A version of this article with abbreviated references will appear in "*100 Years of Superconductivity*" Editors: Horst Rogalla, University of Twente, Enschede, The Netherlands and University of Colorado at Boulder, USA; Peter H. Kes, Leiden University, The Netherlands. Unfortunately the publishers of the book, CRC / Taylor & Francis, rushed the book into press without correcting the many serious errors that they had introduced, despite the protests of the editors and contributors. Please contact the authors directly for their corrected contributions: http://www.eucas2011.org/Book/ListOfAuthors.php This version contains full references and links and has been made available for academic use only.

11.2 Nb-Ti - from beginnings to perfection.

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11.2.1 The High Field Revolution-in-Retrospect

Towards the end of a cryogenics session at the January 1955 Annual Meeting of the American Physical Society in New York, George Yntema (Cornell) reported that he had achieved a field of 0.71 T at 4.2 K in a 3 mm gap using a coil of cold-worked superconducting Nb wire (produced by Fansteel). The 4296 turns of 0.05 mm diameter Formvar insulated cold drawn Nb strand around a soft Fe core carried a reported 1.8 A at 888 A/mm².[1] In Ted Berlincourt's review of the early history of Nb-Ti, he pondered on what might have been if Yntema (who had shared laboratory space with him while they were graduate students in Cecil T. Lane's group at Yale) had his additional finding, that the Nb wire (0.05 mm in diameter) had carried 1.5 A in an externally applied field of 0.5 T (a field well above the H_{c2} of annealed Nb), reported in terms of the substantial current density of 740 A/mm².[2] Even though Yntema helpfully pointed out that "such windings should be useful in various cryogenic experiments," not much attention was paid to this first high current density superconducting magnet. In 1986 Yntema recalled that the only person he knew who had noticed his abstract was John Hulm[3]. Hulm had gone to work at Westinghouse Research Laboratories in Pittsburgh in 1954 after leaving the University of Chicago, where, at the instigation of Enrico Fermi, he and Bernd Matthias had been looking for new superconducting compounds (see Section 11.3)[4]. Indeed Hulm followed up his reading of Yntema's abstract by constructing a solenoid using enameled cold worked Nb wire which achieved 0.6 T in a 19 mm bore in 1955[5].

11.2.2 The High Field Revolution – the one that was noticed

Unaware of Yntema's work, Stan Autler at Lincoln Laboratories published his first paper on Nb solenoids in 1959 [6] and oversaw the construction of a 0.43 T solenoid using silk-insulated Nb wire (127 µm diameter), which reached a field of 1.4 T with an iron core[7]. He also suggested that even greater fields could be reached using materials such as Ti-Mo and Nb_3Sn. Learning of this work while visiting Lincoln Labs, Rudolf Kompfner, at that time the Director of Electronics and Radio Research at Bell Telephone Laboratories (BTL), asked Ted Geballe and Bernd Matthias if this technology could be used for a superconducting shield for masers; Geballe and Matthias indicated that they had been unable to get metallurgists to make the required alloys, and so Kompfner encouraged Earle Schumacher and Morris Tanenbaum of the metallurgy group to help

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[1] G. B. Yntema, "Superconducting winding for electromagnets," Phys. Rev. 98 (1955) 1197, Minutes of the 1955 Annual Meeting in New York City, Abstract W8; doi:10.1103/PhysRev.98.1144

[2] T. G. Berlincourt, "Emergence of Nb-Ti as supermagnet material", Cryogenics 27(6): (June 1987) 283-289 (1987); doi:10.1016/0011-2275(87)90057-9

[3] G. Yntema, " Niobium superconducting magnets," IEEE Transactions on Magnetics 23:2 (Mar 1987) 390-395; doi:10.1109/TMAG.1987.1065154

[4] J. Hulm, "Superconductivity research in the good old days, "IEEE Transactions on Magnetics 19:3(1983) 161-166; doi:10.1109/TMAG.1983.1062255

[5] Private communication to G.B. Yntema reported in 3.

[6] S. H. Autler, "Superconducting Electromagnets", Bull. Am. Phys. Soc. Ser. 11. Vol. 4, (1959) 413

[7] S. H. Autler, "Superconducting Electromagnets," Review of Scientific Instruments 31:4 (1960) 369-373; doi: 10.1063/1.1716985

out[8]. Working under Tanenbaum at the time was J. E. Kunzler, who was diverted from his studies of transport in metals to lead the new superconductor effort in the metallurgy group[9].Kunzler first started work on Mo-Re (discovered by Hulm in 1955 [10]). An ingot was float zone melted and the eventual 178 µm diameter monofilamentary wire was plated with Au for turn-to-turn insulation at the suggestion of Ted Geballe [11]. As outlined in this patent [11], the advantage of using a good-conducting normal metal as "insulation" was that the coil windings could be decoupled from each other while the filament was superconducting but would couple and protect the magnet from destruction when the wire entered the normal state and started to dissipate I^2R heat. Furthermore, metallic "insulators" were much more space-efficient than organic insulation. The resulting 30,000 turn solenoid attained 1.5 T, to their delight matching closely the field predicted by their short sample tests[12]. A resistive solenoid capable of 8.8 T was available at Bell Labs and the next available time was booked to test the highest fields that could be sustained by superconducting strand. To make things more interesting, a wager was initiated between Tanenbaum and Kunzler; Tanenbaum would provide a bottle of scotch for every 0.3 T achieved over 2.5 T while Kunzler would provide one Beefeater martini for each week a much overdue paper on magneto-thermal oscillations was delayed.[13] Because Nb_3Sn had the highest critical temperature, it was also expected to have the highest critical field, but the intermetallic compound was too brittle to wind a solenoid. However, Ernie Buehler, who had fabricated the Mo-Re ingot, developed a method of making Nb_3Sn precursor strand using a mixture of Sn and Nb powders in a Nb tube, providing a route to a wind-and-react magnet. To their great surprise**Error! Bookmark not defined.**, a rectangular rod of bulk Nb_3Sn, that had been sintered and then melted at 2400 °C, was still superconducting at the maximum field of 8.8 T on the very first day of testing, December 14th 1960. Not only did this represent 21 bottles of scotch (the wager with Tanenbaum would eventually be halted at 10 T) but it exceeded the ~7 T estimate for critical field by another group at BTL earlier in the year[14]. Perhaps because of the prevalence of the sponge theory of superconductivity at the time (which did not predict useful high field transport currents in superconductors) Bozorth *et al.*[14] did not note the significance of the current in the tested material (Berlincourt later estimated a critical current density of ~60 A/mm² at 7 T from their magnetization data)[2,15] but to magnet builders the new strand data represented a revolution in the potential of superconductors for magnet application. They next tested the strand and it carried 50 times more J_c than the bulk sample[16]. The best powder-in-tube (PIT) strand with 10%

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[8] J. R. Pierce, "Rudolph Kompfner 1909-1977, A Biographical Memoir, National Academy of Sciences, 1983; http://books.nap.edu/html/biomems/rkompfner.pdf

[9] J. Kunzler, "Superconductivity in High Magnetic Fields at High Current Densities," Reviews of Modern Physics 33:4 (10, 1961) 501-509; doi:10.1103/RevModPhys.33.501

[10] J. K. Hulm, "Superconducting Rhenium Alloys and Compounds," Phys. Rev. 98 (1955) 1539

[11] Theodore H. Geballe, "Insulated Superconducting Wire," US Patent number: 3109963, Filing date: Aug 29, 1960, Issue date: Nov 5, 1963

[12] J. E. Kunzler, E. Buehler, F. S. L. Hsu, B. T. Matthias, and C. Wahl, "Production of Magnetic Fields Exceeding 15 Kilogauss by a Superconducting Solenoid," *Journal of Applied Physics* 32:2 (1961) 325-6; doi:10.1063/1.1736002

[13] J. Kunzler, "Recollection of events associated with the discovery of high field-high current superconductivity," IEEE Transactions on Magnetics 23:2 (1987) 396-402; doi:10.1109/TMAG.1987.106515

[14] R. M. Bozorth, A. J. Williams, and D. D. Davis, "Critical field for Superconductivity in Niobium-Tin," Phys. Rev. Lett. 5:4 (1960): 148; doi:10.1103/PhysRevLett.5.148

[15] T. G. Berlincourt, "Type II Superconductivity: Quest for Understanding," IEEE Transactions on Magnetics MAG-23(2): 403-412 (1987); doi:10.1109/TMAG.1987.1065156

[16] J. E. Kunzler, E. Buehler, F. S. L. Hsu, & J. H. Wernick, "Superconductivity in Nb_3Sn at High Current Density in

excess Sn (over that required to fully reacted the Nb powder) reached almost 1500 A/mm² at 8.8 T (4.2 K) and was given the lowest heat treatment temperature (970 °C). They remarked that “still higher current densities might be obtained at still lower reaction temperatures”. Indeed this was a prescient comment!

The era of high current high field superconductivity had truly begun and the race was on for the highest magnetic field solenoid and the best materials from which to make it.

11.2.3 First International Conference on High Magnetic Fields

James Wong had been experimenting with alloying Nb with Zr at the Wah Chang Corporation to increase the strength of reactor shielding for a nuclear powered plane but he was encouraged by John Hulm and Stan Autler (who had known him when he was a student at MIT)[17] to provide them with Nb-Zr wire, optimized for ductility at 25% Zr. Unlike the BTL PIT Nb_3Sn strand, which required the magnet to be wound in the not-yet superconducting state and then be heat treated at ~1000 °C, to make it superconducting, Nb-Zr (and Nb-Ti) was superconducting as made, making it a much more useful for early experimentation. By September 1961 Wah Chang was producing long lengths of Nb-Zr to support the increasing demand for laboratory scale superconducting magnets[15]. The First International Conference on High Magnetic Fields attracted some 500 attendees to MIT from November 1st to 4th, 1961 and a major source of interest were the latest reports of high field superconducting coils from Bell Telephone Laboratories (6.9 T, 1.5 K, Nb_3Sn 9, Westinghouse (5.6 T using Nb-25Zr)[18], Atomics International (5.9 T using Nb-25Zr)[19]. In “Magnetic Venture” Lady Audrey Wood, co-founder with Martin Wood of Oxford Instruments (OI), captured the excitement: “*Through the next three days of papers and discussions on current ways of making and testing high magnetic fields, the concourse buzzed with talk of the recent breakthrough in superconductivity.”* This culminated in a special session on the Saturday afternoon that was added to allow the presentation of the latest results. “*It was at this Saturday afternoon session that superconductivity really 'arrived' on the magnet-making scene.”*[17] The competition to report for the highest fields that afternoon was intense and yet the technology was still very primitive; Ted Berlincourt of Atomics International (AI) was surprised to learn later that both Bell Telephone Laboratories and Westinghouse used batteries to power their magnets (in the case of Bell Telephone Laboratories a simple car battery) and the AI team were winding their magnets with bare Nb-Zr monofilaments with bare Cu foil between the layers, having been unable to successfully apply an insulator[20]. The antics at AI inspired a limerick by W. J. Tomasch[15]:

A group of young men so frenetic
Struggle with matters magnetic,

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a Magnetic Field of 88 kgauss", Phys. Rev. Lett. 6(3): 89-91 (1961):3 (February 1, 1961): 89; DOI:10.1103/PhysRevLett.6.89

[17] A. Wood, “Magnetic Venture: The Story of Oxford Instruments,” Oxford University Press, Oxford, UK, 2001. ISBN13: 978-0-19-924108-8

[18] J. K. Hulm et al., M.J. Fraser, H. Riemersma, A.J. Venturino, & R.E. Wien, "A High-Field Niobium-Zirconium Superconducting Solenoid", 1962, in High Magnetic Fields; proceedings of the conference, held November 1-4, 1961, at the Massachusetts Institute of Technology, Cambridge, Massachusetts. Published jointly by the M. I. T. Press, Cambridge, Massachusetts, and John Wiley & Sons, New York (1962) 332

[19] R. R Hake, T. G Berlincourt, and D. H Leslie, "A 59-Kilogauss Niobium-Zirconium Superconducting Solenoid" 1962, *ibid*, p.341.

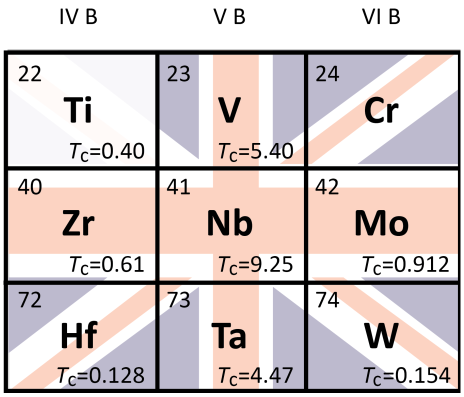
[20] Ted Berlincourt, personal communication for this article 2010.

Each day they conspire,
To wind super wire,
A pastime which some deem pathetic.

As John Hulm recalled, “Those tiny, primitive magnets were, of course, terribly unstable and tended to damage themselves on normalization, for reasons that are now well understood. One had to have faith to believe that these erratic toys of the low temperature physicist would ever be of any consequence as large engineered devices”[21]. New to the faith, Martin and Audrey Wood decided that evening that their fledgling company would build superconducting magnets and, on their return to the UK, they ordered one pound of Nb-25Zr from Jimmy Wong at Wah Chang (who would found Supercon as an independent commercial superconducting wire manufacturer). In March 1962 OI had built the Europe’s first magnet (4 T, 4.2 K, 18 mm bore) using the new superconducting wire. In the end, Nb-Zr proved to be a dead end because the magnets produced by OI using Cu-plated Nb-Zr strand deteriorated over time and they switched to multifilamentary Nb-Ti made by Imperial Metal Industries (IMI) as soon as it became available in March 1967[17].

11.2.4 God Save the Queen

Hulm and Matthias had started a study of solid solution alloys of the transition metals, such as niobium-titanium and niobium-zirconium (alloys that would fit well with the empirical electron to atom ratio rules established by Matthias in 1955)[22] while in Chicago but their attempts to sinter samples from powder in a poor vacuum system resulted in “a lot of nitride and oxidized samples”[23]. At Westinghouse, however, Hulm had excellent facilities for alloy melting and, with Richard Blaugher, would eventually measure the critical temperatures of all the nearest-neighbor body-centered cubic binary solid-solution alloys. Richard Blaugher, who had been hired by John Hulm in 1957, recalled the inspiration for their work on Nb-Ti for the memorial session for John Hulm at the 2004 Applied Superconductivity Conference (Richard Blaugher recalls it occurred just before Christmas 1959)[24]:



IV B	V B	VI B
22 **Ti** T_c=0.40	23 **V** T_c=5.40	24 **Cr**
40 **Zr** T_c=0.61	41 **Nb** T_c=9.25	42 **Mo** T_c=0.912
72 **Hf** T_c=0.128	73 **Ta** T_c=4.47	74 **W** T_c=0.154

Figure 11.2.1: The transition elements making up the ductile BCC superconducting alloys and their relationship to Hulm and Blaugher’s investigation of Nb-Ti. The first row of transition elements is notably missing from Matthias’s table of transition temperatures in reference 22.

> *One day I was sitting at my desk at Westinghouse R&D and John Hulm literally burst into the lab. “Blaugher” he says, “I have been thinking.” John started to sketch out on the blackboard the periodic chart for groups four, five and six. “We have studied V-Nb, Nb-Ta, V-Cr, V-Mo, Nb-Cr, Nb-Mo, Nb-W, Nb-Hf, and Nb-Zr” (which he emphasized by draw-*

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[21] J. Hulm, “Superconductivity research in the good old days”, *IEEE Transactions on Magnetics*, 19:3 (1983): 161-166; doi:10.1109/TMAG.1983.1062255

[22] B. T. Matthias, “Empirical Relation between Superconductivity and the Number of Valence Electrons per Atom”, *Physical Review* 97:1 (January 1, 1955) 74-76; doi:10.1103/PhysRev.97.74

[23] J. K. Hulm, J. E. Kunzler, and B. T. Matthias, “The road to superconducting materials”, Phys. Today 34:1 (1981) 34-43; doi:10.1063/1.2889964

[24] Richard Blaugher, personal communication for this article.

> *ing straight lines linking the various binaries)". We MUST do Nb-Ti!" Why I asked? John replied, because, "God save the Queen, we'll have the Union Jack." Needless to say with this overwhelming logic I proceeded to make up some samples to study this binary. Were it not for John's English background we would have omitted the most important binary for our study.*

Hulm and Blaugher published their wide-ranging study of transition element alloy superconductor critical temperatures in September 1961[25].

11.2.5 The Slow Emergence of Nb-Ti

The Atomics International (AI) group pursued critical field and current (key to application) [26], measuring their first Nb-Ti samples in April 1961[27]. A paper by B. B. Goodman at the IBM Conference on Fundamental Research on Superconductivity in June 1961[28] caused great interest at AI because it accurately predicted the upper critical field of U-Mo from the phenomenological Ginzburg-Landau-Abrikosov-Gorkov (GLAG) theory utilizing Berlincourt's experimental data on the transition temperature and normal state resistivity and Goodman's own measurement of the normal state specific heat. Berlincourt and Richard Hake proceeded to test a wide variety of U-Mo, Ti-Mo and Ti-V alloys and not only confirmed the GLAG predictions of a critical field that was independent of cold work but also showed that Nb-Ti possessed the highest critical field (~14.5 T) of all the ductile superconductors[29].

Early manufacturers of Nb-Ti included Atomics International (Ti-22 at.%Nb = Nb-65wt.%Ti), Westinghouse and Mitsubishi of Japan (Nb-Zr-Ti). Dating back to the days of Nb strands, the Nb and Nb-alloys strands were typically finely drawn monofilaments ~ 250 µm in diameter in order to get the high levels of cold work that gave useful critical current densities for magnet building.

Very large successful bubble chamber magnets were made at this time, starting with the 12 ft Argonne bubble chamber magnet in 1966 [30] which, as the current density of 7.75 A/mm² indicates, was so heavily protected with Cu that it was essentially a helium cooled copper magnet that happened to have several, large, superconducting strands embedded within its copper matrix (Nb-Ti is much more easily bonded to a Cu or Cu-Ni stabilizer matrix than Nb-Zr). The remarkable co-extruded conductor, developed by Jimmy Wong at Supercon, had six 4 mm wide filaments embedded in a 50 mm by 2.5 mm OFHC Cu matrix. The filaments were arranged in 3 separated pairs (2 pairs at each edge and 1 pair in the center) so that the conductor could be

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[25] J. K. Hulm and R. D. Blaugher, "Superconducting Solid Solution Alloys of the Transition Elements", *Physical Review* 123:5 (1961): 1569-1580; DOI: 10.1103/PhysRev.123.1569

[26] Note added after submission by Hake and Berlincourt: Whereas Hulm and Blaugher at Westinghouse were evidently the first to report the superconducting transition temperatures of Nb-Ti alloys, Berlincourt and Hake at Atomics International were the first to report their high upper critical fields and demonstrate that with suitable metallurgical treatment they could sustain high critical supercurrent densities. As discussed by Berlincourt 2,15 those properties, together with easy workability and affordability, single out Nb-Ti as the workhorse of technological superconductivity from among the a vast array of other superconductors as discussed by Berlincourt.2,15

[27] T. G. Berlincourt, R. R. Hake, & D. H. Leslie, Atomics International Laboratory Notebook B063551-B063562, 17 April 1961.

[28] B. B. Goodman, "The Magnetic Behavior of Superconductors of Negative Surface Energy," IBM Journal of Research and Development, 6:1 (1962): 63-67; an abstract is online at <http://bit.ly/e6cs2g>

[29] T. G. Berlincourt and R. R. Hake, "Upper Critical Fields of Transition Metal Alloy Superconductors", *Physical Review Letters* 9:7 (1962): 293-295; doi:10.1103/PhysRevLett.9.293

[30] J. R. Purcell, "DC Superconducting Magnets", Proceedings of The 4th International Conference on Magnet Technology, 19-22 September 1972, Upton, NY, pp: 201:201: http://lss.fnal.gov/conf/C720919/p201.pdf.

riveted at the joints without hitting the filaments[31]. At Fermilab (then the National Accelerator Laboratory (NAL)) John Purcell, builder of the 12 foot ANL bubble chamber magnet, was commissioned to make the magnet for the 15 foot bubble chamber. It too used large, superconducting filaments embedded within the copper matrix. The time constant for the eddy currents between the untwisted filaments to die down was on the order of a day. The magnet, which was very conservatively designed, had a long successful run at NAL.

A group under Ron Fast was responsible for design of some early beam line and detector magnets at NAL. These magnets had fairly open windings to maximize the wetted surface of the conductor and all used the principle of cryogenic stability developed by John Stekly [32]. Conductors used by Fast's group were small rectangular monoliths insulated by Formvar. There was a learning curve in the use of Formvar. Designers at the laboratory had to learn that there were dimensional standards for the base conductor, especially corner radii, that had to be met in order to ensure even coating of the insulating varnish. The magnets were so-called superferric magnets which used the superconductor to magnetize Fe cores and which did not require significant winding current densities. Many of these magnets were superferric versions of more conventional copper and iron magnets.

An important step towards the high J_c strand of today was the discovery by Atomics International in 1965 that they could get useful critical currents (sufficient for a 3.5 T coil) from their Nb-78 at.%Ti (65wt.%Ti) alloy using a single final size heat treatment for a few hours at 400 °C.[33,34] By using heat treatments to introduce precipitation of a non-superconducting phase, an additional method of raising critical current density was now available. The filaments were still "insulated" by electroplating with a thin (~20 µm thick) layer of Cu which was susceptible to oxidation[35]. A major improvement at AI was the in-house fabrication of Cu clad monofilament using Nb-Ti rods inserted in Cu tubes before wire drawing[36]. This was also compatible with a desire for thicker Cu layers for improved magnet stability. Larger currents could be carried by cabling multiple strands together and different combinations of Cu-clad monofilaments could be combined for different coil sections. Al McInturff and his colleagues at AI found that that similar critical current densities could be produced for a wide range of Nb-Ti diameters using their final size heat treatment of Nb-78 at.%Ti (65wt.%Ti) and they speculated that "it is now possible to replace many small conductors with one large conductor. For example, stabilized cables can be constructed in which one large Ti (22 at.% Nb) wire replaces many smaller wires, without loss in current capacity" (1967)[34]. They soon put the idea into practice for a multi sectioned large bore solenoid for Brookhaven, making one section of the magnet with one fairly large copper clad superconductor wire, with copper strands around it. To their great disappointment, this section was totally unstable and was replaced by a multi strand conductor

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[31] J. Purcell, "The Argonne bubble chamber supermagnet", *Magnetics, IEEE Transactions on* 23:2 (1987): 413-415. doi:10.1109/TMAG.1987.1065157

[32] Z. J. J. Stekly and J.L. Zar, "Stable Superconducting Coils," *IEEE Transactions on Nuclear Science,* 12:3 (1965): 367-372; http://accelconf.web.cern.ch/Accelconf/p65/PDF/PAC1965_0367.PDF

[33] J. B. Vetrano and R. W. Boom, "High Critical Current Superconducting Titanium-Niobium Alloy", *Journal of Applied Physics* 36:3 (1965): 1179-80; doi:10.1063/1.1714158

[34] A. D. McInturff, G. G. Chase, C. N. Whetstone, R. W. Boom, H. Brechna, and W. Haldemann, "Size Effect and Critical Transport Current in Titanium (22 at.% Niobium)," *Journal of Applied Physics* 38:2 (1967): 524-6; doi:10.1063/1.1709368.

[35] C. Laverick and G. Lobell, "Large High Field Superconducting Magnet System," *Review of Scientific Instruments* 36:6 (1965): 825; doi:10.1063/1.1719711

[36] G. G. Chase, C. N. Whetstone, A. D. McInturff, "Super History - An informal auto biography", 2010, for this article. Available on-line at: <http://bit.ly/g4tg7m>

cable[36]. Not only was the dependence of large filaments to be electromagnetically unstable unknown at the time but the importance of the twist inherent in the cabling process was not realized outside the Rutherford Laboratory/IMI collaboration at that time, a key insight that would burst on the scene one year later at the 1968 Brookhaven Summer School.

AI closed its superconducting strand operation in 1966 and the members of the AI team formed an influential diaspora across the US with Roger Boom and Bob Remsbottom moving to the University of Wisconsin, Al McInturff to Brookhaven National Laboratory and Clay Whetstone joined Eric Gregory and Bruce Zeitlin at Airco working on scale up techniques using extrusion of Nb-Ti and Cu strand.

Another step towards the multifilamentary strand of today came from an unlikely source; F. P. Levi, an associate director of Rola, a loudspeaker manufacturer in Melbourne Australia was looking for a way to create powerful permanent magnets using a microscopic array of magnetically insulated ferromagnetic filaments and came up with the idea that this could be achieved by "*repeated drawings of assemblies of iron wire encased in non-magnetic tubes. Len* [Rola founder A.L. C. Webb] *agreed enthusiastically, I was relieved of all other duties and within eighteen months and at a cost of about $20,000 . . . we discovered a new method of making permanent magnets*"[37]. Filaments as small as 20 nm were produced[38]. The idea was picked up by Rose and Strauss at MIT, where, the same technique was used to produce Nb filaments as small as 10 nm in diameter in a Cu matrix[39]. Cline, Rose and Wulff were also successful in producing a Nb-Ti in Nb matrix stack[40], shown in Figure 11.2.2, that would look very familiar to modern manufacturers but their attempt at the fabrication of a copper-clad Nb-Ti composite broke after the first

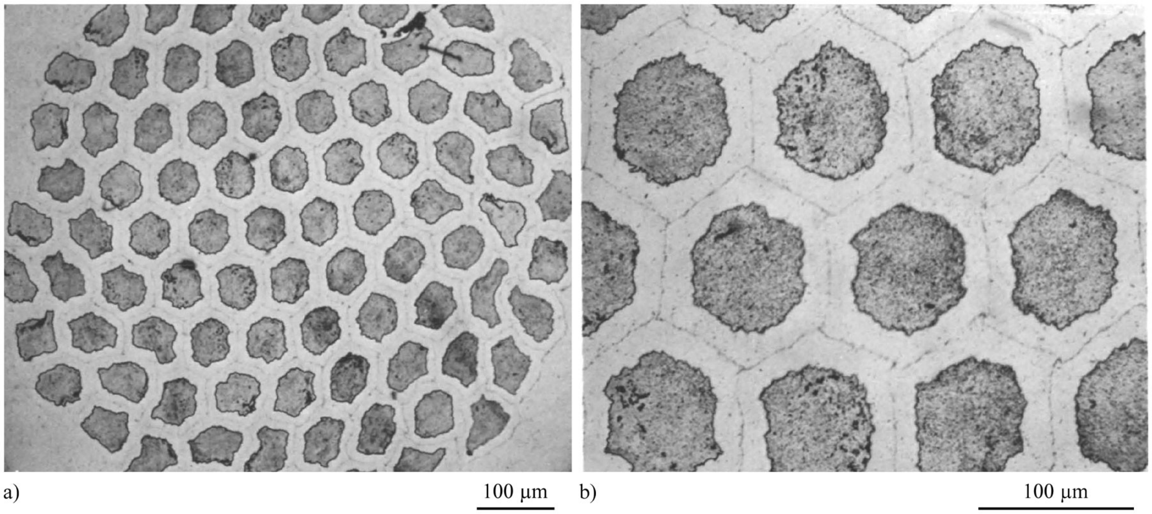

Figure 11.2.2: Nb60%-Ti40% in niobium matrix. These figures are taken from NASA CR 54103 [40] and used with permission of NASA.

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[37] Letter from Dr. F. P. Levi, formerly Associate Director, Rola (Australia) Pty. Ltd., March 6, 1976 quoted in R. J. Clarke, "Innovation in Australian High Technology Industries: Two Case Studies". Australian Economic Papers 18:32 (6, 1979): 89-102; doi:10.1111/j.1467-8454.1979.tb00647.x

[38] F. P. Levi, "Permanent Magnets Obtained by Drawing Compacts of Parallel Iron Wires", *Journal of Applied Physics* 31:8 (1960): 1469-71; doi:10.1063/1.1735864.

[39] H. E. Cline, B. P. Strauss, R. M. Rose, J. Wulff, "Superconductivity of a Composite of Fine Niobium Wires in Copper". *Journal of Applied Physics* 37:1 (1966): 5-8; doi:10.1063/1.1707892

[40] H. E. Cline, R. M. Rose, and J. Wulff, *Research on a Superconducting Niobium-Thorium Eutectic Alloy,* NASA CR 54103 (MIT, July 12, 1964)

bundling (which they suggested was most likely due to the limited ductility of the Nb-Ti alloy).

11.2.6 Rutherford CEGB/IMI Strand and the 1968 Brookhaven Summer School

The first commercial multifilamentary composite, in the Cu-stabilized form we understand it today, was the result of a collaboration in the UK between, first, the Central Electricity Generating Board (CEGB) under Peter Chester, and a little later, Rutherford Laboratory, under Peter Smith (after earlier experimental filamentary wire made for Rutherford by IRD Newcastle in late 66 - early 67)[42], and Imperial Metal Industries, IMI. The "Niomax M" commercial strand contained a hexagonal array of 61 filaments of Nb-60 at.% Ti (~44 wt.% Ti) in a Cu matrix[41]. The Rutherford group needed a wire with which they could build a synchrotron[42]. Production quantities also made their way to Oxford Instruments for magnet production in March 1967[17]. The concepts behind the development of the CEGB/IMI strand were first presented at the 2nd Magnet Technology Conference[43] but its major impact came from the Summer School held at Brookhaven National Laboratory in the USA in 1968 where the success of the new twisted multifilamentary strand in reducing flux jumps [44] was discussed with the leading large scale magnet designers,[45] much to the chagrin of IMI.[42]

11.2.7 Filamentary Superconductors (contributed by Martin Wilson)

> *In the early 1960's, not long after the discovery of high field type 2 superconductors, several manufacturers started to produce long lengths of good quality niobium zirconium and niobium tin wire, closely followed by niobium titanium. Although these wires performed well when tested as short samples immersed in a background magnetic field, they did not perform at all well when wound into magnet coils. Upon investigation, it emerged that one of the problems was a phenomenon known as 'flux jumping'. When field is applied to any type 2 superconductor, it responds by setting up persistent screening currents which try to impede the movement of flux into the centre of the wire – a bit like conventional eddy currents only, being superconducting, these current do not decay. If flux does move through a type 2 superconductor, it dissipates heat. Furthermore, the amplitude of the screening currents decreases if the temperature is raised. It is the combination of these two properties which gives rise to an electromagnetic thermal instability known as a flux jump, whereby the screening currents can suddenly collapse, releasing energy which takes the conductor above its critical temperature. The cure for flux jumping is very simple - subdivide the wire into fine filaments; for Nb-Ti, these filaments must be less than ~ 50 µm in diameter. For ease of handling and also to ensure stability of current flow, many filaments are embedded in a matrix of normally conducting metal such as copper to make a wire of diameter 0.5 mm to 1.0 mm. To avoid magnetic coupling between the filaments, the wire must be twisted like a rope.*

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[41] P. F. Chester, "Superconducting magnets", *Reports on Progress in Physics* 30:2 (7, 1967): 561-614; doi: 10.1088/0034-4885/30/2/305

[42] Martin Wilson, personal communication for this article 2010.

[43] P. F. Smith, in Proc. 2nd Magnet Technology Conference, Oxford, 1967, p. 543.

[44] P. F. Smith, M. N. Wilson, C. R. Walters and J. D. Lewin, "Intrinsically Stable Conductors", Proc. 1968 Summer Study of Superconducting Devices and Accelerators, BNL, p. 913.

[45] The Proceedings of the 1968 Summer Study on Superconducting Devices and Accelerators, June 10 – July 19, 1968, Brookhaven National Laboratory, Upton, NY, has been archived at: http://www.bnl.gov/magnets/Staff/Gupta/Summer1968/index.htm

The first filamentary superconductors were developed in the UK by the Rutherford Laboratory, in collaboration with Imperial Metal Industries using Nb-44wt.%Ti.

With flux jumping cured, it became possible to build magnets operating at very high current density and producing high magnetic fields. Careful design is still needed however to avoid the possibility of mechanical movement under the enormous electromagnetic forces, which can release frictional energy and cause unreliable performance. This problem remains with us right to the present day.

Fine subdivision also brings another advantage: it reduces ac losses. Although superconductors have zero loss under dc conditions, changing magnetic fields cause flux to move within the superconductor which dissipates heat. Reducing the filament size reduces this flux motion and hence the losses, in much the same that laminating a transformer core can reduce eddy current loss. Keeping the ac losses within reasonable bounds has made it possible to build large particle accelerators, such as the Tevatron, RHIC and LHC, in which the superconducting magnets must be ramped up from low field to high field so that they keep pace with the increasing energy of the particle beam. Finer filaments are needed for particle accelerators, typically in the range 5-10 µm. Even finer filaments, in the sub-micron range, have been tried with a view to building 50-60 Hz superconducting machinery for use in power generation and distribution, but unfortunately the refrigeration power needed to remove the residual ac losses at the very low temperatures involved is still too high for economic operation. There are however good reasons for hoping that, if filamentary conductors can be made with the newer high temperature superconductors, they will revolutionize the way that we generate and distribute electrical power.

11.2.8 After the 1968 Summer School

The impact of the 1968 Summer School was immediate; Bill Hassenzahl was working on a high intensity proton accelerator at the Los Alamos Meson Physics facility, using existing monocore strand to make quadrupole magnets of sufficient field (3.4 T)[46]. At less than half the 550 A design current, however, their prototype quadrupole quenched with a large “whoosh” of helium. “During the tests we learned of the new multifilamentary superconducting wire and decided to immediately build and test a racetrack with this advanced conductor”[47].

> *It reached about 320 A, and the quench had a bigger Whoosh, but there was another sound just prior to the opening of the relief valve. I heard a very distinct ping. Neither John Rogers nor Henry Laquer heard the sound. During the course of the design and testing of these magnets, the two of them had been my mentors in cryogenics and superconductivity. Henry Laquer was arguably the first person to observe flux creep in superconductors, and had the earliest patents on flux pumps*[48,49]*, though he did not call them by that name. Probably my only advantage was younger ears. As usual on experiments, this was late on Friday, and several more quenches yielded a small increase in current, and the same ping*

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[46] J. D. Rogers, W. V. Hassenzahl, H. L. Laquer, J.K Novak, R. W. Stokes, “Superconducting Quadrupole Doublet for the Los Alamos Meson Physics Facility”, *Journal of Applied Physics*, 42:1 (1971) 73-79; doi:10.1063/1.1659656

[47] W. V. Hassenzahl, personal communication for this article, 2010.

[48] Henry Laquer, "Superconductive electric switch: United States Patent: 3145284", August 18, 1964.

[49] Henry Laquer, "Incremental electrical method and apparatus for energizing high current superconducting electromagnets: United States Patent: 3150291", September 22, 1964.

> *that only I could hear. On Monday morning, we opened the cryostat and found that there had been significant mechanical motion in the coils. The source of the pings was readily determined to be from sheared rods that had been used to hold a separator in place. This phenomenon was not present in the other coils. When the rod had sheared, a significant fraction of the coil had moved by nearly 7 mm. Prior to shearing the rod it had moved by nearly 3 mm. We decided to remove other rods in a similar configuration and retest the coils.*
>
> *During this test, the coils exceeded the design current of 500 A without quenching, even though there was internal motion of about 1 cm at maximum current. The Whoosh was louder during several quenches in the 515 to 550 A range than it had been in earlier tests. Finally, two quadrupoles were constructed of the twisted multifilamentary wire. They were tested up to the design current, and were eventually used on one of the LAMPF beam lines.*

1.2.9 Making multifilamentary strand

Bill Marancik recalls his work with Eric Gregory in developing early multifilamentary Nb-Ti at the Air Reduction Central Research Laboratory (later AIRCO, Oxford AIRCO and eventually OST):

> "*we used various techniques using Nb-Ti rods inserted between concentric copper tubes, each rod was also separated by copper rods. This composite was swaged from about four inches to several millimeters, insulated with tape and wound into a coil. Because of the gross swaging the copper surface was covered with flakes and the magnet was completely shorted. Using the techniques for the manufacture of Flux core welding wire we wrapped copper foil around both Nb-Ti powder and also around Nb-Ti rods for stacking into copper billets. We then turned to inserting Nb-Ti rods into copper tubes, drawing these to various shapes such as squares, rectangles and finely into hexes. With the hex stacking a more compact configuration was formed in which we were able to produce the first fine multifilament conductor consisting of 98 filaments (we thought this was a huge number). The billet was four inches in diameter by a foot long and was extruded at the*

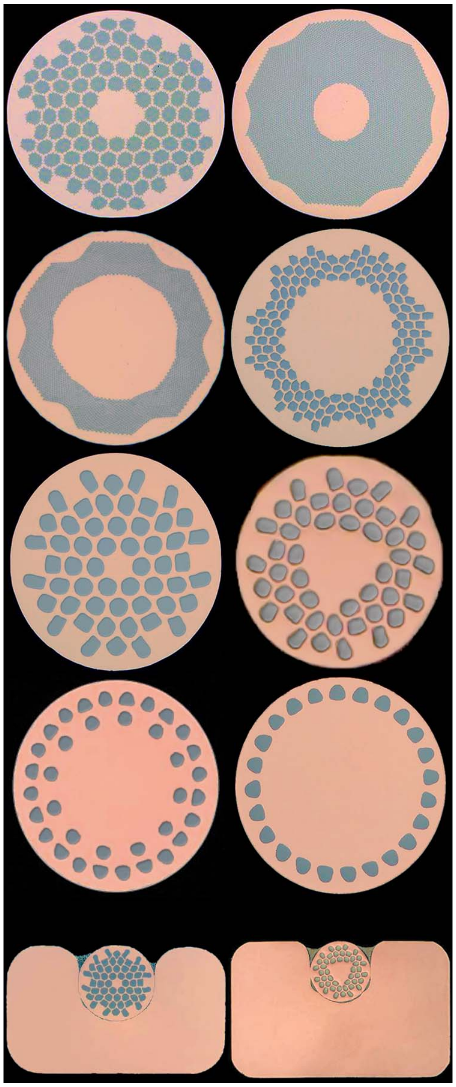

Figure 11.2.3: A variety of Nb-Ti strand and monolith cross-sections from just one manufacturer's current production. Images courtesy of Manfred Thoener of Bruker EST (BEST). The top left strand is a restacked composite for the LHC dipoles and has 7 μm diameter filaments. The large filament strands and strand in channel conductors towards the bottom are for MRI application.

DuPont Metal Center and drawn to final size”[50].

After his brief stay at Airco, Whetstone formed his own company, Cryomagnetics in 1967, to produce Nb-Ti strand and magnets and he was soon joined there by his former AI colleagues, Gordon Chase and Al McInturff. Wanting to produce multifilamentary Nb-Ti in a Cu extrusion can they found that gun drilling holes for the Nb-Ti rods into 24 inch Cu billets was not economical at the time, so they assembled billets by stacking 4 inch interlocking Cu pancakes, which were easy and cheap to drill on at a time[36]. They were now using a Nb-45 wt%Ti (62 at.%Ti) that had been identified as the optimum alloy by McInturff while still at AI (it was not published until much later)[51]. Optimized processing of this low-Ti strand required drawing strains after precipitation heat treatment, which meant that final wire drawing was occurring for filaments with flow stress much increased by the introduction of precipitates. Bonding the Cu pancakes required high extrusion temperatures which caused excessive formation of brittle Cu-Ti intermetallic at the Cu/Nb-Ti interface.

Airco was having similar problems, which were address using solutions that are used to this day:

> “*All our development billets were two, three or four inches in diameter and weighing a few pounds. Several problems became apparent; a Cu-Ti reaction caused hard inclusions to be formed, which was solved by inserting Nb foil between the copper and the Nb-Ti rods. The second problem the ID of the copper tubes turned out to present a contamination problem in the ID of the tubing. This could only be eliminated by forming the single strand composite by extrusion of an Nb ingot in a Cu can. This was drawn, hexed and stacked into an extrusion can. As with any composite where the yield strengths of the components are widely different, center burst is likely to occur and did with the initial billets. This was eliminated using the criteria developed by Betzalel Avitzur. Control of both drawing die angle and per-cent reduction as well as location of the filaments within the array eliminated this difficulty. This has been a key component of our processing in all subsequent conductor developments*[50].

The arrangement of filaments included an optimum filament spacing for mechanical stability[52].

11.2.10 The first strain Measurements (contributed by Jack Ekin)

> *Sometimes discovery is the result of the need for survival. A solid state physicist specializing in low temperature electrical measurements, I found myself as a postdoc thrown into the midst of a group of mostly metallurgists at the National Bureau of Standards specializing in fracture, deformation, and stress. Not much use for an electrical type. This was 1974 at the bottom of the great recession of the time. With few job prospects and*

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[50] W. Marancik, personal communication for this article, 2010.

[51] A.D. McInturff and G.G. Chase, “Effect of metallurgical history on critical current density in NbTi alloys”, *Journal of Applied Physics* 44:5 (1973): 2378-84; doi:10.1063/1.1662569

[52] E. Gregory, T. S. Kreilick, J. Wong, A. K. Ghosh, and W. B. Sampson, “Importance of spacing in the development of high current densities in multifilamentary superconductors”, *Cryogenics*, 27, p. 178, 1987; doi:10.1016/0011-2275(87)90016-6

one year left on my postdoc appointment, I had a two minute "bathroom conversation" with sponsor Mike Superczynski about looking for a possible connection (hopefully) between stress and electrical properties in practical superconductors. The literature from a decade earlier showed there was a very small stress effect on critical temperature, but no one had observed (or looked for, to my knowledge) any effect on the really important parameter, critical current. Fortunately, I got "funded" for $10 k, but never mind, that was enough to justify myself to the administration to at least have a first look.

I kluged together a stress rig made of a stainless steel cable running up out of the measurement cryostat, up over two pulleys, and down to a pan, which I progressively loaded with lead shot filled bags (yes real buckshot), 5 lbs each. I measured critical current while progressively loading the pan, until eventually the wire sample broke and the pan came crashing down onto the floor with a bang that made you jump. Also, I had no protective circuit to "kill" the power supply if there was a thermal runaway quench, so I ran the experiments with my trigger finger constantly poised on the power supply switch, ready to snap it off at the slightest sign of a rapid voltage rise across the sample. If I was too slow by a fraction of a second, my precious sample would melt into a shapeless blob.

Rutherford Cables
(contributed by Martin Wilson)

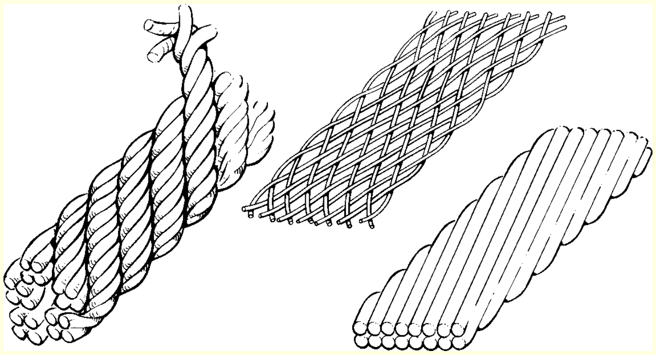

Figure 11.2.4: Three types of transposed cable: rope, braid and Rutherford.

A large synchrotron accelerator comprises a ring of some hundreds of magnets which focus the beam of particles and constrain it to follow a circular orbit. To make sure that the orbit is exactly circular and in the right place, it is essential for each bending magnet to produce exactly the same field, i.e. to carry exactly the same current. The easiest way of ensuring that all magnets carry the same current is to connect them in series. A power supply must apply voltage across the magnet terminals to overcome the self inductance and ramp the magnets up to follow the beam energy. When the magnets are connected in series, this voltage adds up around the ring. To keep the voltage down to a manageable level, the magnet inductance must be minimized, which requires high operating currents of ~ 10 kA. Such high currents require 30 – 50 wires to be connected in parallel and, because they have no resistance, it is difficult to ensure that the current distributes equally between the wires. To achieve such equal distribution, the wires must be perfectly transposed, i.e. they must all changes places with each other along the length of the cable. Fig 11.2.4 sketches three examples of transposed cables: a rope, a braid and a flat twisted cable similar to the Roebel bar. Prototype accelerator magnets were built from each type of cable but the flat twisted type gave the best performance, probably because it can be compacted to high density without damaging the strands. Because it was developed at Rutherford Laboratory, this cable has come to be known as Rutherford cable and has been used in all large superconducting accelerators built to date.

> *After many days of such tension between the expectation of the pan crashing down and vigilance at the power button, my nerves were frazzled. But there it was, a* reversible *stress effect on the critical current (and potentially a job)!*

Thus was made the first observation of stress/strain effects on the critical current of Nb-Ti, and later Nb_3Sn. An unassuming beginning, but the elegant scaling laws that grew out of this work have for decades served the important design work needed for large (i.e. high stress) superconducting magnets.

11.2.11 The birth of Nb-46.5wt%Ti

The Vietnam War and the ensuing recession also took its toll on the strand manufacturing community and Cryomagnetics closed its doors in 1971, leaving only three US wire vendors, AIRCO, MCA and Supercon. The poor state of the commercial superconducting strand business caused concern at Fermilab, which was planning a 1000 GeV superconducting upgrade to its recently completed Cu and Fe 400 GeV main ring. It knew that it would need both a competitive market for the large quantities of strand that would be required, as well as a consistent and high performance product. This incited a program to directly develop production methods for accelerator quality strand[53]. The upgrade to the accelerator, a project known at various times as the "Energy Doubler, the "Energy Saver" and the "Energy Doubler/Saver" and finally the Tevatron, required 774 dipole magnets to steer the beam and 216 focusing quadrupoles. The peak field of 5 T would mean that high strand current densities would be required. Cryostatic stability was not an option under these conditions. Adiabatic stability, ably covered in the Brookhaven Summer Study, required small (<50 µm diameter) twisted filaments, while the high strand current density implied a low copper to superconductor ratio (<2:1). Fermilab chose to make a 23-strand rectangular cable using 0.68 mm diameter strands. The cable configuration, known as a Rutherford Cable[54,55], is basically a flattened helix and is now the standard method for making accelerator magnets (see Rutherford Cables inset).

Each of the three domestic strand suppliers for the Tevatron had a proprietary alloy composition. In order to broaden competition and not be beholden to a vendor with a proprietary alloy, Fermilab decided to supply raw materials to the individual vendors. This also enabled a savings of time, as the laboratory had kits of materials on hand and delivery schedules from the fabricators were significantly reduced. The Tevatron would eventually consume 90% of the Nb-Ti that had ever been produced and the selection of alloy composition would have a major long term impact. Bill McDonald of Wah Chang recalls:

> "*Wire suppliers were buying 45Ti and 48Ti. Wah Chang was the primary (essentially the only) supplier. We had a meeting in the cafeteria at Wah Chang, including Paul Reardon FNAL, Bruce Strauss FNAL, Bob Marsh (Wah Chang salesman), and me (Bill*

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[53] B. Strauss, R. Remsbottom, P. Reardon, C. Curtis, W. McDonald, "Results of the Fermilab wire production program", *Magnetics, IEEE Transactions on* 13:1 (1977): 487-490; doi:10.1109/TMAG.1977.1059392

[54] G. E. Gallagher-Daggitt, Superconductor Cables for Pulsed dipole magnets, Rutherford Laboratory Memorandum No. RHEL/M/A25 (1975) Unpublished.

[55] C. Walters, "Magnetization and design of multistrand superconducting conductors". *Magnetics, IEEE. Transactions on* 11:2 (1975): 328-331; doi:10.1109/TMAG.1975.1058684

> *McDonald). We had met to generate the spec for the alloy to be used in the accelerator magnets. The technical review was finally settled by splitting the difference between 45 Ti and 48 Ti, so that no customer would be given a process advantage. I remember it as being proposed by me, and Bruce remembers it as being proposed by him. No matter. We all agreed and the official alloy was established as Nb-46.5Ti for the Fermi Lab magnets.*

The origin of the Fermilab billet filament stacking arrangement is recalled by Bruce Zeitlin:

> *It was in the early to mid-seventies while I was working for Dr. Eric Gregory at Airco (acquired by BOC) as a young supervisor/engineer of the superconducting wire pilot plant, that a challenge came to us from Westinghouse's Mike Walker. Their superconducting generator program required several thousand fine filaments in a monolithic rectangular conductor. At that time only several hundred filaments were possible due to the difficulties of stacking the hexagonal elements into the extrusion billet. Nb-Ti was typically clad with copper and drawn through a hexagonal die. The resulting rod could not be straightened to the required precision to stack the billet. It occurred to me that if we could obtain straight hexagonal tubing and precision straightened Nb-Ti rod, then assembly would be much easier. We did such. The first 1000 to 2000 filament conductors were assembled by stacking the hexagonal copper tubes and then inserting the precision straightened Nb-Ti rods. This was a quick and scalable process.*
>
> *After the acquisition of the technology and people by Magnetic Corporation of America (MCA), Z.J.J. Stekly's company, this technique was applied to the Fermi accelerator conductor. This is one of the few incidents in which non-accelerator requirements led to improvements in superconductor for accelerators.*

For what became known as the Fermi-kit, Fermilab supplied copper in hexagonally shaped OD and round ID tubes, as well as extrusion cans. The copper supplied was from Phelps-Dodge and had the highest residual resistivity ratio available at that time thanks to the use of virgin Cu rather than remelt (which inevitably includes Fe from recycling).

Bruce Zeitlin also recalled a less fortunate event that led to a standard improvement in the processing of the extrusion billets:

> *Los Alamos in the later part of the seventies required a mixed matrix conductor for John Roger's energy storage program. An ambitious conductor was designed with 2100 filaments of Nb-Ti clad with copper and 90/10 copper nickel alloy assembled in a 254 mm diameter copper nickel billet. Extrusion at RMI was a disaster. The billet stalled. The extrusion temperature was too low for the tough copper nickel. Inspection of the extrusion revealed that we had some major folds and creases in the billet. I entered Dr. Stekly's office at Magnetic Corporation of America in trepidation knowing that we had just cost the company and program 20 to 30 thousand dollars and delayed follow on contracts for the device. John calmly just asked what was the length to diameter ratio of the individual rods. He promptly stated that the internal component buckled, as there was enough free space within the billet such that the rods were unstable under the columnar load. This led to the standard practice of cold isostatic compaction of any billet with a large number of filaments. It was also important to the Fermi conductors as the void space introduced by the slide fit in 2000 tubes led to instances of internal buckling as well. It was a measure of the man that John almost instantly understood the problem and focused on the solution as opposed to chastising me.*

The Fermilab Tevatron strands were eventually manufactured by Intermagnetics General Corporation (now Luvata) and Magnetic Corporation of America (now out of business) using a FNAL recipe that used a single long precipitation heat treatment followed by a cold work final drawing strain used by Airco[56]. Cabling was accomplished by New England Electric Wire Corporation in Lisbon, New Hampshire. Art Greene of NEEC contributes this history of the FNAL cable:

11.2.12 Manufacturing of Cable for the Fermilab Tevatron (contributed by Art Greene)[57]

> *A decision was made on 23 as the number of wires in the Tevatron cable because the conventional cablers at New England Wire had 24 payoffs. Additionally, it was understood then that the geometry of the cross-section of a Rutherford cable required an odd number of strands. During the final manufacturing phase four cablers were utilized, the "Jumbo" cabler where most of the development work was done, two other planetary-type cablers and a tubular strander which ran at a much higher rpm.*
>
> *New England Wire worked with Fenn Manufacturing Co. to develop Turks-Head rolls which were used to shape the originally round 23-strand cable into the desired keystoned cross section. All of the Fermilab cable was rolled twice, first into a rectangular cross section and subsequently into the final keystoned shape. Special mandrels were designed with nose pieces to prevent crossovers of strands just prior to rolling. As magnet fabrication and testing progressed at Fermilab, it was realized by the magnet designers that some type of coating was required on the individual superconductor strands, so that the magnetic field quality would not be adversely affected by eddy currents generated in the cable during ramping of the magnets. The initial choice was to coat all strands with Stabrite, a 95wt % tin with 5wt % silver eutectic alloy. In late-1978 additional magnet testing showed that another type of coating system was required to further reduce eddy current effects. It was suggested that a copper oxide coating be created on the surface of the wire. Further investigation revealed the existence of an Ebonol coating process which was at that time commonly used to blacken the outside of caskets covered in copper foil. Finally, after extensive studies, it was decided by Fermilab that the best results for both good magnet stability and reduced eddy current effects came from cable with both Stabrite and Ebonol coated strands (11 and 12 respectively) then given the name Zebra cable. This decision somewhat complicated things, because all of the strand then available for making cable had already been coated with Stabrite. Next came the request to Robert Meserve to develop a method to remove the Stabrite from almost 10,000,000 feet of wire! A process to deplate the Stabrite was developed and two Ebonol coating lines were also constructed at New England Wire in addition to the original Stabrite lines. The first Zebra cable for magnet production was shipped to Fermilab on August 1, 1979.*
>
> *In late-1979 and 1980 a regular production process developed for manufacturing the*

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[56] P.R. Critchlow, E. Gregory, and B. Zeitlin, "Multifilamentary superconducting composites, *Cryogenics* 11:1 (February 1971): 3-10; doi:10.1016/0011-2275(71)90002-6

[57] Information contained in this summary of the fabrication of Rutherford-type cable for the Fermilab Tevatron comes partially from meticulous log books of daily events kept by Robert Meserve at New England Wire in Lisbon, New Hampshire. Robert, now partially retired as Vice President for Engineering, managed all development work and manufacturing of the superconducting cable for Fermilab magnets.

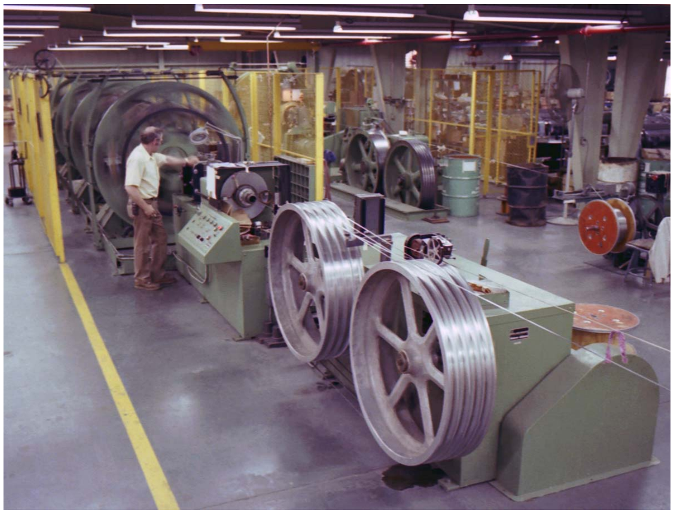

Figure 11.2.4: Manufacturing of Tevatron cable at New England Wire on one of the three large planetary cablers. Photograph courtesy of FNAL.

required large quantities of Tevatron cable. Strand received from Supercon, MCA and IGC was inspected, cleaned and coated with either Stabrite or Ebonol, some after de-plating. Then the strand material was appropriately spooled and fabricated into cable. A final step was to process the cable through a vapor degreaser to remove any oil picked up in cabling and then to apply Kapton tape with a 50% overlap followed by space-wrap of glass tape which had been impregnated with B-stage epoxy. The epoxy was activated by heat at Fermilab after the magnet coils were wound to create a solid superconducting coil package.

One of the complexities of the process was created by the use of the B-stage epoxy which needed to be maintained cold and in low humidity. Spools of glass tape were shipped by Fermilab to New England Wire, stored under refrigeration and later used for insulating the cable. Then a system of refrigerated delivery trucks was created to ship completed spools of cable from New Hampshire to Fermilab. Over 5,000,000 feet of 23-strand cable was manufactured using these methods to create the world's first superconducting magnet accelerator.

11.2.13 Towards a complete description of Nb-Ti

A US DOE program to better understand the development of high J_c in Nb-Ti conductor was initiated under David Larbalestier, who had joined Roger Boom at the University Wisconsin-Madison in 1976. Boom needed the cheapest possible conductor, using the metric of $/kilo-ampere-meter, to make his huge diurnal superconducting magnetic energy storage dreams feasible. Running at 1.8 K using Nb-Ti was central. Larbalestier had been at Rutherford Laboratory in Martin Wilson's group developing the first filamentary Nb_3Sn conductors in collaboration with Jimmy Lee's group at Harwell. When he came to Madison, he got some lengths of Fermilab strand from Bruce Strauss and started to heat treat them according to the published recipes. But the results were quite non-systematic. Over the next 3 or 4 years as the Fermilab and Brookhaven programs proceeded towards large wire production for the Isabelle and Tevatron, there was intense competition in the industry between the remaining US vendors. Larbalestier was very struck by the fact that Airco was approved for Isabelle strand but not for Fermilab strand. Although much more excited about Nb_3Sn developments, he was pushed into Nb-Ti studies both by the need to support the SMES program and by his sheer frustration that logically planned experiments on Nb-Ti produced random outcomes. Finally in 1979-1980, he concluded that the processing of Nb-Ti was sufficiently unpredictable that he wrote a proposal to understand Nb-Ti processing, and finally Dave Sutter in DOE-High Energy Physics decided to fund it. According to Dave the reviews were very mixed. Some people said it was all known anyway, while others said that more understanding would make it impossible to run a commercial business. Sutter imposed one simple condition: hold an annual workshop at which the industry and the magnet builders would come to hear what Larbalestier's group was finding. This review, christened the Nb-Ti Workshop, was first held in July 1983 in Madison. It has evolved into a remarkably successful meeting of the users, fabricators and understanders of conductors, and it now called the Low Temperature/High Field Superconductor Workshop. Over 30 have been held, the latest in Monterey CA in November 2010. At the first workshop, formal presentations were made by UW researchers only; however, it was clear from strong discussion by the attendees that such a researcher-application user-industrial supplier forum was highly appreciated. And indeed, using this workshop-discussion format, there would be a strong contribution from the entire community in future workshops. An important focus of the first workshop was on the fact that US Nb-Ti wire was not competitive with the best R&D wires from Europe and that systematic experiments with US industrial Nb-Ti generally did not produce systematic responses, and that studies of the precipitate structures seen in modern wires were not similar to those reported in the literature. The prospect of doubling the regular US wire production critical current density, J_c, values from ~1600-1800 A/mm² at 5 T in the Isabelle and Tevatron wires to the values over 3500 A/mm², 5 T, 4.2 K, 10^{-14} Ω·m reported by German [58] and Chinese [59] groups attracted both strong applications and industrial interest. Of particular note, the Chinese group at the Baoji Institute for Non-ferrous Metal Research, led by Zhou Lian, had made a wire with J_c at 5 T well above 3000 A/mm². What was decisive was that he gave a length to David Larbalestier at the 1982 Applied Superconductivity Conference and after careful evaluation of all relevant parameters, it became clear that Baoji had indeed made a major J_c breakthrough using their own Nb-Ti ingots and in-house composite wire fabrication. Zhou Lian invited Larbalestier to visit China in 1983. Out of this came the visit of the young Baoji researcher, Li Chengren, to Madison, where he would push J_c to 3680 A/mm² (5 T, 4.2 K) using standard Wah Chang Nb-46.5Ti monofilament strand [60]. What made this possible was the development of an earlier understanding in the period 1980-1982 of the hidden variable that was making systematic experiments on Tevatron and Isabelle wires so unpredictable.

The Tevatron made many huge contributions to the superconductor industry (2011 marks the 29th and final year of the Tevatron's operation). Of crucial importance for this story was the establishment of rigorous specifications for all of the components needed to make multifilamentary composite wires. Starting in about 1978, Larbalestier's first PhD student David Hawksworth, later the leader of the MRI-fabrication company Oxford Magnet Technology, was trying to raise the upper critical field of Nb-Ti by alloying so that better fusion devices could be built. After many studies, an alloy of Nb-43Ti-25Ta was found to have the highest critical field and a special melt was procured from Wah Chang for a fusion magnet coil to be built at General Atomics by John Purcell. But the J_c developed by standard processing was very disappointing, well below the Tevatron values. Examination of the microstructure by graduate student Bill Warnes showed an enormous range of local composition and α-Ti precipitate size that made a hugely non-uniform microstructure.

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[58] Juergen Willbrand and Wolfgang Schlump, "Influence of precipitation density and particle diameter on the current carrying capacity of niobium-titanium superconductors," *Zeitschrift fuer Metallkunde* 66:12 (1975): 714-719

[59] Li Cheng-ren, Wu Xiao-zu, and Zhou Nong, "NbTi superconducting composite with high critical current density," *Magnetics, IEEE Transactions on* 19:3 (1983): 284-287; doi: 10.1109/TMAG.1983.1062365

[60] Li Chengren and D.C Larbalestier, "Development of high critical current densities in niobium 46.5 wt% titanium," *Cryogenics* 27:4 (April 1987): 171-177

This observation was a decisive demonstration for Larbalestier that experiments with bad outcomes are often those with the biggest lessons to teach. When the Tevatron program had fixed the specification of the Nb-Ti alloy, they had fixed the composition, the impurity contents, the grain size and a host of other variables. But they had not fixed the permissible composition variation at the local scale. Because there is a 300 °C difference between the liquidus and solidus of the Nb-Ti alloy, extensive coring is possible in the cast ingots. What the Nb-Ti-Ta alloy showed was local composition variations of 9 at.%, the variation mainly being in the Ti content and on looking at binary Nb-Ti billets, it immediately became clear that these too were inhomogeneous, the local composition varying by as much as ±5 wt.% [61]. It was immediately obvious that systematic studies failed so often because the local Ti content, which controlled the α-Ti precipitation, was not under control. Meetings with Wah Chang were immediately set in place and a long-running collaboration between Bill McDonald at Wah Chang and Peter Lee and David Larbalestier in Wisconsin lead to a 5 year program of more and more homogeneous alloy development. McDonald was immediately able to greatly reduce the coring by turning on electromagnetic stirrers. This first alloy, christened 906 for the last 3 digits of its heat number, was ready for Li Chengren when he came to Madison to continue his high J_c experiments.

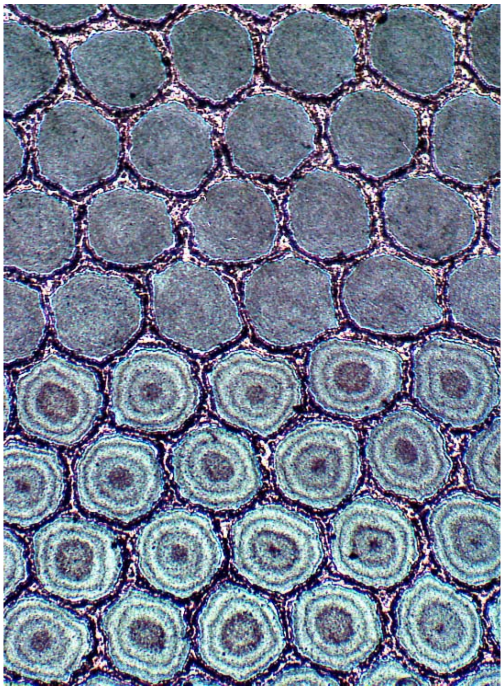
Figure 11.2.5: Highly inhomogeneous tree-ringed Nb-Ti filaments mixed with Homogeneous Nb-Ti in a prototype SSC strand.

Solidified by the DOE-HEP support for the Madison group and all of the strong interactions that were developing in the superconducting wire industry and with the HEP magnet builders, Wah Chang was asked to make a more homogeneous alloy that was free of large Ti-rich regions called freckles, a product that became known as “Hi-Ho”[62].With a tight composition range, a complete description of the optimization of the Nb-Ti microstructure could be reached because systematic experiments became possible[63]. A linear relationship between precipitate volume and critical current density, indicating full summation of the individual vortex-precipitate interactions was soon seen[64]. The extension of a high homoge-

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[61] A. West, W. Warnes, D. Moffat, D. Larbalestier, “Compositional inhomogeneities in Nb-Ti and its alloys”, *Magnetics, IEEE Transactions on* 19:3 (1983): 749-753; doi:10.1109/TMAG.1983.1062305

[62] D. Larbalestier, A. West, W. Starch, W. Warnes, P. Lee, W. McDonald, P. O'Larey, K. Hemachalam, B. Zeitlin, R. Scanlan, C. Taylor, “High critical current densities in industrial scale composites made from high homogeneity Nb 46.5 Ti”, *Magnetics, IEEE Transactions on* 21:2 (1985): 269-272; doi:10.1109/TMAG.1985.1063709

[63] P. J. Lee and D. C. Larbalestier, “Development of nanometer scale structures in composites of Nb-Ti and their effect on the superconducting critical current density”, *Acta Metallurgica* 35:10 (October 1987): 2523-2536; doi: 10.1016/0001-6160(87)90149-0

[64] P. J. Lee, J. C. McKinnell, and D. C. Larbalestier, "Restricted, Novel Heat Treatments for Obtaining High J_c in Nb46.5wt%Ti," in R. P. Reed and F. R. Fickett, eds., "Advances in Cryogenic Engineering (Materials) ", vol. 36, pp. 287-294, New York: Plenum Press, 1990; doi: 10.1007/978-1-4613-9880-6_37

neity alloy supply over a wide range of compositions[65] enabled a unified a view of the impact of composition on precipitate morphology, sensitivity to strain and precipitation rate the explained previous inconsistencies.

By the 6th Workshop on Nb-Ti Superconductors (now jointly organized with Ron Scanlan at Lawrence Berkeley Laboratory) all the US wire manufacturers were able to report critical current densities exceeding 3000 A/mm² (5 T, 4.2 K) in large filament trial billets in preparation for Superconducting Super Collider, which was intended to be the Tevatron's successor, and by the 8th Nb-Ti workshop, held at Asilomar, CA, in March 1988, the technology for obtaining SSC current requirements and beyond had been developed in both 6 and 9 µm diameter filamentary conductors.

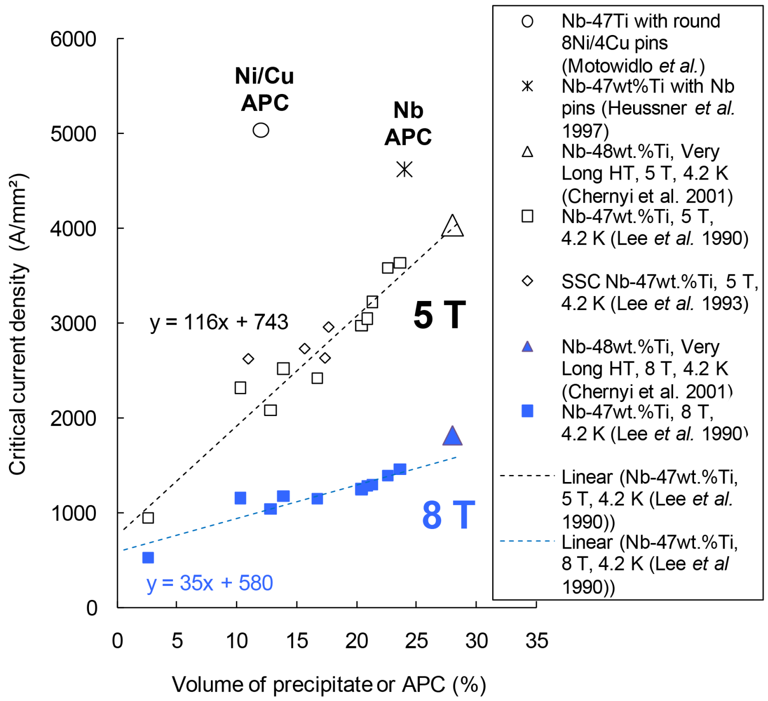

Figure 11.2.6: The critical current density of conventionally heat treated Nb-47wt.%Ti has been shown to be linearly dependent on the volume % of the α-Ti pinning center[64] and Oleg Chernyi and his colleagues extended the 5 T J_c beyond 4000 A/mm² by using 48 wt.% and very long heat treatments to obtain 28 vol.%α-Ti.[67] Using Nb pinning centers [69] the 5 T J_c has been extended to 4600 A/mm² and using ferromagnetic pinning centers (Ni/Cu) the 5 T, 4.2 K J_c has been extended to over 5000 A/m².[70]

In 1993 the SSC was cancelled, but high critical current Nb-Ti had reached maturity and would require little further technical development for the LHC (strand example shown in Figure 11.2.8), where the highest J_c strand had a critical current density at 5 T, 4.2 K of 3194 A/mm².[66] Nevertheless, the performance range of ductile Nb-Ti superconductors has been shown to extend well beyond that used by LHC by increasing the volume of α-Ti or changing the pinning centers completely as shown in Figure 11.2.6; Oleg Chernyi and his colleagues extended the 5 T J_c beyond 4000 A/mm² by using a higher Ti content alloy (48 wt.%) and extremely long heat treatments to obtain 28 vol.% α-Ti[67]. By further alloy modification it seems likely that further improvements could be made. Mechanically introducing pinning centers and multiple re-stacks, "Artificial Pinning Center" (APC) composites can be created that are not restricted in pinning center composition or geometry[68]; using Nb pinning centers the 5 T J_c has been extended to

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[65] P. J. Lee, J.C. McKinnell, and D.C. Larbalestier, "Microstructure control in high Ti NbTi alloys", *Magnetics, IEEE Transactions on* 25:2 (1989): 1918-1921; doi:10.1109/20.92681

[66] Private communication from Thierry Boutboul, LHC, September 2006.

[67] O. V. Chernyi *et al.*, "The Microstructure and Critical Current Density of Nb-48 wt.%Ti Superconductor with Very High Alpha-Ti Precipitate Volume and Very High Critical Current", Advances in Cryogenic Engineering, 48 (B): 883-890, 2002; doi:10.1063/1.1472628

[68] G. L. Dorofejev, E. Y. Klimenko and S. V. Frolov, E. V. Nikulenkov, E. I. Plashkin, N. I. Salunin, V. Ya Filkin,

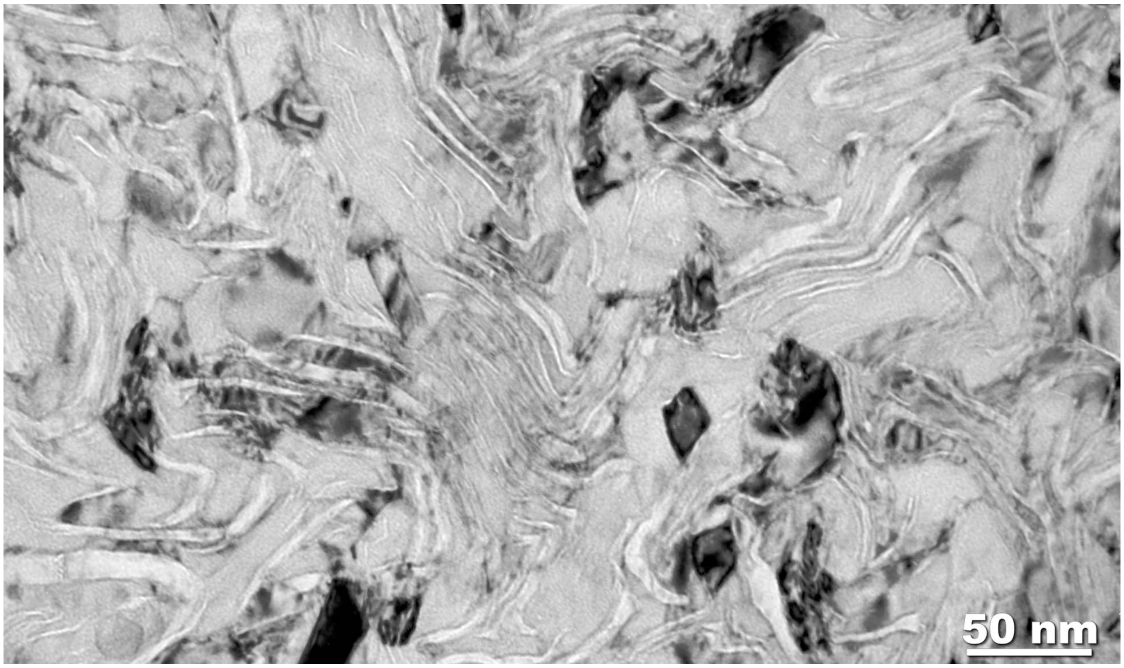

Figure 11.2.7: TEM image of the microstructure (transverse cross-section) of the first 3700 A/mm² (5 T, 4.2 K) multifilamentary strand from a US manufacturer (OST). This previously unpublished image taken on September 5th 1986, shows the dense array of folded α-Ti ribbons (lighter contrast) that create the strong vortex pinning.

4600 A/mm² with only 24 vol.% of pinning centers[69] and by using ferromagnetic pinning centers (Ni/Cu) the 5 T, 4.2 K J_c has been extended to over 5000 A/mm².[70]

11.2.14 Nb-Ti as a commodity

"There is no base business for the applied superconductivity industry without MRI"[71]. Although much of the push for advancing the technology of Nb-Ti superconductors came from the needs of the synchrotron and accelerator communities, the business of Nb-Ti is dominated by wires for MRI imaging. What can be said is that the initial intense developments that took place over the period from 1965 to about 1985 were driven technically by the need to make the highest possible J_c values for fine filament (5 – 10 µm) conductors that could be made into Rutherford cables. Technical, rather than cost considerations were dominant. The success of the wire industry was built, however, on the emerging market for MRI conductors – much simpler, largely monolithic ones, that needed to be made very reliably at the lowest possible cost. The wonderful synergy that developed between the technical understandings emerging from the Nb-Ti workshop – the optimum ribbon nanostructure, and the demonstration that Nb-47Ti was indeed the optimum composition, supported the largely hidden technology developments of

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Current carrying capacity of superconductors with artificial pinning centers", Proceedings of the 9th International Conference on Magnet Technology, Ed. by C. Marinucci and P. Weymuth (pub. Swiss Institute for Nuclear Research, Villigen, Switzerland), pp. 564-566, 1985. ISBN: 3907998006

[69] R. W. Heussner, J. D. Marquardt, P. J. Lee, and D. C. Larbalestier "Increased critical current density in Nb–Ti wires having Nb artificial pinning centers", *Applied Physics Letters* 70:7 (1997): 901; doi:10.1063/1.118238

[70] L. R. Motowidlo, M. K. Rudziak, and T. Wong, "The pinning strength and upper critical fields of magnetic and nonmagnetic artificial pinning centers in Nb47w/oTi wires", *Applied Superconductivity, IEEE Transactions on* 13:2 (2003): 3351-3354; doi:10.1109/TASC.2003.812314

[71] Seung Hong, Oxford Superconducting Technologies, private communication for this article.

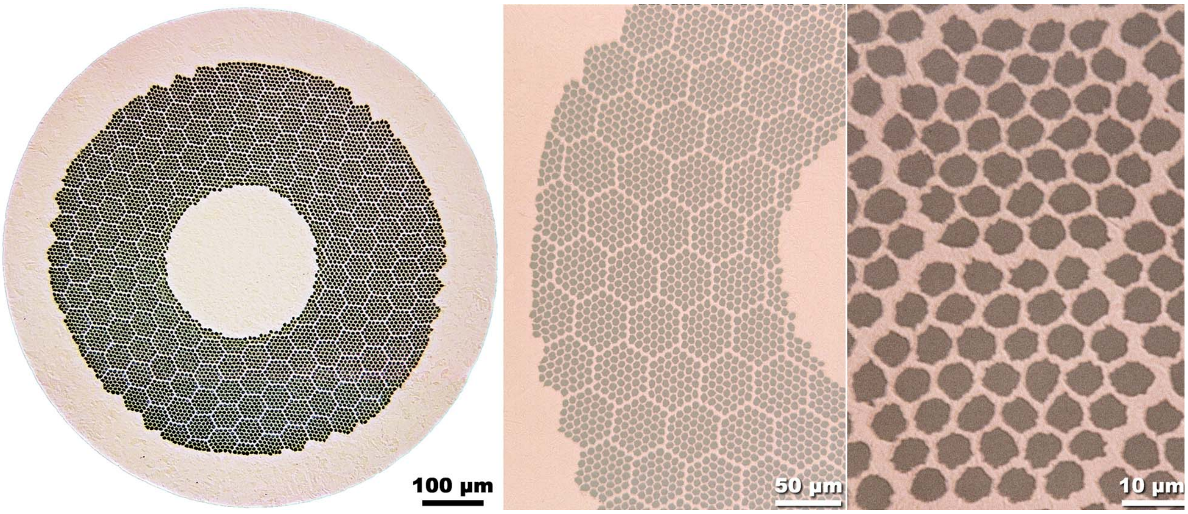

Figure 11.2.8: Outokumpu AS (now Luvata) LHC Inner strand cross-section showing double stacked filament array. In the LHC an operating temperature of 1.9 K allows ultimate central fields of up to 9 T in the dipoles.

MRI. Nb-Ti is the tonnage conductor of superconducting applications, because it is thoroughly understood, manufacturable and the workhorse for more than 90% of all superconducting magnets. But as the subsequent chapters show, Nb_3Sn, MgB_2, $Bi_2Sr_2CaCu_2O_x$, $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$, and now $YBa_2Cu_3O_{7-\delta}$ are all vying to take part or all of its place. History will report in another 10-20 years whether or not the huge industrial and scientific base of Nb-Ti could outweigh the advantages of transition temperatures more than 10 times higher than the 9 K of Nb-Ti.

Further Reading

1. In 1986 the Applied Superconductivity Conference celebrated the 75th Anniversary of the discovery of superconductivity with a symposium on the history of superconductivity. The symposium is published in full in IEEE Trans. Magn., 23, pp. 354-415, 1986

2. The most detailed accound of the events surrounding the discovery of Type II Superconductivity can be found in A. G. Shepelev's the "The Discovery of Type II Superconductors (Shubnikov Phase)" in "Superconductor," Edited by: Adir Moyses Luiz, ISBN 978-953-307-107-7, Publisher: Sciyo, Publication date: August 2010
   http://www.intechopen.com/books/show/title/superconductor

3. The development of the Nb-Ti nanostructure is covered in more detail here:
   "Conductor Processing of Low-T_c materials: The Alloy Nb-Ti," L. D. Cooley, P. J. Lee, and D. C. Larbalestier, in "Handbook of Superconducting Materials", ed. David A Cardwell and David S Ginley (Institute of Physics Publishing, Ltd, Bristol 2003), Volume I: Superconductivity, Materials, and Processes, chapter B3.3.2, pp 603-637

4. And here:
   P. J. Lee, "Abridged metallurgy of ductile alloy superconductors," in J. G. Webster, ed., "Wiley Encyclopedia of Electrical and Electronics Engineering, Vol. 21," New York: Wiley, pp. 75-87, 1999