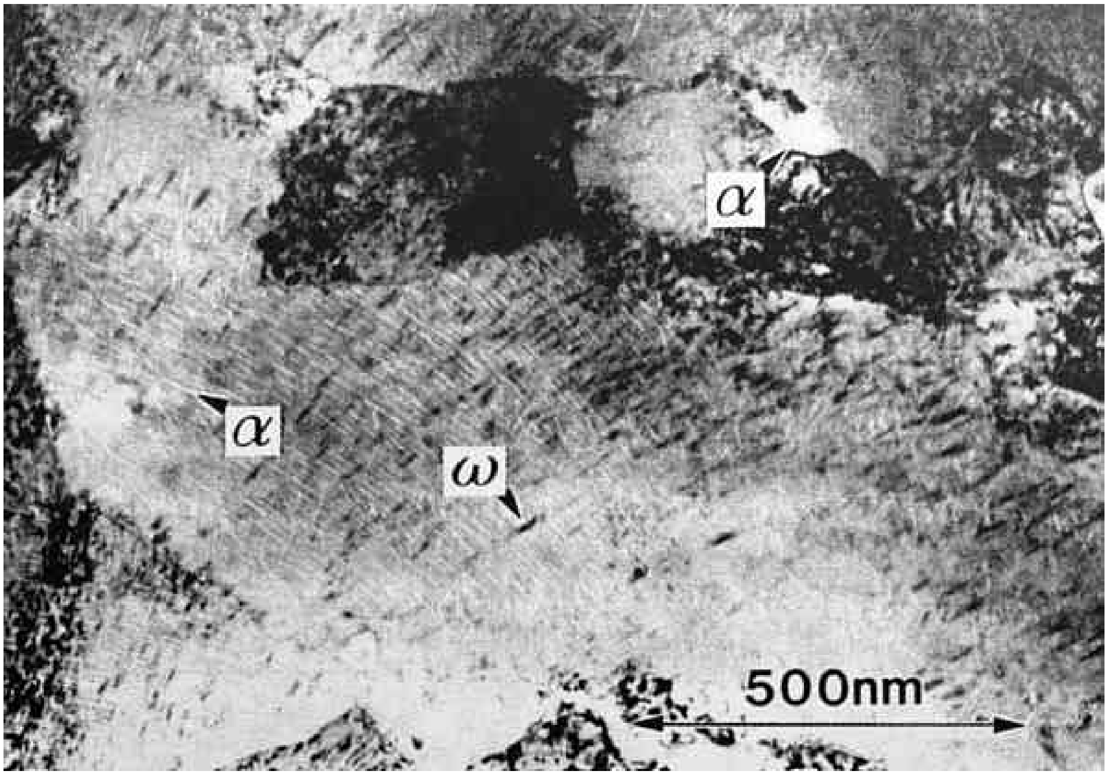

Figure 3. TEM micrograph of transverse cross-section of Nb53wt.%Ti filament after l HT (40hrs/420°C) at a prestrain of 5.

between pre-strain and precipitate morphology is illustrated in Figure 4. With the microstructure now under greater control, the increased precipitation rate found in higher Ti content alloys can be quantified. The results of such a quantification[11] are illustrated in Figure 5 in which the volume of precipitate produced by one and two heat treatments are compared for three alloys, Nb-46.5wt.%Ti, Nb-49wt.%Ti, and Nb-53wt.%Ti. By increasing the Ti content by 6.5wt.% the volume of precipitate was increased by more than 50%.[11] Furthermore, the volume of precipitate produced in only two heat treatments in the 53wt.% alloy (26 volume %) would require six similar heat treatments in Nb-46.5wt.%Ti.[8]

The control now obtainable over the microstructure of high Ti content Nb-Ti alloys promises further advances in the low field performance of binary Nb-Ti alloys. Such control requires an increased prestrain that reduces the available strain space for heat treatment, the increased precipitation rate, however, results in a reduction in the number of heat treatments required.

Acknowledgments

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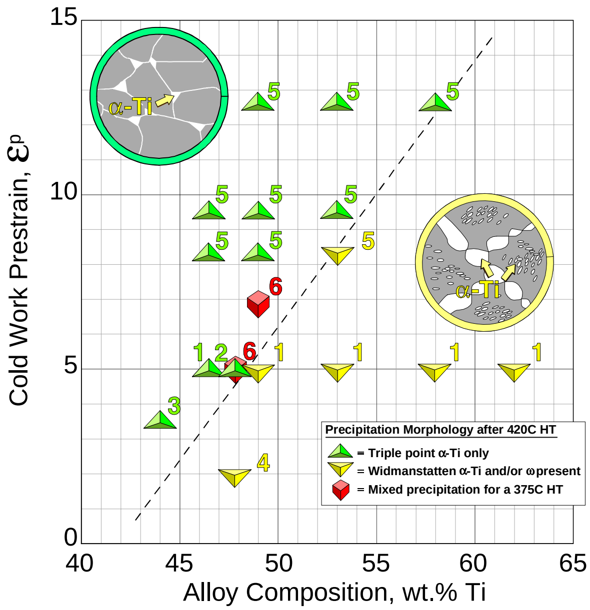

Fig. 4. Plot of prestrain versus composition showing the precipitate morphology after initial heat treat-ment. 1= ref. 7, 2 = ref. 3, 3 = ref. 12, 4 = ref. 9, 5 a ref. 11, and 6 = ref. 12. Red "mixed precipitation" points added for this electronic version only.

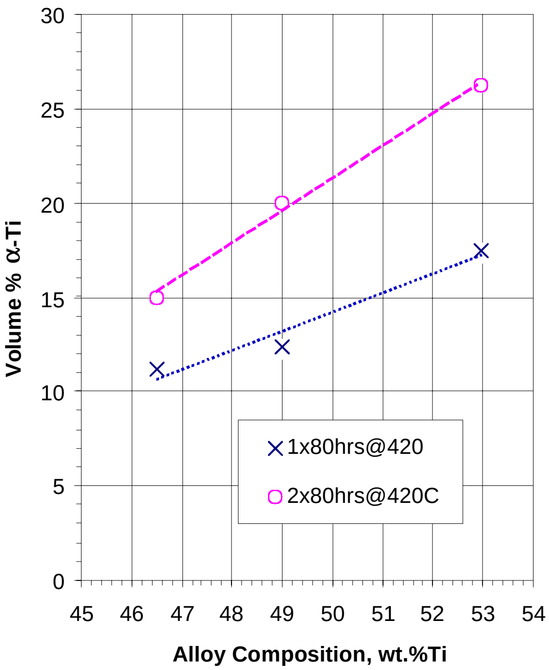

Fig. 5. A plot of composition versus volume of α-Ti precipitation after 1st and 2nd heat treatments, data from Lee et al.[11]

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