The upper critical field of filamentary Nb$_3$Sn conductors

A. Godeke

Applied Superconductivity Center, University of Wisconsin, 1500 Eng. Drive, Madison, WI 53706, USA

M.C. Jewell, C.M. Fischer, A.A. Squitieri, P.J. Lee and D.C. Larbalestier

Applied Superconductivity Center, University of Wisconsin, 1500 Eng. Drive, Madison, WI 53706, USA

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We have examined the upper critical field of a large and representative set of present multi-filamentary Nb$_3$Sn wires and one bulk sample over a temperature range from 1.4 K up to the zero field critical temperature. Since all present wires use a solid-state diffusion reaction to form the A15 layers, inhomogeneities with respect to Sn content are inevitable, in contrast to some previously studied homogeneous samples. Our study emphasizes the effects that these inevitable inhomogeneities have on the field-temperature phase boundary. The property inhomogeneities are extracted from field-dependent resistive transitions which we find broaden with increasing inhomogeneity. The upper 90-99% of the transitions clearly separates alloyed and binary wires but a pure, Cu-free binary bulk sample also exhibits a zero temperature critical field that is comparable to the ternary wires. The highest $\mu_0H_{c2}$ detected in the ternary wires are remarkably constant: The highest zero temperature upper critical fields and zero field critical temperatures fall within 29.5 ± 0.3 T and 17.8 ± 0.3 K respectively, independent of the wire layout. The complete field-temperature phase boundary can be described very well with the relatively simple Maki-DeGennes model using a two parameter fit, independent of composition, strain state, sample layout or applied critical state criterion.

I. INTRODUCTION

The upper critical field ($\mu_0H_{c2}$) of Nb$_3$Sn is strongly dependent on A15 composition $^{1}$. Although data covering the complete field-temperature ($H$-$T$) phase boundary are available for one set of pure Nb-Sn thin films $^{1,2}$ and a few single- and poly-crystals $^{3,4}$, data on practical, inhomogeneous conductors are sparse. Wires may differ substantially in $H$-$T$ behavior compared to homogeneous films and crystals due to inherent inhomogeneities, arising from their manufacture. They exhibit Sn gradients resulting from the solid state diffusion process used for the formation of the A15 layers, and strain gradients caused by lattice imperfections and thermal contraction differences between the composite materials. The Sn gradients originate from the broad composition range of the A15 phase ($\sim 18$ to $\sim 25$ at. % Sn$^5$). The existence of these inhomogeneities as well as their potential influence on the superconducting properties have been recognized for a long time $^{6-13}$, as is the presence of a scaling field for the critical current which lies substantially below $\mu_0H_{c2}$. This scaling field is often referred to as $B_c^*$ but in fact it is an irreversibility field ($\mu_0H^*$) defined by a pinning force or a Kramer function extrapolation $^{14}$ ($\mu_0H_{c2}$). However, to our knowledge no systematic analysis over the complete $H$-$T$ range of the influence of inhomogeneities on $\mu_0H_{c2}(T)$ and $\mu_0H^*(T)$ is available in the literature. Moreover, the critical parameters are often defined at an arbitrary criterion of e.g. 50 % or 90 % of the superconducting to normal transition and the behavior at practically important lower temperatures and higher fields is mostly extrapolated from the slope at $T_c(0)$. In our analysis we concentrate on the entire transition widths in an attempt to extract the range of property distributions that are present in wires and measure these over the full relevant $H$-$T$ range.

For wires, $\mu_0H_{c2}(T)$ data are available in limited fields $^{15-20}$ while a few publications report single high field points at liquid helium temperature $^{17,18,21-23}$. Extrapolation of data measured up to 22 T indicates a zero temperature upper critical field ($\mu_0H_{c2}(0)$) in thin films ranging from 26 to 29 T, depending on resistivity $^{1,2}$. Single- and poly-crystal data, measured over the complete field range, indicate either $\mu_0H_{c2}(0) \approx 24.5$ T for the tetragonal phase or 29 T for the cubic phase $^{3,4}$. The limited amount of wire $\mu_0H_{c2}(0)$ data, mostly extrapolated from 4.2 K, ranges from about 20 T up to $>30$ T $^{15,18,21-23}$. This large spread causes confusion as to what value is to be regarded as realistic in multi-filamentary conductors and was a prime motivation for us to make a more systematic study of the influence of the inhomogeneities that are present in wires. We believe that the broad range of performance boundaries for wires in the literature is mostly a side effect of property distributions caused by these inhomogeneities, combined with the use of either arbitrary or variable criteria for $\mu_0H_{c2}$.

The general consensus is that bronze-route wires are strongly inhomogeneous with respect to Sn content and that the gradients are on the order of 1-5 at. % Sn/μm$^{8,11}$. Analysis of inhomogeneities in bronze-route wires are, however, complicated by their very small A15 layer thickness on the order of 1-2 μm, imperfect circular symmetry and the fact that the higher $T_c$ A15 layers surround the lower $T_c$ regions. The thin layers make accurate measurements of the Sn gradients by electron probe microanalysis impossible, imposing the more complex, more local and less accurate study by transmission electron microscopy. Analysis of the property distributions by
magnetic characterizations are also not possible, since the outer higher $T_c$ regions magnetically shield the lower $T_c$ sections.

Over the past decade, a Powder-in-Tube (PIT) process has become available as an alternative approach to Nb$_3$Sn wire manufacture. The filaments in modern PIT wires are, in contrast to bronze conductors, nearly perfectly cylindrical, contain thick A15 layers on the order of 5-10 µm and have the higher $T_c$, A15 located on the inside, making them magnetically transparent. Initial compositional analysis indicated that they are much more homogeneous with respect to Sn content than bronze wires, exhibiting gradients on the order of 0.35 at. % Sn/µm. Magnetic (VSM) characterizations of these wires up to 14 T have indicated a large separation between $\mu_0 H_{c2}(T)$ and $\mu_0 H_{c2}(T_0)$ which was correlated to Sn gradients. The latter were visualized by a reduction of the difference between $\mu_0 H_{c2}$ and $\mu_0 H_{c2}$ with increasing reaction times, which are associated with a much stronger rise of $\mu_0 H_{c2}$ than $\mu_0 H_{c2}$.

Table I: Overview of the investigated materials. Depicted are the sample names, a short identification number, the heat treatment for each sample, the wire type and the way each sample was mounted. The next column gives the additional elements that were present during formation of the A15 layers. The next column gives the non-Cu critical current density for each wire, either measured in transport or estimated from magnetization data. The last column gives the non-Cu current density in the samples during measurement of resistive transitions.

| Sample name | ID | Heat treatment | Type | Ø [mm] | Mounting procedure | Additions present | $J_{c \text{non-Cu}}^b$ [A/mm²] | $J_{c \text{non-Cu}}^b$ [A/mm²] |
|-------------|----|----------------|------|--------|-------------------|------------------|-----------------|-----------------|
| SMI ternary PIT | B34-4h | 4h/675°C | PIT | 1.0 | GE Varnish on Cu | Cu, Ta | -- | 0.2 |
| SMI ternary PIT | B34-16h | 16h/675°C | PIT | 1.0 | GE Varnish on Cu | Cu, Ta | 870$^c$ | 0.2 |
| SMI ternary PIT | B34-64h | 64h/675°C | PIT | 1.0 | GE Varnish on Cu and Stycast on Ti-6Al-4V | Cu, Ta | 2250 | 0.2 |
| SMI ternary PIT | B34-768h | 768h/675°C | PIT | 1.0 | GE Varnish on Cu | Cu, Ta | 2170$^c$ | 0.2 |
| SMI reinforced ternary PIT | B134 | 80h/675°C | PIT | 0.6 | Stycast on Ti-6Al-4V | Cu, Ta | 1961 | 0.6 |
| SMI binary PIT | B27 | 128h/675°C | PIT | 1.0 | Stycast on Ti-6Al-4V | Cu | 1955$^c$ | 0.2 |
| Vacuumschmelze ternary bronze | VAC | 220h/570°C+ | Bronze | 0.8 | Stycast on Ti-6Al-4V | Cu, Ta | 556 | 0.5 |
| Fabrikanwa ternary bronze | FUR | 240h/650°C | Bronze | 0.8 | Stycast on Ti-6Al-4V | Cu, Ti | 582 | 0.5 |
| UW-ASC bulk | Bulk | 320h/1020°C | Bulk, Sintered | 0.8×1.4 | GE Varnish on Ti-6Al-4V | -- | -- | 0.004 |

$^a$Samples having a data inconsistency of -4%. $^b$At a voltage criterion of 10µV/m. $^c$Estimated from magnetization data.

A second important difference between wires and thin films and crystals is the presence of Cu and ternary additions as e.g. Ta and Ti. Cu is believed to lower the A15 formation temperature and only to exist at the grain boundaries, but its influence on the critical properties is uncertain. The general consensus on ternary additions as Ta and Ti is that they introduce scattering sites, thereby raising the resistivity and thus $\mu_0 H_{c2}(T)$. These ternary additions may also prevent the formation of tetragonal phases which reduce $\mu_0 H_{c2}(T)$.

A third characteristic of wires is the presence of residual strain. Macroscopically, the strain dependence on $\mu_0 H_{c2}(T)$ is well understood in terms of axial strain or deviatoric strain, but its influence on the complete $H-T$ phase transition is still uncertain. The large Cu matrix enforces a compressive axial strain on the A15 layers due to the larger contraction of the Cu with respect to the A15. This causes a thermal pre-compression in the A15 layer which is spatially inhomogeneous.

The combined effects of Sn gradients, the presence of Cu in the A15 layer, the influence of ternary additions and the presence of strains make it questionable whether the $H-T$ knowledge, derived from uniform, well defined laboratory samples can directly be applied to wires. These considerations stimulated our systematic investigation of the effects of these factors on the $H-T$ phase transition in inhomogeneous Nb$_3$Sn wire systems.

A secondary advantage of a systematic characterization of the $H-T$ phase transition in wires is that it also yields a better understanding of the reasonable performance boundaries for the critical current density ($J_c$). Practical scaling relations for $J_c$ depend on the exact behavior of $\mu_0 H_{c2}(T)$, but are empirically based and generally rely on Kramer extrapolations of lower field data. Moreover, the zero temperature Kramer extrapolated critical field ($\mu_0 H_{c2}(0)$) often results in values far beyond 30 T. This seems unrealistically high in comparison to actual measured results, which are limited to about 29.5 T for higher resistivity, slightly Sn-poor pure Nb-Sn film.

Expressions for the $H-T$ phase transition, derived directly from the BCS theory, are readily available in the literature, and have been applied with success to well defined laboratory specimens, but most of the $H-T$ data on wires are empirically described. This leads to continuing discussions in the literature, partly resulting from the use of extrapolated values of $\mu_0 H_{c2}(0)$ without taking account the inhomogeneous nature of the conductors. Although such empirical relations in principle yield sufficient accuracy for scaling of $J_c$, large discrepancies occur between the fitted...
values for $\mu_0 H_{c2}(0)$ and $T_c(0)$ and actual measurements. However well justified empirically, this discrepancy clearly needs understanding.

The goal of this paper is threefold. First we would like to understand the behavior of $\mu_0 H_{c2}(T)$ for inhomogeneous conductors independent of extrapolation method. Second, we would like to investigate the influences of compositional inhomogeneities, strain and the presence of additional elements on $\mu_0 H_{c2}(T)$. To achieve this, we have selected a sample set that contains variations in manufacturing technique, composition and strain state. Third, we would like to generalize the results by exploring whether an overall description for the $\mu_0 H_{c2}(T)$ behavior of Nb$_3$Sn is possible, independent of measuring technique, criterion for the critical field, strain or compositional variation. We do this by investigating the validity of the Maki-DeGennes (MDG) equation, which is the simplest form of the available theoretical descriptions.

The paper is organized as follows. In Section II the sample materials are presented and in Section III the experimental procedure is explained. In Section IV the experimental data are presented. First we show that inhomogeneities can be visualized by electro-magnetic characterizations and we concentrate on the effects of inhomogeneity reduction by extended heat treatment times on the full $H$-$T$ phase transitions. Second we make comparisons between various conductor types and then we investigate general aspects of the overall $H$-$T$ data. In Section V we discuss the inhomogeneity effects on $\mu_0 H_{c2}(T)$, make comparisons between high field resistive and transport $J_c$ data and analyze the validity of the theoretical fits. Our overall conclusions are presented in Section VI.
II. SELECTED SAMPLE MATERIALS

In Table I an overview is presented of the selected samples with concise identification, the reaction conditions, fabrication procedure, sample cross-section, mounting technique, alloying additions, the non-copper $J_c$ and the non-copper resistive transition measuring current density. Cross-sections of the wires are shown in Figure 1.

The ITER type Furukawa (FUR) and Vacuumschmelze (VAC) wires were selected because they have been characterized extremely well in transport critical current density in various laboratories during the ITER benchmark tests55, 56, 57. They are representative of ternary conventional bronze-route manufacturing methods, have a relative low $J_c$, small filament size and probably large Sn gradients across the A15 areas. The Powder-In-Tube conductors from Shape Metal Innovation (SMI) were chosen since, due to their ‘inside out’ design, they are magnetically transparent55, 26, have a relatively large A15 cross section enabling microscopic composition analysis and are therefore favorable research wires that were already extensively characterized27, 28. They exhibit close to present record current densities and have, compared to bronze-route wires, less steep Sn gradients. A binary (B27) and two ternary versions (B34 and B134) of this wire type were selected. The main difference between the ternary versions is the presence of a Cu-Nb reinforcement tube around the outside of B134, as opposed to the pure Cu matrix used for the regular ternary (and binary) wire. The Cu-Nb reinforcement of B134 results in slightly more deformation of the outer filaments and a larger compressive A15 strain state. The pure Cu matrix ternary conductor (B34) was additionally selected for study of inhomogeneity effects using increased reaction times, which result in increased A15 layer thickness and less steep Sn gradients29. A bulk sample, produced at the University of Wisconsin’s Applied Superconductivity Center (UW-ASC), was used to investigate the behavior of pure binary, Cu-free Nb$_3$Sn.

The first set of samples is a ternary PIT wire (B34) that was heat treated for various times at 675 °C. A reaction time of 64 hours was recommended by the manufacturer (SMI) to yield optimal $J_c$. A specific mounting procedure was applied in an attempt to force the samples into a reproducible, although unknown, strain state after cool down to test temperatures. Four wires were mounted with General Electric Varnish onto a copper substrate. One sample, reacted for 64 hours was mounted with Stycast 2850FT on a Ti-6Al-4V substrate of copper. One sample, reacted for 64 hours was recommended by the manufacturer (SMI) to yield optimal $J_c$. A specific mounting procedure was applied in an attempt to force the samples into a reproducible, although unknown, strain state after cool down to test temperatures. Four wires were mounted with General Electric Varnish onto a copper substrate. One sample, reacted for 64 hours was mounted with Stycast 2850FT on a Ti-6Al-4V substrate of copper.

In the initial stage of the reaction, the light gray region around the core is Nb$_3$Sn, and the darker gray shell around this is A15. At 16 hours the Nb$_3$Sn has disappeared. The bottom row of Figure 1 shows a magnified view across the whole A15 layer in the ternary PIT wire reacted for 64 hours at 675 °C. The core is located to the right and the Nb(Ta) tube to the left. Large, 1-2 µm diameter A15 grains arising from conversion of the initial Nb$_3$Sn, are found next to the core and are not believed to contribute to $J_c$. The large central region is fine-grain A15 with an average grain size of about 140 nm. Similar thick, fine-grain A15 layers have been observed in high $J_c$ Oxford Instruments Superconducting Technology Internal Sn strand material57, which also has an abundant Sn source in the core. Columnar grains are found in the last ~1 µm before the Nb(Ta) tube. A possible cause of the columnar grains is their low Sn content. Thermodynamics dictates that a portion of the A15 layer must be Sn-poor. We also noted that the 768 hour reaction extent was not much greater than after the 64 hour heat treatment, suggesting that the reaction quickly tends to exhaustion.

The second sample type in Table 1 is also a ternary PIT wire manufactured by SMI (B134) which is similar in build to B34 but in this case with a Cu-Nb reinforcement tube surrounding the Cu matrix. It can be expected that the reinforcement results in a larger thermal pre-compression in the A15 from reaction temperature down to room temperature caused by the higher strength of the Cu-Nb as compared to a pure Cu matrix. The third cross-section in the top row of Figure 1 shows that the Cu-Nb reinforcement results in additional deformation of the outer filaments as compared to the not-reinforced PIT wires.

The next sample in Table 1 is a binary PIT wire (B27), again manufactured by SMI. The only difference from the ternary PIT (B34) wire according to the manufacturer is the use of pure Nb tubes. This wire was heat treated beyond the manufacturer’s recommended time which was 47 hours at 675 °C since earlier magnetic characterizations on this wire indicated that a longer reaction of 128 hours yields slightly improved properties57. The top right picture in Figure 1 shows the overall cross-section, while the A15 layer is shown in the lower part for B27, again with the Sn source located at the right side. Large grain A15 is visible at the core interface as in the ternary version of this wire. The remainder of the A15 layer is comparable to the central region of the ternary wire, i.e. fine grain (~140 nm) A15 but the columnar grains at the Nb-tube/A15 interface are less pronounced.

The Vacuumschmelze wire was heat treated with the manufacturer’s recommended reaction. This bronze-route wire uses Nb-7.5 w. % Ta rods that are embedded in a Sn rich bronze matrix that is in its turn surrounded by a Ta diffusion barrier and embedded in a Cu matrix. It is visible in the second cross-section from Figure 1 that the Nb rods are bundled into sub-elements. In the inset ~5 µm filaments are shown with the light gray areas representing the A15. The voids might be caused through Sn depletions in the bronze or possible polishing side effects. The A15 layer cross-section in the bottom part of Figure 1, which has the bronze located to the right, shows that columnar grains are formed over a large area on the A15/Nb(Ta) interface side. A much smaller fine grain equiaxed (high Sn) region which is only about 2-3 grains thick is visible at the bronze interface. A much lower Sn diffusion rate is characteristic of bronze wires as compared to the PIT wires.

The Furukawa bronze-route wire was also heat treated with the manufacturer’s recommended reaction. This wire is constructed with Nb rods embedded in a Ti-containing Sn-rich...
current sampling a greater range of A15 Sn compositions than inhomogeneous A15 distribution results in the measuring although radially inhomogeneous. The sample is a cutout from the wires which are longitudinally rather homogeneous, published earlier.

Field Emission Scanning Electron Microscopy (FESEM) a larger bulk section, produced by Hot Isostatic Pressing (HIP). That are stoichiometric, as well as others low in Sn.

This sections of this not equilibrated sample show local A15 areas therefore three-dimensional inhomogeneities in contrast to the wire samples, which show mainly radial Sn gradients. Cross-sections of this not equilibrated sample show local A15 areas that are stoichiometric, as well as others low in Sn. This inhomogeneous A15 distribution results in the measuring current sampling a greater range of A15 Sn compositions than in the wires which are longitudinally rather homogeneous, although radially inhomogeneous. The sample is a cutout from a larger bulk section, produced by Hot Isostatic Pressing (HIP).

III. EXPERIMENTAL PROCEDURE

A. Resistive characterizations

The majority of the H-T investigations in this paper were performed by resistive characterizations at constant temperature and ramping field. Data for fields ranging from 12-30 T were taken at the National High Magnetic Field Laboratory (NHMFL), Tallahassee FL using a resistive high field magnet while data up to 15 T were obtained using a superconducting solenoid at the UW-ASC. The resistive technique was chosen since this method is easy to set up at the NHMFL and is consistent with our earlier transport Jc characterizations. A disadvantage is that only part of the full property distributions is detected in this way, due to relatively low excitation current densities. A second disadvantage is that the normal state signal of the wires typically was only on the order of 200 nV at zero field, whereas the background noise in the high field resistive magnet area was typically on the order of 50-150 nV. The strong magneto-resistance of the Cu matrix of the wires fortunately delivered an enhancement factor of 4-10 in signal at high field. A nice advantage of the resistive technique is that it is possible to characterize multiple samples at once, saving valuable high field magnet time. The wire sections in the small current resistive measurements were about 8mm long with a voltage tap separation of 1-3 mm and current taps at both ends. The dimensions of the bulk needle sample were 7.75×1.42×0.80 mm3. Samples were mounted on a strain-fixing substrate, according to Table I. The resistive measurements were performed at a small current (generally 0.2-0.6 A/mm2, see Table I), constant temperature and a swept field dμ0H/dt = 10 mT/s. Typical variations in temperature during a constant temperature sweep ranged from ±5 mK in the best case to ±50 mK in the worst case. Low current, constant field, swept temperature (dT/dt = 3 mK/s) measurements were confirmed to deliver the same μ0Hc2(T) data as the swept field characterizations. A detailed description of the technique has been published elsewhere.

A systematic inconsistency of ~4% exists between the NHMFL and UW-ASC datasets for the variable reaction ternary wires (B34), the Furukawa wire and the bulk sample, as indicated in Table I. Temperature measurements in both institutes were confirmed to be correct and the same probe is used in both systems. The magnets in both institutes were recalibrated but no errors were detected. The Furukawa sample has been re-measured in a different resistive magnet over the full temperature range. These results average out the jump that was initially visible between the separate low- and high field datasets. An error source could also be that the strain changes with thermal cycling, despite the attempts to force the samples into a reproducible strain state. An axial strain difference of ~0.06% could result in the observed ~4% inconsistency. No corrections are made on the data since no unambiguous error source could be found and the data should therefore be regarded to be reliable within ~±2%.

B. Data reduction techniques

A typical set of resistively measured transitions is shown in Figure 2 for a ternary PIT wire (B34) reacted for 64 hours at 675 °C. The measured transitions are fitted by a shifted and normalized tangent hyperbolic function multiplied by a polynomial term which describes the magneto-resistance.
The function uses only two fitting parameters, namely $T_c(0)$ and the diffusion constant of the normal conducting electrons $D$. The other parameters are the reduced Planck constant ($\hbar$), the magnetic flux quantum ($\phi_0$) and the Boltzmann constant ($k_B$). Although fuller characterization of paramagnetic limitation and spin-orbit scattering effects, as well as strong coupling corrections and non-spherical Fermi surfaces is provided by Werthamer, Helfand and Hohenberg, we did not find that this added any additional insight into our data on inhomogeneous Nb$_3$Sn conductors.

IV. RESULTS

A. Magnetic tests of inhomogeneity in variably reacted wire

The ternary PIT wires (B34) with 4, 16, 64 and 768 hour reactions at 675 °C were investigated by SQUID magnetization measurements. The resulting normalized magnetic moments as function of temperature at an applied field of 5 mT are shown in Figure 3. At 5 K full flux exclusion can be observed.

| Criterion | Parameter | B34-4h | B34-16h | B34-64h | B34-768h | B134 | FUR | VAC | B27 | Bulk |
|-----------|-----------|--------|---------|---------|----------|------|-----|-----|-----|------|
| 10%       | $\mu_2$ | 27.0   | 27.4    | 28.9    | 29.0     | 28.2 | 28.0 | 27.3 | 26.6 | 26.5 |
|           | $T_c$ [K] | 16.8   | 17.1    | 17.6    | 17.4     | 17.8 | 17.2 | 17.4 | 17.8 | 16.5 |
| 90%       | $\mu_2$ | 28.1   | 28.4    | 29.4    | 29.5     | 29.1 | 28.9 | 28.5 | 27.4 | 28.3 |
|           | $T_c$ [K] | 17.2   | 17.3    | 17.8    | 17.7     | 17.9 | 17.4 | 17.7 | 17.9 | 16.6 |
| 99%       | $\mu_2$ | 28.8   | 29.0    | 29.7    | 29.7     | 29.6 | 29.3 | 29.2 | 27.8 | 29.3 |
|           | $T_c$ [K] | 17.3   | 17.5    | 17.9    | 17.8     | 18.0 | 17.5 | 17.8 | 17.9 | 16.7 |
which makes low
indicates some penetration of the A15 layer through the Nb
However, a depression of the “Nb” transition to ~7.5 K
while barely enhancing the signal magnitude at

\[ \mu \Delta H_c(0) = 27.0-28.1T \]

\[ \mu \Delta H_c(0) = 1.0T \]

\[ \mu \Delta H_c(0) = 27.4-28.4T \]

\[ \mu \Delta H_c(0) = 0.5T \]

\[ \mu \Delta H_c(0) = 28.9-29.4T \]

\[ \mu \Delta H_c(0) = 29.0-29.5T \]

Around 8.5 K the initial transition occurs for the 4 h, 16 h and 64 h reactions. The first transition in the 768 h heat treatment lies substantially lower, starting at 7 K and ending at 8.1 K. The main A15 transitions occur between 10 % and 90 %.

The bold line is an extrapolation of a least squares fit on the data and the lines were calculated with the MDG description. The 1% and 99% lines are used to quantify the extremes of the measured transitions. The main part of the detected property distribution occurs between 10% and 90%. Clearly visible is the reduction of the \(\mu H_c(0)\) transition width (10-90%) with increasing heat treatment time from 1.2 T to 0.5 T. The best A15 sections that are detected (\(\mu H_c(0)-99\%\)) only increase by ~3%, but the lower end of the detected property distribution (\(\mu H_c(0)-1\%\)) increases by ~10%. The reduction of detected \(T_c\) width, as derived from the MDG fits is less obvious but still present, resulting in a width (10-90%) reduction from 0.4 K to 0.3 K.

Figure 4 and summarized in Table II, where the points represent the 1, 10, 50, 90% and 99% normal state-resistance fits to equation 1. The lines are MDG fits to equation 2, using \(T_c(0)\) and \(D\) as free parameters. The majority (~50%) of the detected property distributions are present between the 10% and 90% normal state points. The UW-NHMFL data inconsistency mentioned earlier is visible around \(T = 11-12\) K, causing deviations from the overall \(\mu H_c(T)\) fits at \(T = 12-17\) K.

It can be seen that the \(\mu H_c(T)\) transition width reduces from 1.2 T to 0.5 T as the reaction increases from 4 to 768 hours at 675°C. The better A15 sections (\(\mu H_c(90\%)\)) increase by 1.4 T from 28.1 T to 29.5 T. The lesser A15 sections (\(\mu H_c(10\%)\)) in contrast increase by 2.0 T from 27.0 to 29.0 T and their stronger rise in comparison with \(\mu H_c(90\%)\) is the origin of the transition width reduction with increasing reaction time. The majority of the reduction occurs between 16 h and 64 h. The width reduction effect on \(T_c(0)\) is less pronounced but still visible, changing from 0.4 K to 0.2 K minimum (10-90%). The best A15 sections detected (99%) rise by ~3% in \(\mu H_c(0)\) from 4 to 768 hours, whereas the lower transition (1%) rises ~10%. The extremes in \(T_c(0)\) change by ~3% for the best detected sections and ~4% for the lower transition. Optimum critical current densities, expressed as the critical current divided by the total Nb(Ta)+A15+core package area, are achieved at 64 hours. Maximum \(J_c\) appears to correlate to the rise to maximum \(\mu H_c(90\%)\) seen on going from 16 to 64 hours, before any Sn escapes from the Nb(Ta) tube or additional grain growth that lowers the flux pinning occurs.

The MDG fits at 12 K derived from the resistive transition data are compared to the magnetic data in Figure 5. The points and dotted lines are derived from the resistive data in Figure 4.

**B. Resistive visualization of inhomogeneity reduction via elongated reactions**

To expand the inhomogeneity investigations on the ternary PIT (B34) wire to the whole \(H-T\) range, we switched to the resistive characterization of the \(\mu H_c\) transition, as shown in
and the shaded areas are the magnetically (VSM and SQUID) derived property distributions from Figure 3 and a reproduction from earlier published\textsuperscript{27} VSM data at $T = 12$ K. The decreasing difference from $-5.4$ T to $-2.4$ T between the magnetically derived $\mu H_c(12$ K) and $\mu H_c(12$ K) with increasing reaction time is a direct result of the Sn gradient reduction in the A15 layer, in agreement with recent simulations\textsuperscript{23}. The reduction of the transition width in the resistive characterizations is less obvious (from $\sim 1.1$ to 0.6 T for the 10 and 90 % normal state resistance points) but still clear. The highest $\mu H_c(12$ K) as detected in the resistive characterizations (at 99 % normal state resistance) is identical to and increases in the same way as the VSM determined $\mu H_c(12$ K). The SQUID derived $T_c(0)$ transition width reduces from 1.3 to 0.9 K, whereas the resistive transition width (between 10 and 99 % normal state resistance) reduces from 0.4 to 0.3 K. The SQUID derived $T_c(0)$ values, however, are substantially lower than in the resistive data. The magnetic and resistive characterization techniques are therefore, apart from the highest detected $\mu H_c(12$ K) values, only qualitatively comparable. This stems from the fact that small current density resistive transitions sample only a small fraction of the A15 with the highest property.

C. Inhomogeneity differences between the conductors

An overview of the collected $\mu H_c(T)$ data for all resistively characterized samples, reacted ‘normally’ (see Table I), is given in Figure 6 and summarized in Table II. The plots are again separated in temperature for enhanced visibility and developed as for the ternary PIT data in Figure 4.

The order from top to bottom in Figure 6 is approximately of increasing inhomogeneity. The transition widths with respect to $\mu H_c(0)$ range from 0.5 T for the ternary PIT wire (B34) reacted at 64 hours, to 1.8 T for the bulk sample. The fitted $T_c(0)$ variation ranges from 0.1 K for the binary and reinforced ternary PIT wires and the bulk sample to 0.3 K for the Vacuumschmelze bronze wire. It is interesting to observe the

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**Figure 5:** Summarized result from Figure 4 depicting the effect of reaction time on the resistively measured $\mu H_c$-99 % (12 K), $\mu H_c$-90 % (12 K), $\mu H_c$-10 % (12 K), $T_c$-99 % (0), $T_c$-90 % (0) and $T_c$-10 % (0) calculated from the MDG fits (dotted lines plus data points) in comparison with the SQUID and VSM data from Figure 3 and Fischer et al.\textsuperscript{23} (depicted by the shaded regions). The general trends for the critical fields versus reaction time as measured resistively are similar to the magnetic data but the detected transition widths are much smaller in the resistive characterizations. The highest $\mu H_c(12$ K) values (i.e. at 99 % normal state resistance) for the resistive data coincide with the VSM determined $\mu H_c(12$ K) values. A difference is visible between the detected $T_c(0)$ values in addition to the reduced transition width in the resistive characterizations.

**Figure 6:** Collected $\mu H_c(T)$ data for resistively characterized samples from 1 to 99 % normal state. The plots are separated in temperature for enhanced visibility. Included are the range of values and detected property distribution widths for the 10-90 % normal state criteria. The points were derived from the resistivity fits on the data and the lines were calculated with MDG. The main part of the detected property distribution occurs between 10 % and 90 %. The 1 % and 99 % lines are used to quantify the extremes of the measured transitions. The order from top to bottom is approximately of increasing inhomogeneity.
substantial differences that occur between the two bronze conductors. The Ti-alloyed Furukawa wire exhibits a smaller distribution (0.9T and 0.2 K) and better overall properties than the Ta-alloyed Vacuumeschmelze wire (1.2 T and 0.3 K).

The bulk needle sample shows a very large distribution in $\mu H_{c2}(0)$ of 1.8 T, or even 3.9 T if the outer extremes (1 and 99 %) are taken into account. This large spread is indicative of its large Sn inhomogeneity. This sample exhibits a three dimensional distribution of local areas of high and low Sn content and no continuous high A15 quality current path therefore exists. The wire samples, in contrast, exhibit a radially distributed Sn gradient and thus have longitudinal parallel paths of different, but approximately constant Sn content. The ternary PIT wire (B34) is known to exhibit its large Sn inhomogeneity. This sample exhibits a three dimensional distribution of local areas of high and low Sn content. The ternary PIT wire is identical to the magnetically (VSM) derived $H_{c2}$ data for the limited overlap that is available at $T = 12$ K. This comparison validates our conclusion that the highest detectable $\mu H_{c2}$ can be probed using completely different characterization methods.

**D. The best properties detected in various conductors**

The highest accessible $\mu H_{c2}(T)$ data (at 99 % normal state resistance) for all samples are collected in Figure 7 and Table II. Considering the varying transition breadths in Figure 6, the highest $\mu H_{c2}(T)$ A15 sections in the ternary wires are strikingly constant. These high $\mu H_{c2}(T)$ A15 regions are present in all ternary wires and are very comparable: $\mu H_{c2}(0) = 29.5 \pm 0.3$ T and $T_c(0) = 17.8 \pm 0.3$ K. A small difference can be observed between the bronze and PIT conductors, the highest detected phase boundaries in the bronze wires being slightly lower than in the PIT wires. This small difference in $\mu H_{c2}(T)$ is in strong contrast to their large critical current density differences which are approximately 4 times higher in the PIT wires (see Table I).

The inset in Figure 7 compares the binary PIT wire to the binary bulk needle. The binary PIT shows a reduced maximum $\mu H_{c2}(0)-99$ % of 27.8 T but a comparable $T_c(0)-99$ % of 18.0 K. The bulk needle has a reduced $T_c(0)-99$ % of 16.7 K but, in comparison to the binary PIT wire, a very high $\mu H_{c2}(0)-99$ % of 29.3 T which is in the range of the ternary wires.

**V. DISCUSSION**

**A. Overall behavior of the H-T phase boundary**

The upper (90-99 %) transitions of all ternary wires are remarkably similar (Figure 7), in contrast to the large range of transition breadth (Figure 4 and 6) and grain morphology differences (Figure 1). The binary PIT wire has a suppressed $\mu H_{c2}(0)$ while retaining a comparably high $T_c(0)$ in comparison to the ternary wires. The bulk needle exhibits a $\mu H_{c2}(0)$ which is comparable to the ternary wires but shows a suppressed $T_c(0)$. Orlando et al.\(^2\) have shown similar behavior in thin films with varying resistivity: Increasing the resistivity caused $\mu H_{c2}(0)$ to rise, at the cost of a reduction in $T_c(0)$.

Their optimal dirty film ($\rho_{99} = 35 \mu\Omega cm$) had $\mu H_{c2}(0) = 29.5$ T and $T_c(0) = 16.0$ K at a 50 % resistive criterion, compared to 26.3 T and 17.4 K for a $\rho_{99} = 9 \mu\Omega cm$ film. Our bulk needle exhibited a resistivity just above $T_c(0)$ of 22 $\mu\Omega cm$ and $\mu H_{c2}(0)$ and $T_c(0)$ at a 50 % normal state resistance criterion are 27.4 T and 16.5 K respectively. However, our bulk needle was far from fully homogenized, meaning that for a more honest comparison a higher resistive criterion might be more suitable. A 90 % normal state resistance results in $\mu H_{c2}(0) = 28.3$ T and $T_c(0) = 16.6$ K. Both the 50 % and 90 % values appear consistent with the thin film data from Orlando et al., i.e. at $\rho_{99} = 22 \mu\Omega cm$ our bulk needle phase boundary is positioned between the 9 and 35 $\mu\Omega cm$ thin film data. In addition, a (partial) transformation to the tetragonal phase could also reduce $\mu H_{c2}(0)$.

The upper (90-99 %) resistively determined transitions for the ternary PIT wire are identical to the magnetically (VSM) derived $H_{c2}$ data for the limited overlap that is available at $T = 12$ K. This comparison validates our conclusion that the highest detectable $\mu H_{c2}$ can be probed using completely different characterization methods.

**B. Comparisons of resistive H-T data and transport $J_c$ data**

To investigate how $\rho(\mu H)_T=4.2K$ data correlates to $J_c(\mu H)_T=4.2K$ characterizations, a Kramer plot of high field $J_c$ data on a ternary PIT wire (B34-64h) is combined with a resistive $\mu H_{c2}(4.2 K)$ transition on the same wire in Figure 8. The $J_c$ sample was mounted with Stycast on a helical Ti-6Al-4V barrel\(^29\) with reduced diameter and the resistive $\mu H_{c2}$ transition sample was mounted in an identical way to reproduce the same strain state. The Kramer plot of the $J_c$ data is indeed highly linear, almost up to $\mu H_{c2}(4.2 K)$. The extrapolated value for $\mu H_{c2}(4.2 K)$ (24.9 T) is in agreement with magnetic (VSM) Kramer data at 4.2 K found by Fischer\(^30\) which were measured in the range $\mu H = 6-14$ T, showed perfect Kramer linearity and also extrapolated to $\mu H_{c2}(4.2 K) = 25$ T. This indicates a straight Kramer plot from $\mu H = 6$ to $-23$ T, the magnetic field value for which the
critical current data starts to deviate from linear Kramer behavior. The observed linearity and small tail for the PIT wire is in agreement with recent modeling using actual measured Sn gradients as input data.

Two particular points of the resistive transition are important: One occurs at 10 μV/m, the voltage criterion used in the transport $J_c$ characterization, and one at 99 % of the normal state resistance, identifying the highest observed value for $μ_0H_{c2}(4.2 \, K)$. The 10 μV/m point on the resistive transition represents a critical current density of 0.2 A/mm² at 4.2 K and ~25 T (the excitation current) and yields $0.2/25^2 = 1 \, A/\, T$ on the Kramer axis. The transport $J_c$ data at 4.2 K and 25 T yield $3 \, A/\, T$, which is a very reasonable agreement.

The linear Kramer extrapolation of the measured $J_c$ data yields $μ_0H_{c2}(4.2 \, K) = 24.9 \, T$ as the scaling critical field for $J_c(4.2 \, K)$. The highest $μ_0H_{c2}(4.2 \, K)$ detected in the resistive transitions, yields 26.9 T for 99 % normal state resistivity. We can now estimate the hypothetical $J_c(4.2 \, K)$ gain which would be achieved if the entire A15 layer would be of the highest $μ_0H_{c2}(4.2 \, K)$ quality and grain size and grain boundary densities could be retained, in a way similar as recently done by Cooley et al. who used an identical approach based on modeling the A15 Sn gradients. We use the definition of the Lorentz force that balances the bulk pinning force:

$$J_c(μ_0H) × μ_0H = -F_L(μ_0H), \quad (3)$$

and combine this with the temperature- and field dependence of the bulk pinning force in its most recent form to yield:

$$J_c(μ_0H, T) = \frac{\text{Const}}{μ_0H} \times [μ_0H_{c2}(T)]^2 \times h^{1/2} (1 - h)^2, \quad (4)$$

where $h = μ_0H / μ_0H_{c2}(T)$. Increasing the critical field for $J_c$ scaling from 24.9 to 26.9 T at $T = 4.2 \, K$ thus would result in a rise in non-Cu $J_c(12 \, T, 4.2 \, K)$ from 2250 A/mm² to 2883 A/mm². This indicates that the non-Cu area in this PIT wire carries ~78 % of what would hypothetically be achievable. The difference with the value found by Cooley et al. (60 %) is due to different area normalization for $J_c$.

It should be noted that a Kramer extrapolated $μ_0H_{c2}(4.2 \, K) = 24.9 \, T$ arises from a MDG fit using $μ_0H_{c2}(0) = 27.7 \, T$ and $T_c(0) = 17.6 \, K$. These values are a little smaller than for the lower resistive transition at 1 % normal state resistance which yielded $μ_0H_{c2}(0) = 28.3 \, T$ and $T_c(0) = 17.8 \, K$ as fitted values. This indicates that the values required for critical current density scaling lie slightly below the range of transitions that are detected in the resistive characterizations. This is indicated in the inset in Figure 8, where the highest- and lowest detected $μ_0H_{c2}(T)$ transitions are plotted together with the transition that is appropriate for $J_c$ scaling.

C. The use of the MDG description

The overall accuracy of the MDG description is demonstrated in the normalized plot in Figure 9. All of our resistively measured data are well described by the MDG fit, independent of whether 1, 10, 50, 90, or 99 % is applied as the criterion for defining $μ_0H_{c2}$. Included are resistive measurements on the samples from Table I as well as additional sample material, magnetic data using the onset of superconducting behavior, literature data that has been measured resistively and with RF techniques and Kramer extrapolated critical field data resulting from transport $J_c$ characterizations. The route that was followed in order to arrive at Figure 9 is that all $μ_0H_{c2}(T)$ data points for a specific sample were least squares fitted with the MDG equation and the resulting values for $μ_0H_{c2}(0)$ (calculated from $T_c(0)$ and $D$) and $T_c(0)$ were used as normalization parameters. The only deviations occur very close to $T_c(0)$ in a few of the samples, but the overall shape of the normalized phase boundary is strikingly similar.

The MDG description clearly is efficient in describing the $H$-$T$ phase transition for Nb$_3$Sn independent of the applied criterion, strain state or sample layout, as is visible from Figure 9. It has the advantage of using only two fitting parameters although the physical meaning of these two parameters can be argued. This description was originally intended to describe dirty, weak coupling superconductors, uses a spherical Fermi surface approximation and does not take into account paramagnetic limiting or spin orbit scattering. The inhomogeneities that are present in practical wires, combined with the lack of accurate A15 resistivity data however rule out rigorous connections to microscopic theories. For theoretical connections to the fundamental parameters, as well as for corrections of the applied approximations, physically more realistic models are required. However, these are not yet available.
The samples in bold are presented in this publication. The temperature phase boundary have taken place. Figure 9 makes a convincing statement that a simple generalized function is valid for all investigated Nb3Sn.

An experimental study has been made of the field-temperature phase boundary in the present generation of optimized but still inhomogeneous wires. The highest detected upper critical fields at zero temperature and the critical temperature at zero field are remarkably constant for all investigated ternary wires and amount to 29.5 ± 0.3 T and 17.8 ± 0.3 K respectively.

Reduction of wire inhomogeneities by extended heat treatment times was shown by mapping the transition breadth and was demonstrated to be qualitatively the same for resistive and magnetic characterization techniques. The reduction of breadth is attributed to Sn enrichment of the A15 layers in the wires.

The Maki-DeGennes function has proven to yield an excellent description for the field-temperature behavior of Nb3Sn for the entire range of investigated conductors as well as for the thin film and poly- and single crystal data from the literature. In normalized form all \( \mu H_c(T) \) dependencies follow one single description, independent of composition, strain state, sample layout or transition criterion. The function also fits the Kramer extrapolated critical fields.

We can additionally introduce a function that summarizes the available \( \mu H_c(\text{at. \% Sn}) \) data:

\[
\mu H_c(\text{at.\% Sn}) = -10^{-30} \exp\left( \frac{\text{at.\% Sn}}{0.348} \right) + 5.77 \text{at.\% Sn} - 107, 
\]

which includes a \( \mu H_c(T) \) suppression near the stoichiometric Sn concentrations due to a cubic to tetragonal phase transition, and combine these with (2) and (6) to fix the \( H-T \) phase boundary as a function of Sn content if we neglect strain influences.

**VI CONCLUSIONS**

We can summarize Devantay’s \( T_c(\text{at. \% Sn}) \) data on homogeneous bulk samples\(^{16,19}\), using a linear fit as usually applied\(^{16,29,30}\) or more accurately with:

\[
T_c(\text{at.\% Sn}) = \frac{-12.3}{1 + \exp\left( \frac{\text{at.\% Sn} - 22}{0.9} \right)} + 18.3.
\]
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