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Improved Niobium 47 Weight % Titanium Composition by Iron Addition

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The effect of iron addition on the ductility and superconducting properties of Niobium Titanium alloys has been explored. The conclusion of this study is that iron up to 2000 parts per million by weight is not deleterious to the fabricability of multifilamentary composites. The hardening rate of the iron rich alloy as filaments in a copper matrix was observed to be the same as for low iron versions of the same nominal composition. The development of the pinning microstructure was monitored and compared to the critical current properties of the wire. The results for multifilamentary wires are compared for heats with and without high iron contents. The results were reproduced in the production environment from a full-scale ingot. The critical current density for the iron-enriched material is elevated relative to the low iron material at high fields. The beneficial influence of iron on the superconducting properties is discussed in light of the microstructure. A designed experiment was conducted to explore the range over which the chemistry can be varied without affecting the behavior. The goal is to broaden the range of iron allowed in Niobium Titanium alloys for superconducting applications.

Keywords: niobium, titanium, iron, hardening rate, fabricability, multifilamentary, superconductivity, microstructure, flux pinning, critical current density, specification, designed experiment, upper critical field, composite

I. Introduction

The specification of raw materials for superconducting applications is a subject that is given a lot of attention, particularly when very large devices are being designed, such as the Superconducting Supercollider (SSC) or the Large Hadron Collider (LHC). Wire manufacturers often apply very stringent specifications for the alloy manufacture. Just as often, the technical justification for tight chemical specification is missing or forgotten. Tight chemical specifications can greatly increase the difficulty and cost of manufacturing the alloy. Current specifications for the niobium titanium alloy limit iron content to 200 parts per million by weight or less. The goal of this study was to provide a technical justification for a broadening of the limit to higher iron levels.

The recent changes in the world political environment have greatly impacted the titanium industry. The result has been that several high cost manufacturers of titanium sponge have ceased production. These producers were primarily those using sodium to reduce their titanium. That process was particularly conducive to making low iron titanium. Low iron titanium is less available and more costly than in the past. In the production of niobium titanium alloys, the iron content of the alloy is largely determined by the titanium since the niobium is electron beam refined.

The conventional wisdom has been that iron has a significant impact on the hardening rate of niobium titanium alloys. Teledyne Wah Chang has consistently provided alloy with iron levels less than 125 parts per million by weight (ppm) for many years. Many titanium alloys, however, have iron as an alloy ingredient to improve the fabricability and strength. For conventional titanium alloys, oxygen, nitrogen and carbon are the three most significant hardening agents and iron is considered weak.[1] Even so, the SSC and the Relativistic Heavy Ion Collider (RHIC) programs both specified the niobium titanium alloy with less than 200 ppm iron.[2,3] (Table I.)

The trade-off for low iron in magnesium reduced titanium sponge is elevated nickel and chrome levels due to a switch in liner materials from mild steel to superalloys to improve reaction vessel life. It is easier to find low nickel, low chrome and high iron than it is to find low iron, low nickel and low chrome.

There is limited information in the literature on iron solubility in NbTi. Kirshenina et al investigated the effect of 0.7

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atomic % iron in Ti 22 at.% Nb 2 at.% Zr[4]. The critical temperature (T_c) of the alloy was raised to 10 K from 6 K with the iron. The alloy showed two transitions and was not fully superconducting until 6 K. It is possible that the iron caused precipitation of TiFe leaving a niobium enriched region closer to Ti 35 at%Nb (Nb 47 wt.%Ti) which would be superconducting at 10 K. Nishimura and Zwicker investigated the effect of iron and niobium on the superconducting properties of titanium and concluded that alloys with high niobium and low iron (less than 3 at.%) showed an increase in T_c when annealed in the range 400 to 500°C[5]. Alloys with low niobium and high iron showed a decrease in T_c. Neither of these studies addressed the solubility of iron in Nb-47 Ti nor did they investigate the fabricability or development of critical current density.

Dr. Ann West and Dr. David Larbalestier once did a study of the effect of Iron on critical transition temperature of Nb-47 Ti but the result to was never published[6]. Iron levels above 1.5 wt.% were needed to see a significant decrease in T_c. The work demonstrated that the iron remained in solution and that enough iron slowly caused the T_c to drop.

II. Experimental

1. The Plan

To meet the goal of providing technical justification for a broadened specification and widening the availability of titanium sponge for alloy production, a plan was created to determine the potential for improvement, involve customers in the process and explore the practical limits to a new specification. Initially, small consumable electrode arc cast ingots (200 mm diameter, less than 35 kg) with increased iron level were produced using Pratt and Whitney 1201E grade sponge as well as controls with normal chemistry and these were processed into multifilamentary wire. Samples were taken during processing and the mechanical and superconducting properties tested for comparison.

The success of this work led to the casting of a full size production ingot using the same input materials (greater than 2000 kg) and distribution of billets to various wire manufacturers to be processed into normal production style multifilamentary billets. At the same time, a designed experiment (full factorial) was initiated to explore, independently, the effect of oxygen, titanium and iron over a broader range on the hardening rate behavior of binary alloys using buttons melted in a tungsten electrode, inert gas, arc furnace.

2. Hardness Testing

Samples were taken after casting and extrusion and at various steps in the wire drawing process. The samples were mounted and polished using normal procedures[7]. Hardness values for the button samples were taken using a Wilson Tukon Hardness tester and the average of three positions (edge, mid radius and center) reported. Data for the monofilaments made from the buttons were obtained at large sizes using the Tukon and at smaller sizes using a LECO M400 D3. Low hardness values were dismissed when porosity was simultaneously observed and these were not included in the averages.

The data for the multifilamentary composites was taken using a LECO M400 A tester and the average of ten separate filaments reported.

Table I: Chemical Analysis of Nb 47 Ti Alloy

Element	Normal ≈2000kg	HF-1 35 kg	HF-2 35 kg	HF-3 2000 kg	SSC-MAG 4000
Titanium	46.3	46.3	46.5	46.2	47 ± 1 wt%
Niobium	Balance	Balance	Balance	Balance	53 ± 1 wt%
Oxygen	627	470	503	483	1000 ppm Max
Hydrogen	< 3	< 3	< 3	4	35 ppm Max
Carbon	60	80	100	46	200 ppm Max
Iron	61	590 *	535 *	517 *	200 ppm Max
Tantalum	620	700	700	665	2500 ppm Max
Nitrogen	40	30	30	30	150 ppm Max
Nickel	< 25	35	25	<25	100 ppm Max
Silicon	< 50	40	35	<25	100 ppm Max
Copper	< 10	45	40	<10	100 ppm Max
Aluminum	< 20	80	65	80	100 ppm Max
Chromium	< 25	70 *	50	51	60 ppm Max
Hardness (Cast)	146.5	141	145	145	170 average
Hardness (Extruded)	148	151	149	142	170 product

* means analysis is above the SSC specification

3. Analytical Testing

All the chemical testing was performed in the analytical laboratory at TWC. The button melts were tested for tungsten and found to be contaminated. Between melts, the buttons were radiographed and inclusions noted were removed by drilling. While some tungsten is believed to be in solution, the analytical results do not distinguish between dissolved and included material. The large variability in the tungsten level is a result of the number of inclusions sampled. Metallic analyses were run on turnings and interstitial analyses were run on solid pieces.

4. Critical Current Testing

The critical current testing reported here for the initial test billets was done in the cryogenic test lab at TWC. The method is described elsewhere.[8] All tests were run at

4.2 Kelvin. The production wires were tested in the manufacturer's laboratories using their standard practices, also at 4.2 Kelvin.

5. Multifilamentary Billets

The initial test of the 200 mm diameter arc cast ingots were made by constructing 54 filament billets. The ingots were machined to extrusion diameter, extruded and drawn to rod. The rod was air annealed and water quenched after a total strain from casting of 3.1. The rods were drawn to size, a further strain of 4.0, and then inserted in hexagonal shaped, round inner diameter pure copper tubes. These were stacked with a filler rod in a copper can and extruded to multifilamentary rod. The composites received four heat treatments, spaced at strain intervals of 1.0, of 40 hours at 375 °C each. Samples were taken before and after each heat treatment for hardness testing and at strains of 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0 after the fourth and final heat treatment.

The production billets were constructed according to standard designs of the wire manufacturers. IGC constructed a 300 filament, nominal 4:1 copper to superconductor ratio composite and OST constructed a production size composite with a relatively low copper to superconductor ratio. Details of the processing are not given here but they were unchanged from the normal processing of low iron alloy within each shop.

The heat treatment and cold working history of the alloy during processing at TWC is specific to each customer and is different between the two cases here. The multi-filament billet processing at the manufacturers are also significantly different. The history of the high iron trial material was not different, however, from normal low iron material at either TWC or the customer according to their specific processes.

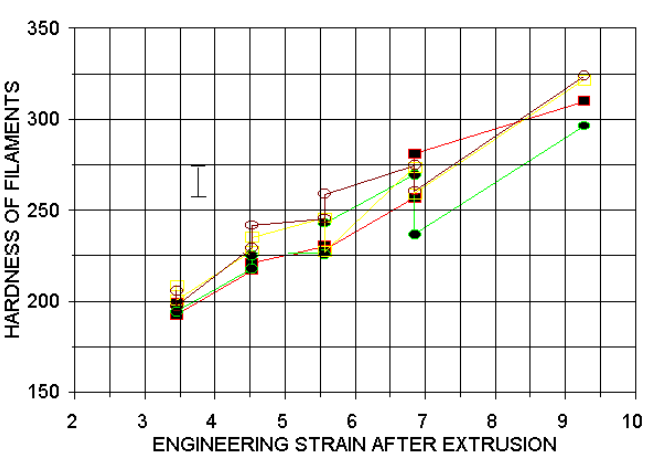

Figure 1 Hardness of Nb47Ti filaments (average of ten impressions) in a 54 filament composite as a function of strain after extrusion. Samples with iron about 600 ppm (□HF-1;❍ HF-2) are compared to samples with iron less than 100 ppm (■ Standard; ●HiHo).

6. Monofilament Billets

The designed experiment billets were cast as buttons and then subsequently drop cast into 1.9 cm diameter ingots of approximately 0.4 kg. The castings were sampled for chemistry and machined to 1.3 cm diameter for drawing. The machined ingots were inserted in pure copper tubes and swaged to 0.9 cm. Then the monofilaments were drawn to 0.05 cm taking samples at strain intervals of 0.5 between 1.5 and 7.5. The strain was calculated from the wire diameter and the non-copper fraction measured using image analysis.

7. Electron Microscopy

Scanning electron microscopy (SEM) was performed on the fourth heat treatment samples of the small scale multifilament billets using the technique of Fasse[9] on a JOEL 35C SEM. Samples were then sliced from these specimens for transmission electron microscopy (TEM). Samples were prepared for TEM from both the first heat treatment and the fourth heat treatment by jet electro-polishing with a solution of 2 vol.% Hf_{aq}, 5 vol.% $H_2S0_{4\ aq}$, and 93 vol.% methanol at -40 °C using a rapidly cycling polishing current density.[10] TEM was performed on a JOEL 200 CX TEM/STEM at 200 KV. The images were processed on a Megavision 1024XM system using enhanced atomic number contrast composite images created by combining multiple images taken at small tilt variations about the β-110 drawing axis [11]. Image analysis to determine the precipitate volume was performed using SigmaScan Pro software by Jandel.

III. Results

1. Multifilamentary Billets - Research Scale

The initial 200 mm diameter ingots were designated HF-1

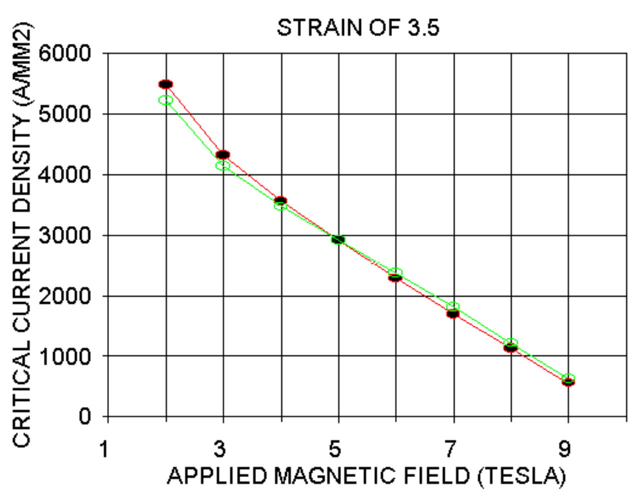

Figure 2 Critical current density of 54 filament composites. The HF-2 sample (❍) with iron about 600 ppm is compared to the high homogeneity sample (●) with iron less than 100 ppm. The current density at 5 Tesla was highest for both wires at a strain of 3.5 after the last heat treatment.

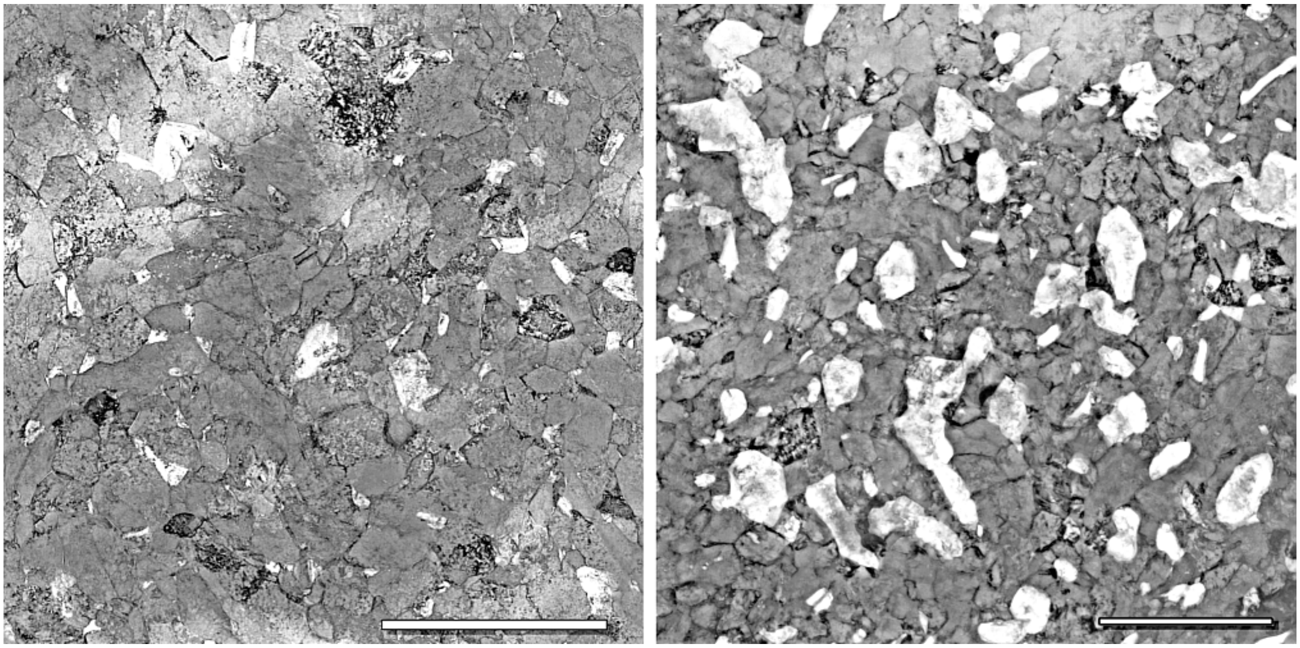

Figure 3 Transmission electron micrographs of the low iron, high homogeneity (< 100 ppm), 54 filament wire after the first heat treatment (left) and after the fourth heat treatment (right). The scale marker for the left micrograph is one micron, and the marker for the right is 500 nm.

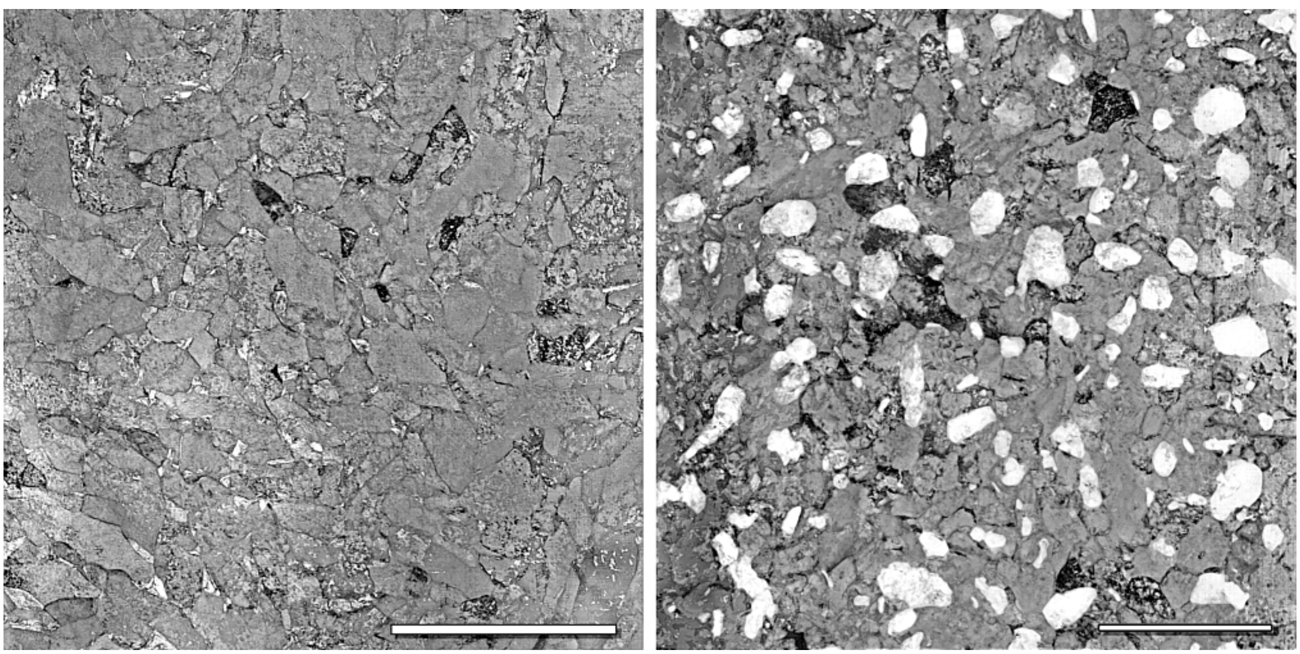

Figure 4 Transmission electron micrographs of the high iron, HF-2 (about 600 ppm), 54 filament wire after the first heat treatment (left) and after the fourth heat treatment (right). The scale marker for the left micrograph is one micron, and the marker for the right is 500 nm.

and HF-2. The chemistry for these ingots are listed in Table I along with the chemistry for a normal production heat of Nb47Ti and the SSC specification for alloy. The copper content of the 35 kg ingots were slightly elevated relative to normal production experience and this is an artifact of the small ingot process.

There were no significant differences noted in the processing of the multifilament wires that could be contributed to the iron content. Samples were taken after extrusion, before and after all four heat treatments of 40 hours at 340 °C and at a strain of 2.5 after the last heat treatment. The hardness data for two billets with normal low iron material, one standard and one a high homogeneity grade, are plotted along with the billets made from HF-1 and HF-2 in figure 1. Within the statistical variation of the numbers, there is no observable difference on the hardening characteristics with iron as the main variable.

Critical current measurements were made over the applied field range of 2 to 9 Tesla (figure 2). The critical current density was monitored as a function of strain to look for the peak at various fields. The current density at 5 Tesla peaked for both the high and low iron materials at a strain of 3.5 after the last heat treatment. This was expected for the wire design and thermo-mechanical processing. It was noted that the critical current density began to develop sooner (higher critical current density at lower strains) in the high iron material and fell off more steeply after the peak than the low iron material. The wires were checked for extrinsic effects and none were found. The high iron material performed significantly better at higher fields. Normalized $J \times B$ curves indicated that the extrapolated H_{c2} values were increased for the high iron heats by as much as a quarter Tesla. Whether this is a real change in H_{c2}, a change in the irreversible magnetization field or just random variability is not currently known. All the high iron cases, research or production, have slightly higher current density above 5 Tesla than low iron material, however.

Figures 3 and 4 show the TEM micrographs after the first and fourth heat treatments. The high iron material produces a finer, more uniform distribution of precipitates, yet the average size is practically the same; both versions yield log normal size distributions [12]. What is interesting is that the high iron billets have less total volume alpha titanium precipitated, 17.4% versus 20.9 %for the low iron yet the current densities are very similar. The SEM back-scattered images were striking in the difference of how much alpha had precipitated but failed to produce quantitative results.

2. Multifilamentary Billets - Production

The chemistry for the full-scale production ingot is listed in Table I as HF-3. HF-3 was constructed of the same lots of material as HF-1 and HF-2. The only variation from the normal processing of a high homogeneity Nb47Ti heat was the source of the titanium sponge and its chemistry. As customers were enlisted to participate in evaluating the material, sections of the ingot were processed with the standard methods to the customer's normal production billet dimensions. The minimum amount received for evaluation by a customer was 200 kg. Neither IGC nor OST reported any abnormal wire processing problems and both produced commercial quantities of wire with the material. The critical current performances for the production wires are plotted in figures 5 and 6. The processing of the alloy by TWC, the construction of the multifilament billets by the manufacturers and the wire designs chosen are all significantly different. In both cases, the high iron material appears to perform better than the normal process data, but these data are still within the normal process variations. The IGC wire had a high copper to superconductor ratio and index ("n") values were obtained for the tests and a J_c criteria of 10^{-12}Ωcm was used. The "n" values were as good or better than standard production. The OST J_c criteria was 0.1 μV/cm.

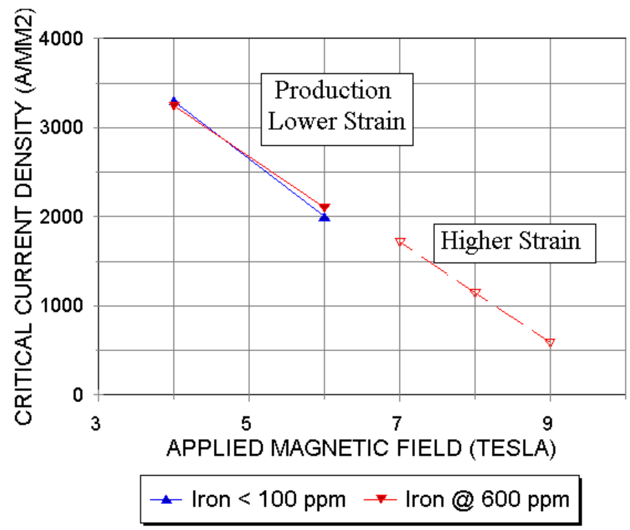

Figure 6 Comparison of high iron material in production with normal, low iron material. Data taken at 4.2 K and 10^{-12} Ωcm. Wire data courtesy of Intermagnetics General.

IGC performed SEM tests on their heat treated wire and observed the same finer pattern in the production material as was observed in the 200 mm ingots relative to normal production.

3. Monofilaments - Designed Experiment

The success of the initial ingots raised the question as to how much iron could be added and how did it affect the mechanical properties as the oxygen and titanium levels varied. A full factorial experiment was designed in which the titanium was varied from 44 to 55 weight percent, oxygen from 500 to 1000 ppm and the iron from 200 to 1200

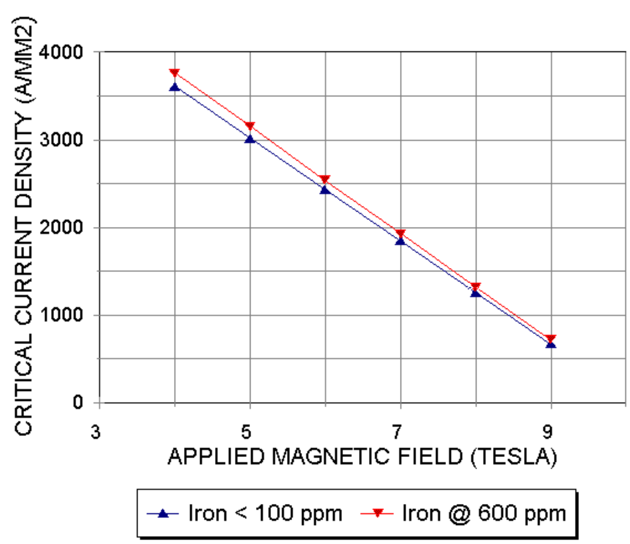

Figure 5 Comparison of high iron material in production with normal, low iron material. Data taken at 4.2 K and 0.1 μV/cm. Wire data courtesy of Oxford Superconducting Technology.

ppm. A total of 20 trials were run: 8 pairs, 1 triplet and 1 control. The as cast chemistry hardness was measured and then, the buttons were drop cast into a cylindrical shape. The chemistries for the buttons are listed in Table II.

The small castings were machined, inserted in copper tubes, swaged and then drawn without heat treatment. The as cast structure did not remain circular in cross section within the copper sheath. A representative plot is given in figure 7 and shows a peak in hardness between an initial drawing strain of 0.5 and 2.0. All samples exhibited this peak to some extent. It was more pronounced with higher oxygen and titanium levels. The data between strains of 1.75 and 7.25 were fit to a linear curve to smooth the data. Figure 8 (A - D) show the effect of chemistry on both the hardness and the hardening rate as oxygen, iron and titanium vary over the ranges studied.

Both the hardness slope (rate) and the zero strain intercept (Initial Hardness) could be fit to the chemistry with the following expressions:

$$\text{Rate} = 18.60 - \text{Oxygen}\,(0.0011) - \text{Iron}\,(0.0013) - \text{Titanium}\,(0.2295) - \text{Tungsten}\,(0.0003)$$

and

$$\text{Initial Hardness} = 96.03 + \text{Oxygen}\,(0.0211) + \text{Iron}\,(0.0089) + \text{Titanium}\,(0.7669) + \text{Tungsten}\,(0.0005)$$

where $\text{Hardness}_{(\epsilon = 1.75 - 7.25)} = \text{Initial Hardness} + \text{Rate} \times \text{Strain}$

As can be seen from these relations, the iron, relative to the oxygen, plays a different role in the rate than it does in the initial hardness. The Titanium component plays the dominant role in both expressions. As the oxygen level increases the effect of the iron changes. At low oxygen levels, the iron increases the overall hardness. At high oxygen levels, the iron decreases the overall hardness.

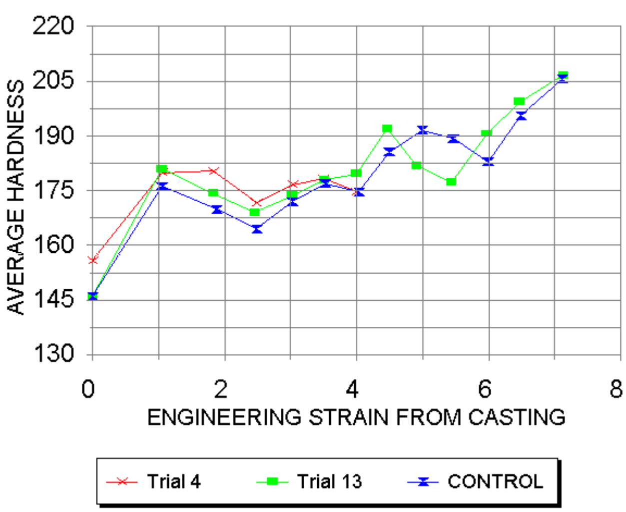

Figure 7 Hardness data (average of three impressions) for monofilament data taken from trials 4 and 13 compared to the control sample. Trials 4 and 13 had the same aim points for chemistry.

The next step is to monitor the hardening rate as the samples are heat treated and processed.

IV. Discussion

The most fascinating part of this program is that the critical current development in the Nb-47Ti alloy deviates from the accepted and well established relationship between volume percent alpha-Ti precipitate and current density [13]. Perhaps this is an artifact of the older data, which is almost exclusively produced with materials having iron contents below 125 ppm. This work demonstrates that iron influences the size distribution without changing the middle of the distribution. The difference between the high and low iron is on the extremes of the distribution and since the critical current density will be dependent on the size [14] as well the total number of right sized precipitates, the missing precipitation in the high iron material does not harm the critical current density. The much finer precipitation during the first heat treatment explains the earlier critical current development in the high iron billets, since the smallest precipitates will be right sized at lower drawing strain. The sharper peak in critical current density with strain also is a result of not having the larger precipitates available.

The balancing effect the iron has on the hardening rate relative to oxygen may be due to local variations in the oxygen content. When iron has a significant presence, greater than 200 ppm, the local variation in oxygen content will be less important and a more uniform precipitation will occur. IGC reported more consistent results between billets than they normally see. This could be a macroscopic reflection of what the TEM and hardness data indicate.

The impetus for this work was both economic and technical as a result of changes in the titanium industry. Though iron levels in titanium sponge lots vary significantly from producers, iron can be alloyed in. It can not be alloyed out. The refractory metals niobium and titanium both have a high affinity for oxygen making control of oxygen pick up and uniformity difficult in the consolidation process. Increasing the iron content by sponge chemistry and alloying could be beneficial to production control and variability.

So far, all the multifilament wire made with high iron levels have been processed to relatively large filament sizes. The initial 54 filament billets using HF-1 and HF-2 had filament diameters about 32 microns at the peak strain of 3.5. Both the production wires had filaments sizes of nominally 50 microns.

We propose that the iron limit for Nb47Ti alloys be increased from 200 ppm to 600 ppm, at least for applications with filament diameters larger than 35 microns, since it does not have any deleterious impact on critical current density or fabricability. Controlling the iron content could be a

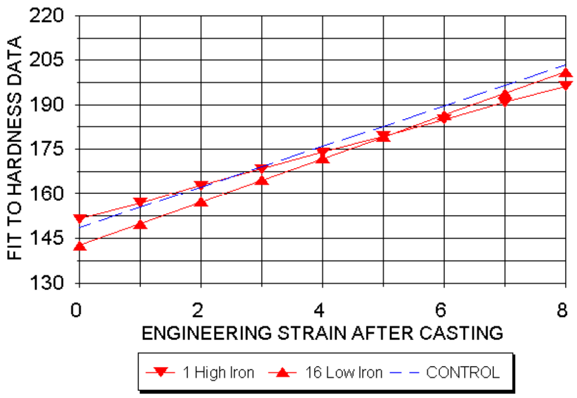

Figure 8 A Low oxygen, low titanium data sets. Actual data has been smoothed by linear regression to show trend. High iron aim was 1000 ppm, low iron aim was 200 ppm and control iron was 83 ppm.

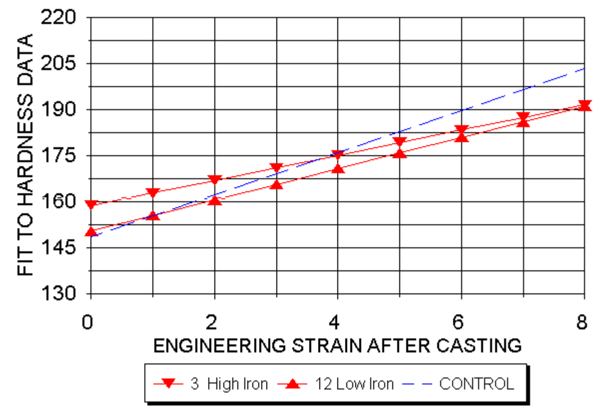

Figure 8 B Low oxygen, high titanium data sets. Actual data has been smoothed by linear regression to show trend. High iron aim was 1000 ppm, low iron aim was 200 ppm and control iron was 83 ppm.

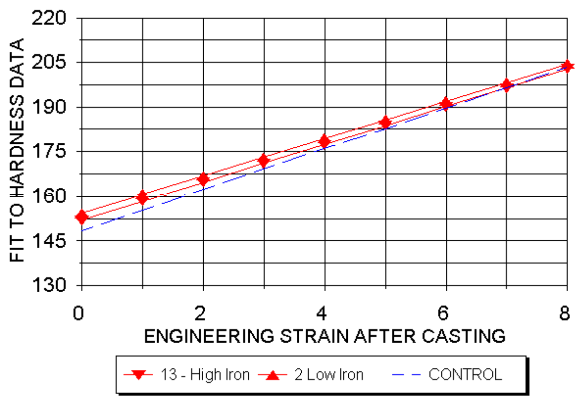

Figure 8 C High oxygen, low titanium data sets. Actual data has been smoothed by linear regression to show trend. High iron aim was 1000 ppm, low iron aim was 200 ppm and control iron was 83 ppm.

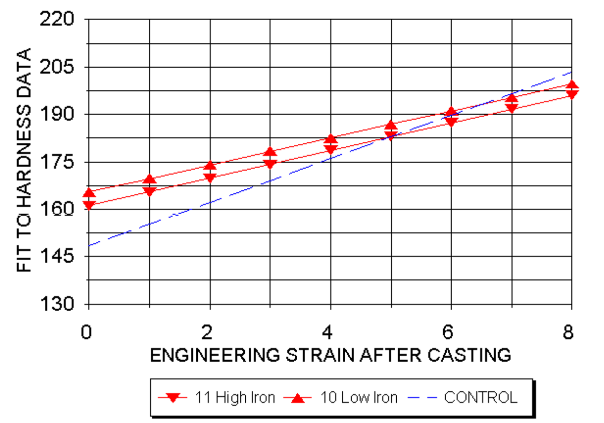

Figure 8 D High oxygen, high titanium data sets. Actual data has been smoothed by linear regression to show trend. High iron aim was 1000 ppm, low iron aim was 200 ppm and control iron was 83 ppm.

Table II. Chemistry Aim/Actual for the Designed Experiment

	AIM	ACTUAL	AIM	ACTUAL	AIM	ACTUAL	AIM	ACTUAL
TRIAL PAIRS	PPM	OXYGEN	PPM	IRON	WT%	TITANIUM	PPM	TUNGSTEN
1 , 6	400	500 , 500	1000	1100, 760	44	44.2 , 43.9	<50	2600 , 220
2 , 14	800	900 , 730	200	330 , 260	44	47.3 , 44.1	<50	160 , 570
3 , 19	400	530 , 510	1000	1200 , 1000	55	53.0 , 52.9	<50	440 , 1400
4 , 13	800	860 , 620	1000	1000 , 1000	44	43.6 , 44.1	<50	540 , 400
5 , 17 , 18	600	680 , 510 , 740	600	690 , 640 , 760	48	48.1 , 48.2 , 49.3	<50	1200 , 930 , 110
7 , 12	400	610 , 500	200	220 , 220	55	53.7 , 54.2	<50	650 , 690
8 , 16	400	480 , 470	200	340 , 230	44	45.8 , 44.9	<50	720 , 540
9 , 10	800	910 , 1190	200	310 , 260	55	50.7 , 54.4	<50	3500 , 540
11, 15	800	790, 800	1000	890 , 1200	55	52.6 , 52.8	<50	360 , 980
20	600	760	<100	83	46.5	46.3	<50	580

Trial 20 was button melted from a slice of a production ingot.

way to improve the achievable current density if the thermo-mechanical processing is optimized when iron is considered. After fine filament processing demonstrates the same behavior, the specification could be increased for applications down to five microns.

Work is in progress to determine the effect of iron on the precipitation and hardness of NbTi alloys with thermo-mechanical processing.

V. Conclusions

- Iron up to 600 ppm by weight in niobium 47-weight % titanium does not impact the fabrication or critical current development when processed identically to the same alloy with less than 100 ppm iron. The specification limit should be increased to 600 ppm.
- The presence of Iron at levels above 200 ppm moderates the effect of oxygen variation and encourages a more uniform precipitation.
- Higher titanium alloys should benefit from iron addition as well.
- The effect of heat treatment needs to be studied for other chemistries (high and low titanium).

Acknowledgments

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