*P. J. Lee and D. C. Larbalestier, invited presentation at Interwire 2001, Atlanta, GA, May 16th 2001, published in the February 2003 edition of Wire Journal International in a slightly edited (no APC and cabling sections) and monochromatic form.*

# Niobium-Titanium Superconducting Wires: Nanostructures by Extrusion and Wire Drawing

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**Abstract:**

*Nano-fabrication is not new to the manufacturers of superconducting wires, in fact the dominant commercial superconductor, Nb-Ti, relies on a mechanically induced nano-structure for its remarkable properties. In this paper we show how these wires are manufactured so that their microstructure achieves the same scale as the superconducting vortex spacing.*

Recent attention on superconductors has emphasized the importance of the critical temperature, that being the temperature below which a material is superconducting. At present the highest temperature superconductor has a critical temperature of 138 K ($Hg_{0.8}Tl_{0.2}Ba_2Ca_2Cu_3O_{8+\delta}$).[1] The most important parameter, however, for commercial application, is the critical current density, which is the maximum current density that a superconductor can carry without dissipating heat. Nb-Ti has become the dominant commercial super-

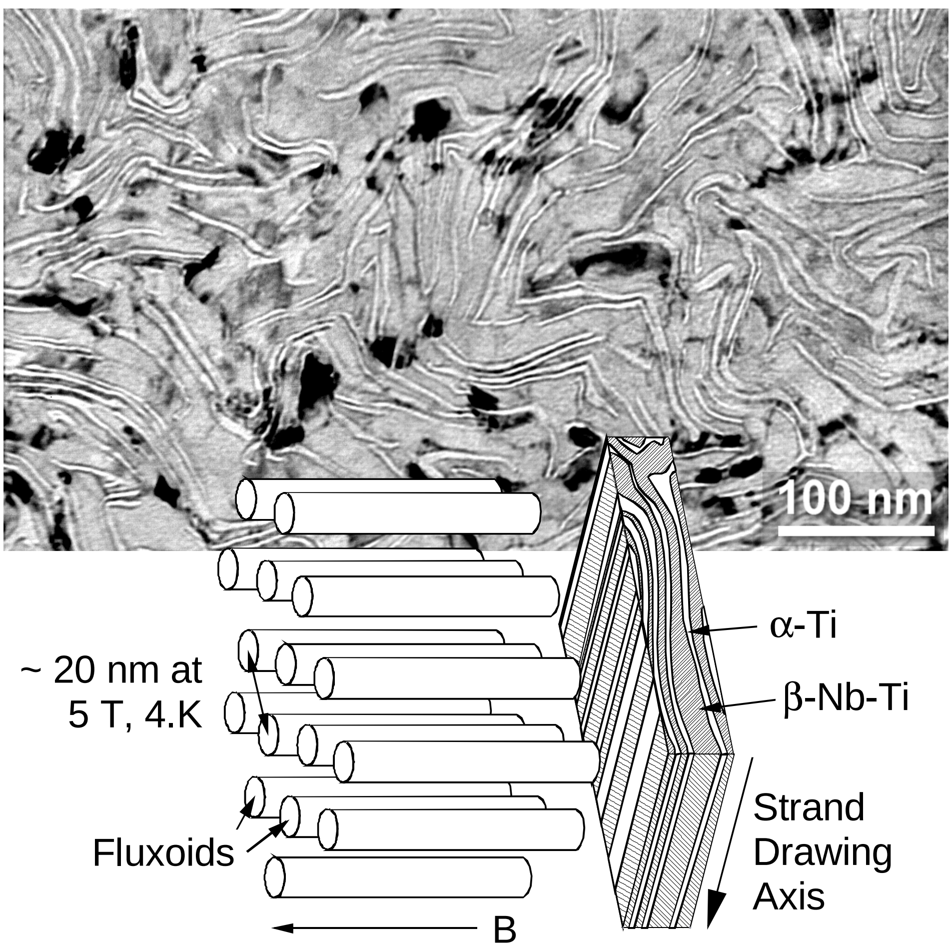


**Fig. 1 A transmission electron microscope image of the microstructure of a Nb-47 weight % Ti superconductor in transverse cross-section reveals a densely folded array of second phase pins which are 1 nm to 4 nm in thickness. For comparison, a schematic illustration of the fluxoid diameter and spacing at 5 T and 4.2 K is superimposed is compared with the three-dimensional microstructure below.**

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conductor because it can be economically manufactured in a ductile form with the prerequisite nano-structure that is needed for high critical current. In this a paper we will show how this is achieved in a ductile alloy like Nb-Ti.

## Nano-inhomogeneity

For the optimization of a superconducting microstructure a parallel can be drawn between increasing mechanical strength and increasing critical current density. In standard metallurgical practice, a metal can be strengthened by introducing a second phase that restricts the movement of dislocations.

For type II superconductors (all superconductors that can carry a significant critical current in magnetic field), high critical current density is attained by restricting the movement of the superconducting vortices through the superconductor by introducing regions of different superconducting properties. At the same time the barrier to vortex movement should not interfere with the overall transport of electrical current or significantly reduce the superconducting properties. Each flux vortex (“flux line”) has a non-superconducting (“normal”) core around which supercurrents circulate. The radius of the normal core equals the “coherence length”, $\xi$, of the superconductor and the radius of the supercurrent ring is equal to the “London penetration depth,” $\lambda_L$. Under most circumstances, the equilibrium arrangement for the vortices is a triangular pattern. The vortices experience a Lorentz force, $F_L$, which is equal to $J \times B$, where $J$ is the current density $B$ is the flux density, whenever a current flows. In Nb-Ti in an applied field of 5 T and a temperature of 4.2 K this gives an equilibrium fluxoid radius of 5 nm and spacing of 22 nm. Two methods have emerged to introduce the vortex-pinning sites into Nb-Ti. The first and predominant method is to heat treat Nb-Ti so that normal, that is non-superconducting, alpha-Ti precipitates are produced. The precipitate heat-treated microstructure is then deformed to its optimum nano-structure by wire drawing. $\alpha$-Ti is not superconducting and vortex cores bind strongly to it when drawn to ~1 nm thickness. In Fig. 1 we show the equilibrium fluxoid spacing (at 5 T, 4.2 K) superimposed on an optimized Nb-Ti filament microstructure. The precipitation heat treatment method is cost efficient but limits the volume of pinning sites to the volume of alpha-Ti to 18-28 % of the filament. An alternative method called the Artificial Pinning Center (APC) method mechanically creates the desired pinning array at large size and then uses extrusion and wire drawing to reduce the assembled array to the optimum size. This method provides freedom in both the choice of pinning material and in its quantity. Because the initial component sizes must be large enough to handle but must then be reduced to the 10 - 1 nanometer scale, multiple extrusions and re-stacks of the extruded components are performed. The APC method can produce the highest performance Nb-Ti composites for application below 5 T but the additional assembly and extrusion costs have so far kept this product from widespread application. This paper will consequently focus on the “conventional” heat treatment process.

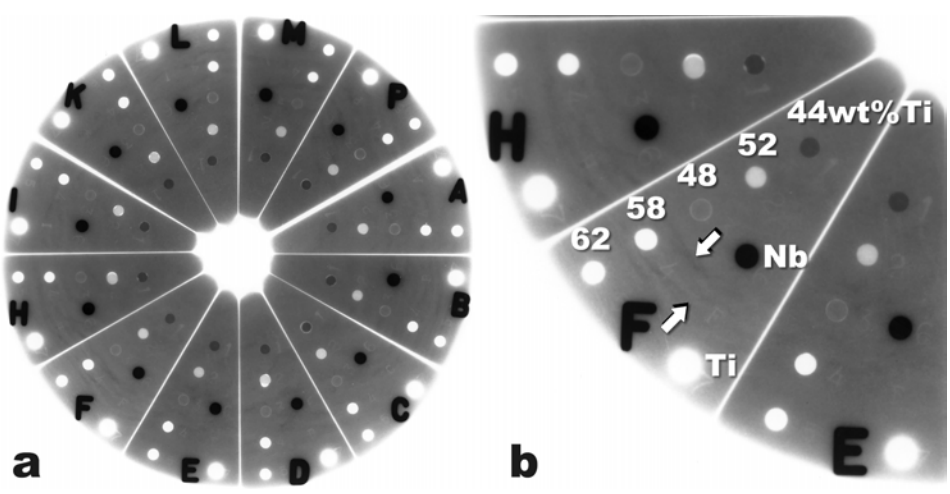


**Fig. 2 a) Flash radiograph standards created for the Superconducting Supercollider from single 150 mm diameter high homogeneity Nb-67 wt.% Ti slice. In b) a detail of slices E, F and H is shown. Arrows indicate tree-ring alloy inhomogeneity.**

## Micro-homogeneity

The upper critical field, $H_{c2}$ (4.2 K), peaks sharply in the range of 40 weight % Ti to 50 weight % Ti with a maximum value of 11.5 T at 44 weight % Ti. The critical temperature drops continuously over this range with increasing Ti content. There are only two stable phases in the Nb-Ti system, the BCC $\beta$-Nb-Ti and the hexagonal close-packed (HCP) $\alpha$-Ti with a composition of 1 to 2 atomic % Nb. The composition of Nb-Ti not only determines the superconducting properties but the amount of $\alpha$-Ti pinning center that can be formed and the precipitate morphology. The higher the Ti content the more pinning center that is formed and the lower the cost of the alloy (Ti is far cheaper than Nb). Under ideal processing conditions, the precipitate heat treatments produce alpha-Ti precipitates at grain boundary intersections only. [2] This type of precipitation produces a uniform array of precipitates controlled in distribution by the $\beta$-Nb-Ti grain size prior to heat treatment. Too much Ti, however, reduces the superconducting properties and increases the likelihood that Widmanstätten precipitation is formed in the Nb-Ti grains. The fine Widmanstätten precipitates significantly increase the hardness of the Nb-Ti filaments and may result in filament non-uniformity and ultimately strand breakage. Thus not only must the initial alloy composition be correct and uniform over the entire alloy billet, it must also be uniform on a sub-millimeter scale in order to control the strand ductility and the flux-pinning properties. Furthermore, with final filament sizes often below 10 µm and sometimes below 1 µm there must not be any hard inclusions or un-melted Nb in the initial Nb-Ti billet. The considerable difference in melting points between Ti and Nb makes this a particular hard alloy to manufacture. Because the vital $\alpha$-Ti

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**Fig. 3 Final 9 µm Nb-47 wt.% Ti filaments from a composite fabricated by IGC-AS with inset the original Nb-Ti alloy billets (courtesy Wah Chang) and the clean-room assembly of Nb-Ti rods into a Cu extrusion billet (courtesy OI-ST). The filaments in the background image have been revealed by etching away the Cu stabilizer matrix.**

precipitation behavior is so sensitive to composition, it was not possible to develop an optimum process understanding until homogeneous alloys were first produced in the early 1980s. The alloy composition that has become standard for this process is Nb-47 wt%Ti, which has proven to be the best compromise between precipitate yield for critical current density and alloy composition for the other superconducting properties. Although local compositional variations are required to be less than ± 1.5 wt. % Ti, a full micro-chemical analysis of each billet would be prohibitively expensive. Instead, flash radiographs are used on billet slices in order to reveal chemical variations across the billet radius. In Fig. 2 we show a flash radiograph standard developed for the Superconducting Supercollider Project.[3] The flash radiograph standards are made from a single slice of a high homogeneity 150 mm Nb-47wt%Ti billet by cutting it into 12 segments and inserting alloy pegs of known composition. In the detail of segments E, F and H shown in Fig. 2b you can observe that the tree-ring inhomogeneity indicated by the arrows is well within the contrast range of the 44 and 52 pegs. Ti-rich regions, called "freckles" after their appearance in the flash radiographs, indicate a non-ideal melt. Billets containing freckles command a lower price. Impurity content is kept low and consistent but recent work has shown that increasing the allowable content of iron can considerably refine the precipitate size without adversely affecting the precipitate volume.[4]

In summary the desired properties of the initial alloy billet are[5]:

1. The correct overall alloy composition to optimize $H_{c2}$, $T_c$ and precipitation for pinning. SSCL specifications are typical at 47 wt%, + 1 wt%.

2. Uniform composition over the entire billet to ensure optimum physical and mechanical properties over the entire filament.

3. Chemical homogeneity on a microstructural level in order to ensure uniform precipitation of the correct morphology. Some level of coring is inevitable but with good control this can be kept within ± 1.5 weight % Ti.

4. Low and controlled levels of impurity elements in order to ensure predictable superconducting and mechanical properties.

5. Elimination of hard particles (typically Nb-rich) which do not co-reduce with the alloy so as to avoid filament drawing instability and strand breakage. The exterior of the final Nb-Ti rod must also be free of hard particles and must be smooth enough that it does not easily pick up particles during subsequent handling. Shaving before installation on multi-die drawing machines is common.

6. A fine (typically ASTM grain size 6 or smaller) and uniform grain size, since grain boundaries in the Nb-Ti matrix control the distribution of precipitate nucleation sites. A fine grain size also improves diffusion barrier uniformity. Where high critical current is less important a larger grain size has been used to increase ductility.

7. Low hardness (typically a Vickers hardness number of 170 or less) in annealed starting rods to ease co-deformation with softer stabilizer material.

## Strain Space

The diameter of the initial cast Nb-Ti ingot ranges from 200 mm to 500 mm and this is typically reduced to 150 mm by hot forging before being fully annealed in the single phase β region (approximately 2 hr at 870 °C). In Fig. 3 we compare the initial Nb-Ti alloy billet with the final filaments from the superconducting wire. The dimensional reduction is performed by extrusion and wire and rod drawing with as little cold work loss as possible. In Fig. 4 we illustrate the fabrication route in terms of microstructural development and engineering true strain with key steps in the process. The total strain available for developing the microstructure is called the

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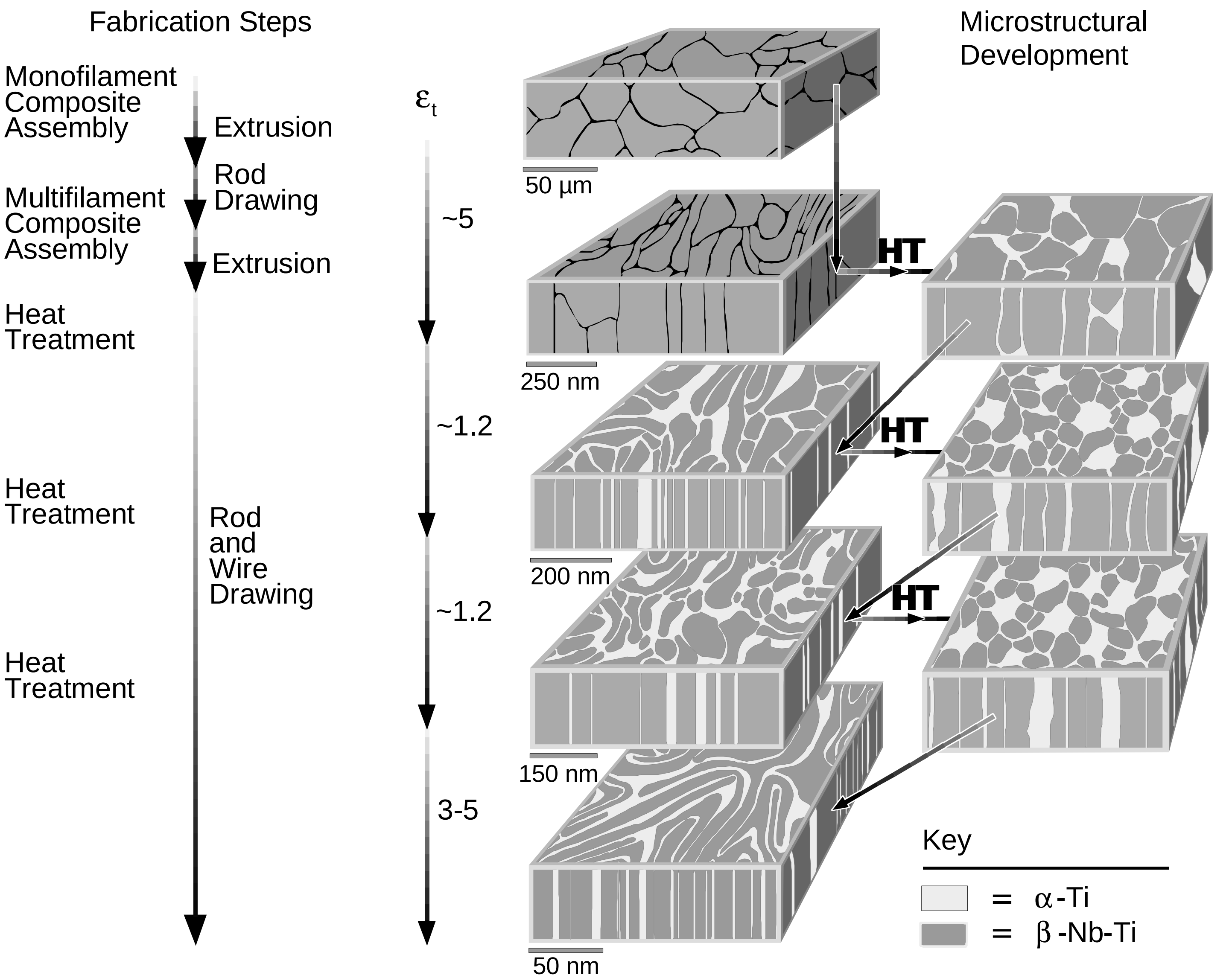


**Fig. 4 The key processing steps and microstructural changes produced by heat treatment and drawing in the manufacture of Nb-Ti superconducting strand by the "conventional" process.**

*strain space* and is determined by the strain between the final recrystallization anneal and the final strand size. If the final recrystallization anneal is carried out at a diameter of 30 mm and the final filament diameter is to be 9 µm, a total strain space of 16.2 is available (2 × ln (30000/9)). In practice the effective strain space is reduced a little during "warm" extrusion at 550-650 °C. The amount of strain lost can be estimated from the hardness of the Nb-Ti which increases with cold work.[6] The strain space can be further subdivided into three regions each requiring a minimum cold work strain to be effective:

1. Prestrain, $\varepsilon_p$, the cold work strain before the initial precipitation heat treatment (~5).
2. The inter-heat treatment strain, $\Delta\varepsilon_{HT}$, the strain required between precipitation heat treatments (~1)
3. The final strain, $\varepsilon_f$, the strain required to reduce the precipitate size to final optimum pinning size (4-5).

Avoiding hot-working and reducing the temperature of extrusion to the minimum required for good bonding is important because cold work plays a critical part of the process.

The cold work performs seven primary functions:

4. Encouraging the formation of the preferred precipitate phase and morphology.[7,8]
5. Increasing the density of precipitate nucleation sites.
6. Increasing the grain boundary density thereby, increasing diffusion rates (grain boundary diffusion being considerable faster than bulk interdiffusion).
7. Reducing the average diffusion distance to the precipitate nucleation site.

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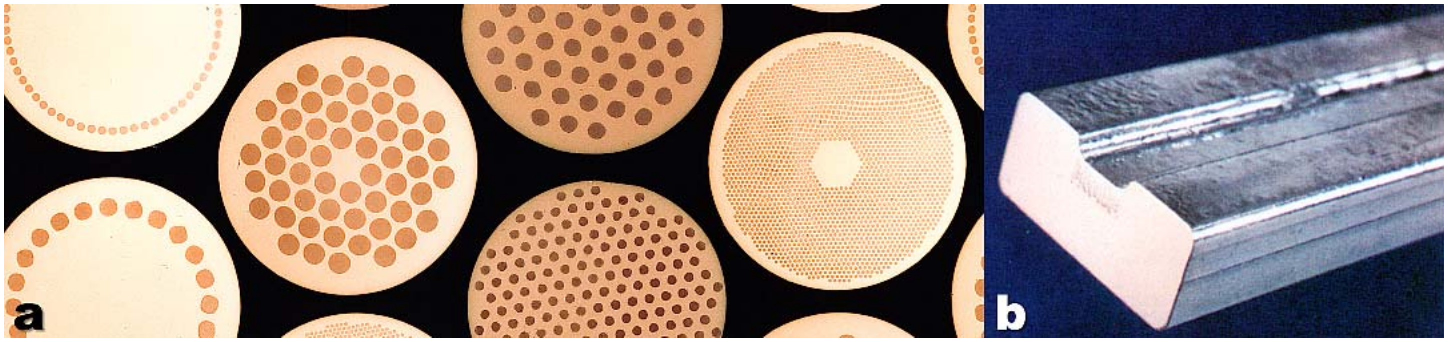


**Fig. 5 a) Strand is manufactured in a wide variety of geometries and that range expanded after wire drawing by such techniques as b) wire-in-channel (images courtesy of OI-ST).**

8. Increasing the volume of precipitate by multiple strain/heat treatment cycles.[9]
9. Improving micro-chemical homogeneity by mechanical mixing (aided by plain strain inter-curling of β-Nb-Ti grains), both prior to heat treatment and after heat treatment when local Ti depletion may have occurred.
10. Reducing the precipitate dimensions from the precipitation scale of 100 nm to 300 nm diameter to the pinning scale if 1 nm to 5 nm.

The first fabrication step is to combine the superconductor with a stabilizer.

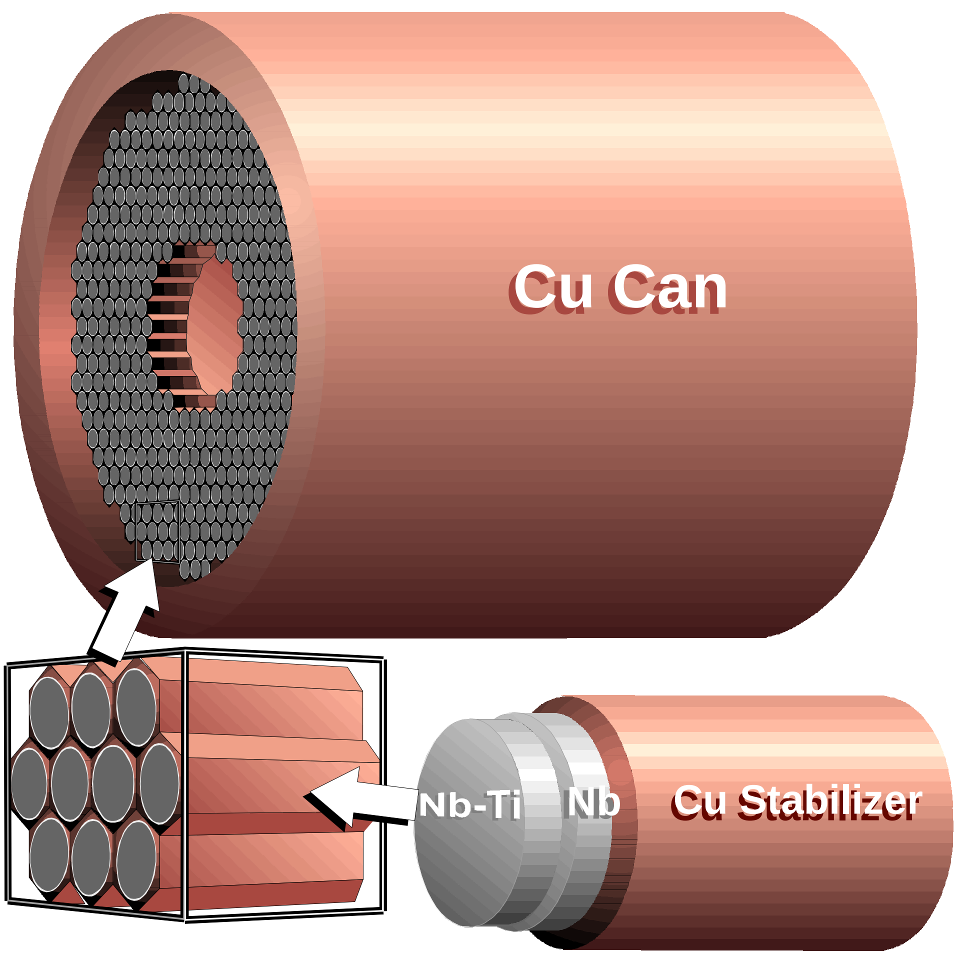


**Fig. 6 Schematic illustration of composite billet assembly starting from a Nb-Ti rod, a diffusion barrier wrap and a Cu extrusion can.**

## Electromagnetic Stabilization

All superconducting wires are in fact composites with filaments of the superconductor embedded in a matrix of high electrical and thermal conductivity. The matrix material provides electrical and thermal stability and protection from burnout when the superconducting device reverts to the normal state ("quenches"). For Nb-Ti high purity Cu has the required electrical and thermal conductivity, high heat capacity, good strength at both low temperature and during processing and the ability to be co-deformed with the Nb-Ti. More expensive and more difficult to co-deform than copper, high purity aluminum has a greater in-field thermal conductivity and electrical conductivity, as well as a lower heat capacity, a lower density and greater radiation resistance than Cu. Because it is so difficult to co-process, it is most commonly added after the strand has been processed (reference 10) as in the example shown in Fig. 5 where a Cu-stabilized strand has been continuously soldered into channel in an aluminum conduit. Cu-Ni can also be found as a stabilizer for ac applications where a high resistivity matrix is required to reduce eddy current loss in the matrix and coupling between the sub-micron filaments (for example references 11 and 12).

## Diffusion Barrier and Billet Assembly

When copper is used as an interfilamentary stabilizer it will react with the Ti in the Nb-Ti filaments during precipitation heat treatment producing hard Cu-Ti intermetallics on the filament surfaces.[13] On subsequent drawing, the intermetallics

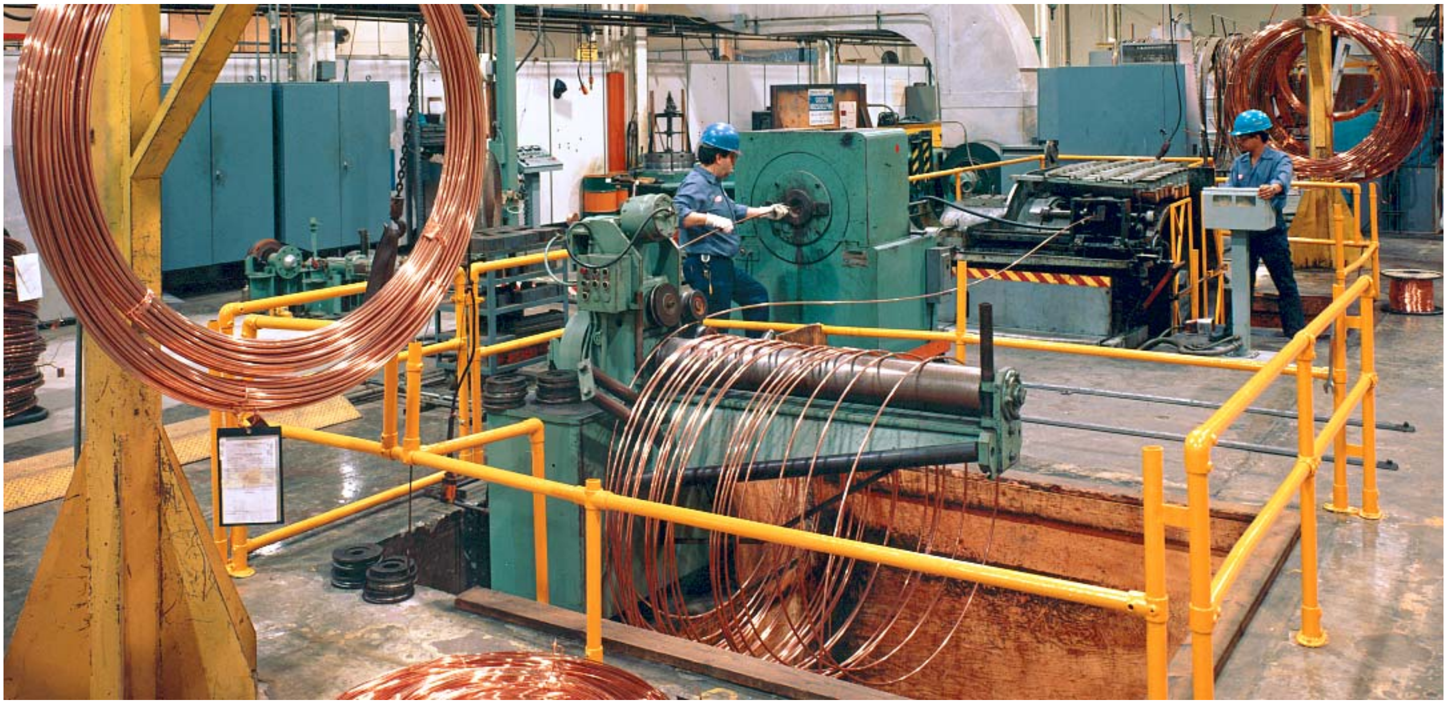

**Fig. 7 A large size draw of Nb-Ti superconducting strand on a bull-block. Photograph courtesy of Oxford Instruments-Superconducting Technology.**

produce sausaging of the filaments. It is important to reduce sausaging to minimum for two reasons. Firstly, because a local restriction in a filament results in the maximum current density in that area being exceeded, requiring the excess current to be transferred resistively through the stabilizer to adjacent filaments. Secondly the sausaging may result in strand breakage. Very long lengths of unbroken strand are desired because joints between strands are not superconducting. By placing a layer of Nb between the Cu and the Nb-Ti the reaction can be suppressed. [14] The smaller the final filament diameter the larger the volume of barrier that is necessary to suppress intermetallic formation. [15] For a final filament diameter of 6 µm, the Nb diffusion barrier required to prevent intermetallic formation represents 4% of the non-stabilizer area and this increases to 9% for 2.5 µm filaments. Conversely, for very large filament applications, a barrier may not be needed. When required the barrier is most commonly applied by wrapping Nb foil around the Nb-Ti ingot and then placing the wrapped ingot in an extrusion can made of the inter-filamentary stabilizer. A subsequent warm extrusion bonds the three components together, creating a monofilamentary composite. In Fig. 6 we show the key elements in the composite assembly. The monofilament is rod-drawn so that it can then be re-stacked to create the final multifilamentary composite which may contain from 50 to 4000 filaments. For even greater numbers of filaments (strands with up to 40,000 have been fabricated) a further re-stack and extrusion may be used. All billet assembly should be performed in a clean-room environment because any hard particulate incorporated into the assembly could result in strand fracture at small size. The thickness of the Cu around each filament is important because it controls both the drawability of the composite and the strand magnetization, which is associated with filament coupling, that occurs when the filaments are too close. Gregory et al.[16] have shown that for optimum mechanical stability in Nb-Ti/Cu multifilamentary composites, a filament spacing to filament diameter (s/d) ratio of 0.15 (ratios of 0.15 to 0.20 are now typical). This ratio fixes the ideal Cu wall thickness for the monofilament. Additional stabilizer may be added during multifilamentary billet assembly by increasing the wall thickness of the extrusion can or adding a center core. Increasing the outer Cu thickness reduces the impact of damage to the strand surface. Alternatively, adding a Cu core may reduce the chances of center-burst during extrusion. Ghosh et al.[17] established that for DC magnet application a minimum Cu thickness of 0.4-µm to 0.5-µm was required to reduce the magnetization associated with proximity filament coupling. For s/d ratios of 0.15 to 0.20, the minimum Cu thickness requirement limits the minimum filament diameter to ~3 µm when using a pure Cu matrix. If smaller filaments are required, Ni or Mn can be added to the Cu between the filaments.[11,18,19]

### Precipitation Heat Treatment

Precipitation heat treatments are applied after sufficient cold work has been introduced after the multifilamentary extrusion. If too little cold work is applied before heat treatment, Widmanstätten precipitation will occur, causing increased hardness (reducing drawability) and producing a non-optimum precipitate size and distribution. The amount of prestrain required increases linearly with weight % Ti.[20] Consequently a higher prestrain is required to suppress Widmanstätten in a chemically inhomogeneous alloy than would be required in a homogeneous alloy. For Nb-47wt% Ti, the required prestrain is 5 but this rises steeply to 9 for a 55 wt% Ti alloy. Heat Treatments are typically at 375 ºC to 420 °C, ranging from 20 to 80 hrs in duration. An initial heat treatment will only yield approximately 10 volume % precipitate. By applying additional cold work strain, the effect of the slow diffusion rate at these temperatures is again overcome and more precipitate is produced. The strain between heat treatments is 0.8 to 1.5 and at least three heat treatments are usually applied. A linear relationship between the optimized critical current density and the volume of precipitate Nb-47 weight % Ti alloys.[21] 20 % of the volume as α-Ti precipitate is sufficient to produce a critical current density of > 3000 A/mm² at 5 T, 4.2 K. Increasing Ti content increases the precipitation rate and amount but higher pre-strains are required.

## Final Wire Drawing

Wire drawing of the BCC β-Nb-Ti produces a plain strain condition which is supported by inter-curling of the Nb-Ti grains. This results in distortion of the α-Ti precipitates into densely folded sheets during final wire drawing. The folding process rapidly decreases the precipitate thickness and spacing with a dependence of $d^{1.6}$ (where d is the strand diameter) and increases the precipitate length per area with a dependence of $d^{-1.6}$, as measured by Meingast et al. [22] The critical current density increases as the microstructure is refined until it reaches a peak, after which there is a steady decline. The peak in critical current density for a monofilament or a multifilamentary strand with uniform filaments occurs at a final strain of approximately 5. If the filaments are non-uniform in cross-section (sausaged) the peak occurs earlier and at a lower critical current density. A strand that has a premature (and lowered) peak in critical current density during final drawing is described as extrinsically limited because it has not attained the intrinsic critical current density of the microstructure. The most common sources of extrinsic limitation is sausaging of the filaments due to intermetallic formation or lack of bonding between the components of the composite. High performance strand has sausaging reduced to a very low level (a coefficient of variation for the filament cross-sectional areas of approximately 2%). With tight quality control, uniform properties and piece lengths exceeding 10 km should be expected. In Fig. 7 we show a large size draw on a bull-block being performed at Oxford Instruments-Superconducting Technology in Carteret New Jersey.

Just before a multifilamentary strand has reached final size it is usually twisted about its drawing axis. The twisting is required to reduce flux-jump instability caused by varying external fields, instabilities caused by self-field, and to reduce eddy-current losses. The tightness of the required twist increases with the expected rate of change of field. The required twist pitch for strand for the Superconducting Super Collider, a relatively steady-state magnet, was approximately 80 rotations along the drawing axis per meter, for ac application with a similarly sized strand the number of twists per meter might be 300. The twisting occurs just before the strand is reduced to final size so that it can be locked in by a final drawing pass or by final shaping. Final shaping of the strand cross-section using independently adjusted rollers operating along the strand surface can produce square or rectangular cross-section filaments if required. Individual strands can be cabled or braided together to form a conductor with a higher current carrying capacity. The most common design for Nb-Ti magnets is the Rutherford cable, which consists of a fully transposed, flat cable. Using this approach, high aspect ratio cables can be produced with as many as 46 strands. [23] Strands may also be drawn into a larger conduit or combined with external stabilizers.

## The Present and Future

After 30 years of development high performance ductile Nb-Ti/Cu superconducting strands are manufactured as a commodity product for applications where large, uniform magnetic fields are required (e.g. MRI, NMR, accelerator magnets). A precise 1-2 nm scale microstructure is produced in km lengths in strands that are strong and very tough. For MRI application the global annual demand was approximately 60,000 to 70,000 km in the year 2000, a strand value of $50M. Standard production procedures rarely produce critical current densities exceeding 3200 A/mm² at 5 T, 4.2 K yet laboratory scale strand now exceeds 4000 A/mm² using high impurity alloys. Investigating the use of high impurity alloys to modify the precipitation behavior of Nb-Ti could yield significant improvements in performance in the future.

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