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Role of elemental additions in Nb₃Sn phase formation in wires

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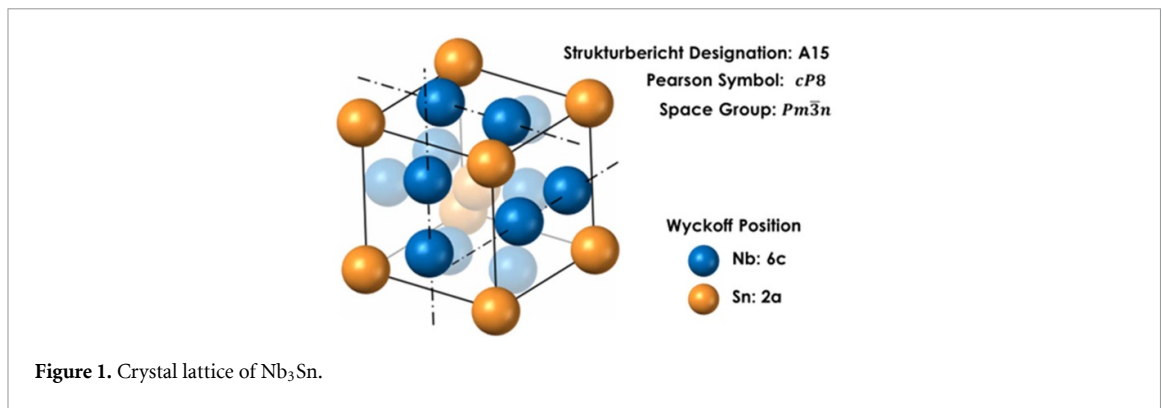
Keywords: Nb₃Sn, thermodynamics, elemental addition, growth kinetics, zinc addition, titanium addition

Abstract

Nb₃Sn remains an indispensable core material for high-field applications, including fusion reactors, particle accelerators, and nuclear magnetic resonance systems, owing to its manufacturability and superior high-field performance, making it a key enabling material for advanced energy technologies required for a sustainable and decarbonized future. The enduring appeal of Nb₃Sn stems from the incomplete elucidation of its phase formation more than 70 years after its discovery, which suggests significant potential for further performance enhancement. This article reviews recent progress in Nb₃Sn conductor development from the perspectives of thermodynamics, diffusion kinetics, and microstructural control via elemental additions. In contrast to previous reviews that primarily focused on conductor performance, processing routes, or artificial pinning center technologies, the present review places particular emphasis on phase formation during diffusion reactions and on the mechanisms by which elemental additions modify reaction pathways, intermediate phases, diffusion behavior, and microstructural evolution. This review focuses on the control of the Sn chemical potential through Cu addition, Sn concentration gradient within the Nb₃Sn layer resulting from slow bulk diffusion, and Cu diffusion along the Nb₃Sn and Nb grain boundaries. The unique role of Cu in facilitating the bronze process is discussed alongside the chemical reactivity of Cu with Nb and Sn. Furthermore, this review systematically summarizes the effects of Ti and Zn additions, including variations in interfacial compound formation depending on the Ti addition site, grain-boundary pinning and grain size modification associated with Cu segregation, and effective multielement addition strategies. Finally, the strengthening of the tin core through elemental additions as a primary method for achieving optimal drawability in advanced Nb₃Sn conductors is addressed. This review provides a thermodynamic and kinetic framework for understanding Nb₃Sn layer formation through elemental-addition strategies.

1. Introduction

Before the discovery of NbTi superconductors [1, 2, 3], Matthias *et al* discovered Nb₃Sn superconductors (A15-type compound superconductors) in 1954 at Bell Laboratories (figure 1) [4]. Despite a history spanning 70 years, these materials continue to fascinate superconducting materials scientists. As a type-II superconductor, Nb₃Sn possesses a coherence length that exceeds the grain boundary thickness, which allows crystal defects such as grain boundaries to function as effective flux-pinning sites without weak coupling issues [5–11]. High-performance Nb₃Sn superconductors are comprised of dense compound polycrystals, for which low-temperature diffusion synthesis using Nb and Sn alloys is well-suited to produce refined, dense crystal structures. The superconducting properties depend on a comprehensive range of crystallographic factors, including the homogeneity (concentration gradient) of the superconducting layer, grain boundary density, and the precipitation of impurities. Complex thermodynamics, governed by the geometric arrangement of precursor cross-sections, composition ratios, diffusion reaction conditions, elemental additions, and other factors, control the formation of these dense polycrystalline layers. Thus, the synthesis of the Nb₃Sn superconducting layer offers considerable scope for leveraging materials science expertise. This fundamentally different development philosophy remains distinct from that of



rare-earth barium copper oxide (REBCO) superconductors, which require highly oriented single-crystal superconducting layers, further contributing to the scientific interest in the Nb₃Sn system. In addition, the wire-fabrication processes for Nb₃Sn conductors are well suited to large-scale production, representing a further advantage over REBCO conductors for many practical magnet applications.

Numerous review articles on Nb₃Sn conductors have been published over the past decades. Representative recent reviews include the review by Godeke, which summarized composition and morphology effects, strain sensitivity, Cu/Ti/Ta additions, and grain-size-related issues [12]; the review by Xu, which focused on phase fractions, stoichiometry, Sn content, Ti addition, artificial pinning centers (APCs) based on internal oxidation, and reaction pathways toward high- J_c conductors [13]; and the review by Barzi, which reviewed the historical development of Nb₃Sn wires and conductors from the perspective of magnet applications, including magnetothermal instabilities and subelement deformation [14]. For a broader overview of previous reviews, readers are referred to [15].

Although a substantial body of knowledge on Nb₃Sn superconductors has been accumulated and systematically reviewed over many years, achieving precise control of Nb₃Sn layer formation remains challenging, even for materials specialists. This difficulty arises from the strong interplay among thermodynamics, diffusion kinetics, phase evolution, and microstructural development during the reaction process. To provide a unified perspective on these interrelated phenomena, the author previously published a review in 2023 focusing on the relationships among processing, microstructure, and superconducting performance, together with introductory discussions on growth kinetics, nucleation theory, chemical potential, elemental additions, and matrix strengthening [15].

Nb₃Sn superconductors possess a critical temperature exceeding 18 K [16], and a critical magnetic field of approximately 23 T at 4.2 K [17]. Ti and Ta additions significantly enhance the critical magnetic field (B_{c2}) to approximately 27 T [17]. Beyond these intrinsic properties, its manufacturability establishes Nb₃Sn wire as the primary candidate for next-generation particle accelerators, such as the Future Circular hadron-hadron Collider (FCC-hh) and prototype DEMOnstration Power Station (DEMO) magnet conductors. Estimated total shipments for both applications are expected to reach several thousand tons. Furthermore, Nb₃Sn wire remains essential for nuclear magnetic resonance systems with resonance frequencies of 400 MHz or higher (corresponding to magnetic fields of 9.4 T or greater) in drug discovery and related fields. Demand for this wire is projected to remain robust.

The recent development of high-performance Nb₃Sn conductors is being driven by the demanding requirements of future large-scale projects, including the FCC-hh at CERN [18] and various global fusion-energy programs. These programs encompass Q-DEMO, an ITER-sized demonstration reactor project led by QST in Japan [19], EU-DEMO [20, 21], DTT (Divertor Tokamak Test facility) in Italy [22], and two Chinese fusion projects: CFETR (China Fusion Engineering Test Reactor) [23] and CFEDR (China Fusion Energy Demonstration Reactor) [24]. These demanding projects have stimulated Nb₃Sn development activities worldwide, involving Japan [25, 26], the European Union, the United States of America [27–29], South Korea [30, 31], China [32], and Russia [33]. The broader research and development community encompasses universities, national laboratories, and industrial suppliers working across multiple countries and regions. Major institutions include CERN, Fermilab, LBNL, NIMF, ACIP, KEK, QST, NIFS, NIMS, and leading universities such as the University of Geneva, the University of Oxford, the University of Twente, TU Wien, Ohio State University (OSU), and Tohoku University. These organizations are actively investigating superconducting properties such as B_{c2} [34–37], stress-strain tolerance [38–45] and thermo-magnetic instability [46, 47], and neutron irradiation effects [48–50], as well as phase formation and microstructural evolution.

A primary performance target for next generation Nb₃Sn conductors is a critical current density (J_c) of 1200–1500 A mm⁻² at 15 T, corresponding to the target specification for FCC-hh magnets [18, 51]. To achieve this target, wire manufacturers and research institutions worldwide are vigorously pursuing conductor optimization. Bruker-OST/Bruker USA Restacked Rod Process (RRP) wires have long served as a benchmark for high-performance conductors [52] and have been widely adopted by major institutions such as CERN. A notable example of project-driven conductor development is provided by KAT (Kiswire Advanced Technology), which developed Nb₃Sn wires meeting the more demanding performance requirements of the DTT project, beyond those specified for ITER conductors [30]. Furthermore, OSU/Fermilab/Hyper Tech's APC Nb₃Sn wires have recently demonstrated critical current densities exceeding the FCC target specification, representing a significant advance in high-performance conductor development [29]. In parallel, recent compact-fusion reactor concepts have increased the demand for high- J_c Nb₃Sn conductors. Consequently, achieving high J_c within increasingly limited magnet volumes has become one of the most important objectives in contemporary Nb₃Sn wire development.

Enhancing wire strength is also essential for large-scale magnets to withstand increased electromagnetic forces. The FCC-hh magnet requires resistance to lateral compressive stresses of 200 MPa [53–55]. Similarly, the DEMO magnet requires high-strength, high-performance wires capable of withstanding complex stress–strain distributions, including bending, tension, and compression, arising from intricate wire trajectories in the multistage twisted cables [45, 56–58].

Additionally, reduction of the effective superconducting filament diameter in Nb₃Sn wires is required under time-varying magnetic fields to mitigate hysteresis losses and improve stability [59, 60]. Consequently, next-generation Nb₃Sn superconducting wires must simultaneously satisfy three key requirements, namely, high J_c , high mechanical strength, and a low effective filament diameter.

Against this backdrop, and building upon the author's previous review [15], this paper reviews recent research in Nb₃Sn conductor development from the perspectives of thermodynamics and growth kinetics to elucidate the Nb₃Sn layer formation mechanism and to achieve simultaneous critical current density and mechanical strength improvements. The present review places greater emphasis on phase formation during diffusion reactions and on the role of elemental additions in controlling reaction pathways, intermediate phases, diffusion behavior, and microstructural evolution in superconducting wires. Compared with previous reviews, this article provides a more in-depth discussion of these topics and incorporates several insights emerging from recent studies.

This review outlines the diffusion-based formation of Nb₃Sn layers, emphasizing chemical potential as a vital concept for understanding phase formation and layer growth. Furthermore, this article explains how elemental additions establish interfacial diffusion barriers or accelerate phase formation. This paper demonstrates that elemental additions facilitate matrix microstructural control and that wires possessing both high strength and superior performance result from appropriate element selection and cross-sectional microstructural regulation. Finally, this paper introduces recent findings demonstrating that elemental additions control the Nb₃Sn grain boundary composition, thereby modifying the contribution of elemental pinning to the grain-boundary pinning force.

2. Fundamental issues in Nb₃Sn layer formation

A summary of representative Nb–Sn–Cu phases related to the Nb₃Sn diffusion reaction is provided in table 1 [61–63]. These phases appear during different stages of the reaction process and play important roles in controlling diffusion pathways, reaction kinetics, and Nb₃Sn layer formation.

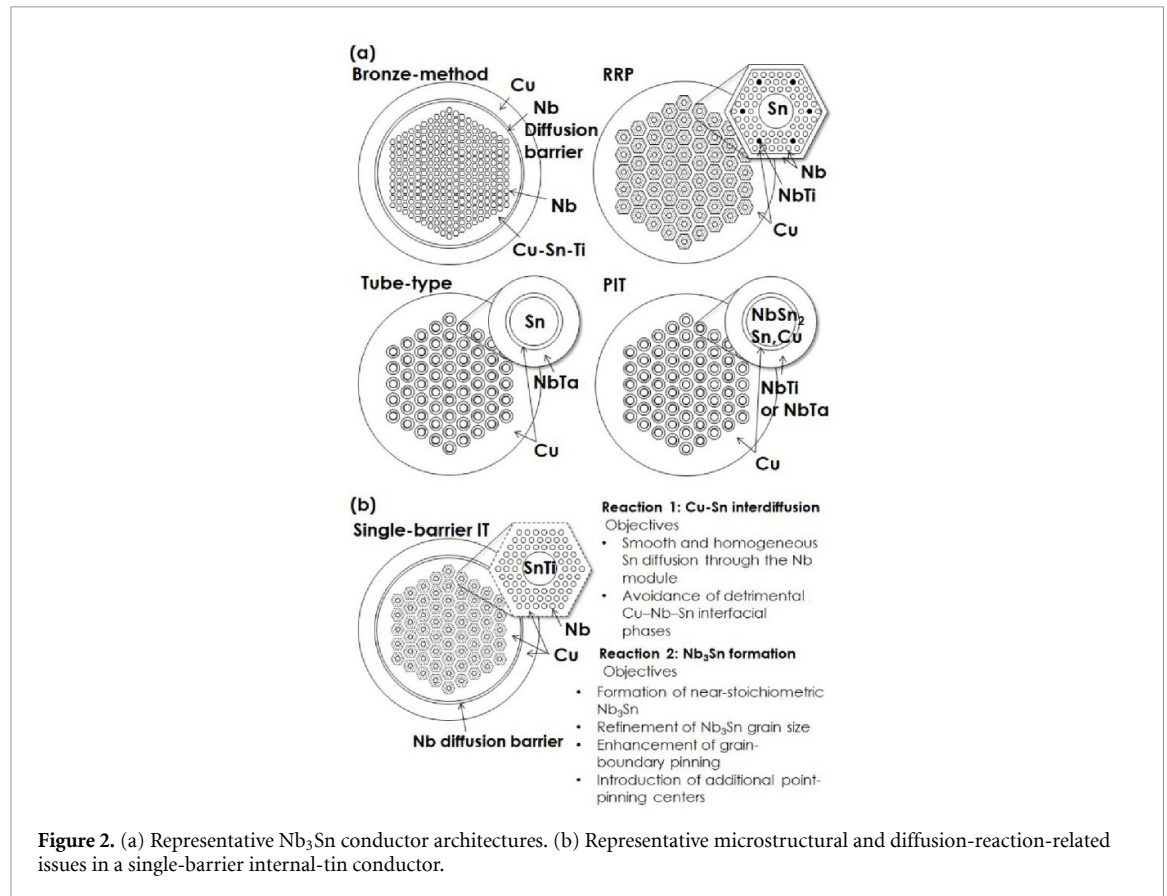
As discussed throughout this review, industrial Nb₃Sn conductors are fabricated through diffusion reactions between Nb and Sn-containing Cu alloys. The conductor architectures used to realize these diffusion reactions can be broadly classified into the bronze-route, internal-tin (single-barrier, RRP, tube-type, and distributed-Sn designs), and powder-in-tube (PIT) processes. Representative conductor architectures are schematically illustrated in figure 2(a).

Compared with the bronze-route process, the internal-tin and PIT processes separate the Sn source from the Cu matrix, thereby avoiding the limitation of Sn supply inherent to bronze-route conductors and enabling higher Nb₃Sn volume fractions and higher non-Cu J_c values. Another important characteristic of RRP and PIT conductors is that Ti or Ta is introduced from the Nb-module side, which contributes to the formation of a more homogeneous Nb₃Sn layer, as discussed in later sections.

Since the present review focuses on thermodynamics, diffusion kinetics, and phase formation during Nb₃Sn layer growth, it is useful to first identify the principal issues associated with diffusion reactions in multifilamentary conductors. Therefore, figure 2(b) summarizes several representative microstructural

Table 1. Representative Nb–Sn–Cu phases involved in the Nb₃Sn diffusion reaction.

Phase	Remark
Bronze	Cu–Sn solid solution containing typically 8–9 at% Sn, used as the Sn source during reaction heat treatment.
Nausite [61, 62]	Nb–Cu–Sn ternary compound ((Nb _{0.75} Cu _{0.25})Sn ₂) formed at ~360 °C–550 °C, subsequently decomposing into NbSn ₂ and Nb ₆ Sn ₅ .
NbSn ₂ [63]	Sn-rich intermediate intermetallic phase ($T_c \approx 2.68$ K), generally regarded as an undesirable precursor phase during Nb ₃ Sn formation.
Nb ₆ Sn ₅ [63]	Sn-rich intermediate intermetallic phase ($T_c \approx 2.07$ K), formed prior to Nb ₃ Sn and generally undesirable for conductor performance.
Nb ₃ Sn	A15-type superconducting compound ($T_c \approx 18$ K), serving as the primary current-carrying phase in practical superconducting wires.



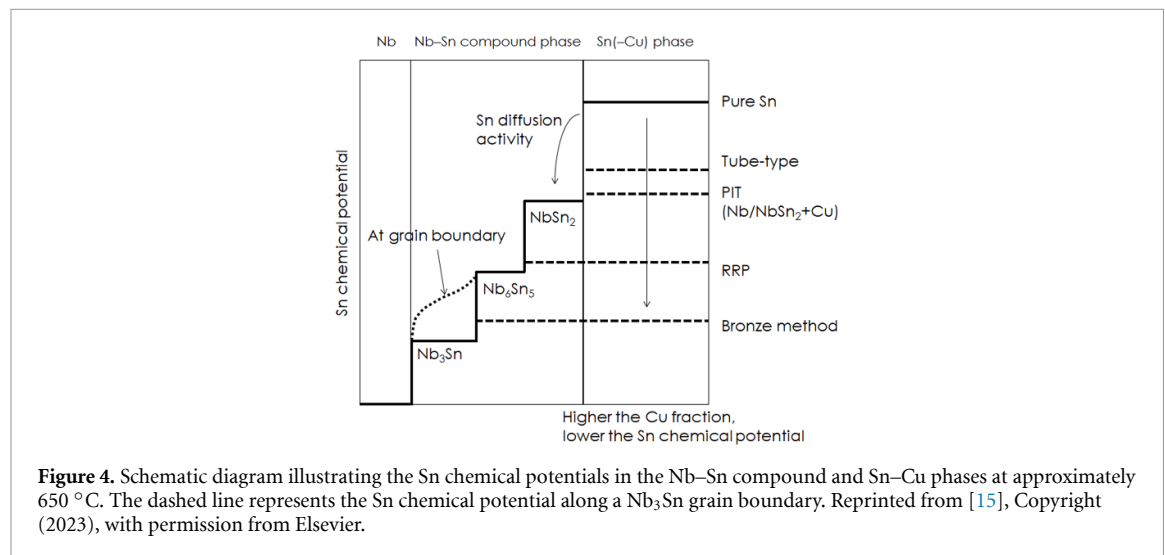
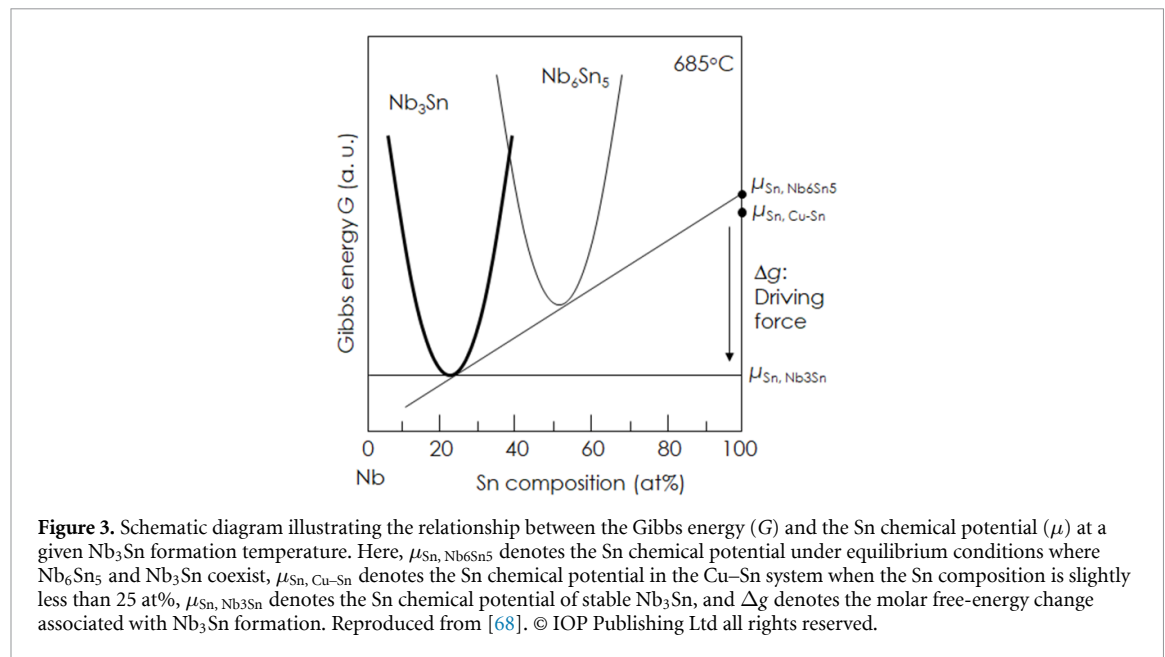
and reaction-related issues using a single-barrier internal-tin conductor as an example. Elemental additions are among the most effective approaches for addressing many of these issues and are therefore a central focus of this review.

This section reviews several classical models relevant to Nb₃Sn layer formation alongside recent findings by the author on elemental diffusion phenomena.

2.1. Role of Cu in the Sn diffusion driving force

As shown in the equilibrium phase diagram, the melting point of completely molten Nb₃Sn exceeds 2200 °C; such elevated temperatures induce significant grain growth within the Nb₃Sn layers. For context, in other A15-type compound superconductors, such as Nb₃Al, the grain size produced directly from the molten state via electron beam melting typically reaches several hundred nanometers or larger [64]. This coarsening is detrimental to Nb₃Sn superconductors, where grain boundaries serve as the primary flux-pinning sites. Conversely, the formation of a binary Nb/Sn diffusion couple at low temperatures primarily yields the NbSn₂ compound phase at the interface, which inhibits the formation of the desired Nb₃Sn phase [15].

The bronze-route reaction process was developed as a strategic solution to this problem. This method facilitates a diffusion reaction between a bronze alloy, in which Sn (or Ga) is solidified in Cu and Nb



(or V), thereby enabling the synthesis of thick Nb_3Sn (or V_3Ga) layers [65–67]. Heat treatment is generally conducted at 650 °C – 750 °C, producing Nb_3Sn grain sizes in the range of 100–200 nm. This manufacturing protocol remains the industrial standard for producing Nb_3Sn wires. Chemical potential serves as a critical conceptual framework for understanding the thermodynamics of Nb_3Sn layer formation facilitated by Cu additions.

While Fick’s first law dictates that elemental diffusion fluxes proceed from regions of higher concentration to lower concentration, this principle does not apply to systems where intermediate phases form at the interface. In the bronze-route reaction, the Sn diffusion flux proceeds from the Cu alloy with a Sn concentration of approximately 9% to the Nb_3Sn phase with a higher Sn concentration of approximately 25%. This phenomenon, historically characterized as ‘uphill diffusion’ [40], is governed by the chemical potential, which describes the movement of elements as a release of free energy between two phases. The chemical potential is defined as the Gibbs energy per mole of a substance (element or compound) when present in a mixture. The magnitude of the difference in Gibbs free energies per mol between the precursor and product phases determines the reaction strength and direction; consequently, the chemical potential gradient acts as the driving force for diffusion.

Figure 3 schematically illustrates the relationship between the Sn chemical potential and the Gibbs energy of Nb_3Sn within the Nb–Sn system at a Nb_3Sn formation temperature of 685 °C. Furthermore, figure 4 provides a conceptual illustration of this relationship regarding the compound phases and the Sn chemical potential.

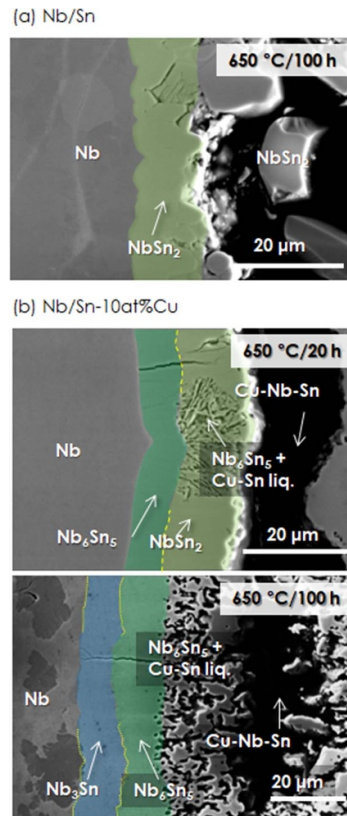


Figure 5. Role of Cu in the Nb/Sn diffusion reaction. Scanning electron microscope (SEM) images of (a) Nb/Sn and (b) Nb/Cu–Sn heat-treated at 650 °C. During the Nb/Sn diffusion reaction, NbSn₂ forms predominantly, whereas phase formation in Nb/Cu–Sn proceeds through Nb₆Sn₅ and subsequently to Nb₃Sn. Reprinted from [15], Copyright (2023), with permission from Elsevier.

Figure 5 compares the actual interfacial microstructure in the Nb/Sn and Nb/Sn-10at%Cu diffusion couples after the reaction [15]. In the absence of Cu, the chemical potential of Sn remains extremely high, indicating a strong Sn diffusion driving force, preferentially forming NbSn₂, which has a relatively high Sn chemical potential. In pure Sn, this high Sn chemical potential persists, facilitating the continuous growth of NbSn₂ and precluding the formation of Nb₆Sn₅. Conversely, the addition of a small quantity of Cu to the Sn core initially produces NbSn₂, but subsequently decreases the Sn chemical potential, thereby slowing the growth rate of NbSn₂. Subsequently, Nb₆Sn₅ with a lower Sn chemical potential emerges at the reaction front. As the reaction proceeds, the growth rate of Nb₆Sn₅ decreases, allowing the fine-grained Nb₃Sn phase to nucleate and grow at the reaction front. Eventually, Nb₆Sn₅ decomposes to form coarse-grained Nb₃Sn while simultaneously liberating Sn, which contributes to the formation of fine-grained Nb₃Sn [69, 70]. Furthermore, the solid solution of Cu into NbSn₂ and Nb₆Sn₅ destabilizes these intermediate phases [71]. This destabilization effectively increases the Sn chemical potential, further promoting the growth of the Nb₃Sn layer.

In the bronze-route process, the Sn chemical potential within Cu–Sn is lower than that in NbSn₂ and Nb₆Sn₅, leading to the development of Nb₃Sn while bypassing the formation of these intermediate phases. However, a reduced Sn chemical potential induces a pronounced Sn concentration gradient across the Nb₃Sn layer [15].

Reducing the dimensions of the diffusion couple provides an alternative strategy to promote Nb₃Sn phase formation. In the Nb/Al diffusion reaction, the energetically stable Nb₂Al typically develops at the reaction interface and functions as a diffusion barrier that inhibits Nb₃Al formation [72, 73]. Conversely, minimizing the multilayered Nb/Al diffusion pair to the nanoscale enables Nb₃Al formation via diffusion reactions at temperatures below 800 °C [72, 74]. This phenomenon results from the destabilization of the generated Nb₂Al as the reduction in the diffusion couple structure increases the surface energy of the phase. Following this mechanism, reducing the Nb/Sn(Cu) diffusion couple destabilizes intermediate compound phases such as NbSn₂, thereby enhancing Nb₃Sn phase formation.

During the Cu and Sn mixing stage, the formation of the NbCuSn compound nausite ((Nb_{0.75}Cu_{0.25})Sn₂) at approximately 400 °C influences the Nb₃Sn phase formation [61, 62, 75, 76].

Nausite forms at 360 °C – 550 °C [77], and significantly influences the mutual diffusivity of Cu and Sn, particularly during the Cu–Sn mixing stage [62]. Controlling nausite formation may contribute to improving the homogeneity of Sn distribution during the early stages of reaction. Since nausite subsequently decomposes into NbSn₂ and Nb₆Sn₅ [62], its formation may also influence the subsequent formation of Nb₃Sn at higher temperatures.

2.2. Nb₃Sn growth kinetics

Grain boundaries substantially affect both flux pinning and the Nb₃Sn layer growth rate. Crystal defects such as grain boundaries generally have low activation energy for atomic migration, facilitating the movement of atoms along grain boundaries, as compared to bulk diffusion. In the polycrystalline Nb₃Sn layer, a large amount of Sn diffuses to the reaction front through the Nb₃Sn grain boundaries. Molecular dynamics simulations have revealed that Sn grain boundary diffusion in Nb₃Sn has been estimated to be approximately 7–14 orders of magnitude faster than bulk diffusion over the temperature range relevant to Nb₃Sn reaction heat treatments (~923–1023 K) [78]. The difference decreases with increasing temperature because bulk diffusion generally exhibits a stronger temperature dependence than grain boundary diffusion. This indicates that during the Nb₃Sn layer growth process, once Nb₃Sn grains are formed, the Sn composition in the grain varies minimally via bulk diffusion, resulting in an essentially unchanged Sn concentration gradient across the Nb₃Sn layer [79, 80]. These findings suggest that enhancing the Sn diffusion driving force while simultaneously minimizing the Nb/Sn diffusion couple length is essential for maximizing the improvement in the Sn concentration gradient within the Nb₃Sn layer. This accounts for why RRP wire achieves a smaller Sn concentration gradient, as compared to the tube-type and PIT wires [15].

Instead of the concentration-gradient form of Fick's first law, the diffusion flux can be expressed in terms of the chemical-potential gradient as

$$J_i = -C_i M_i \frac{\partial \mu_i}{\partial x} \quad (1)$$

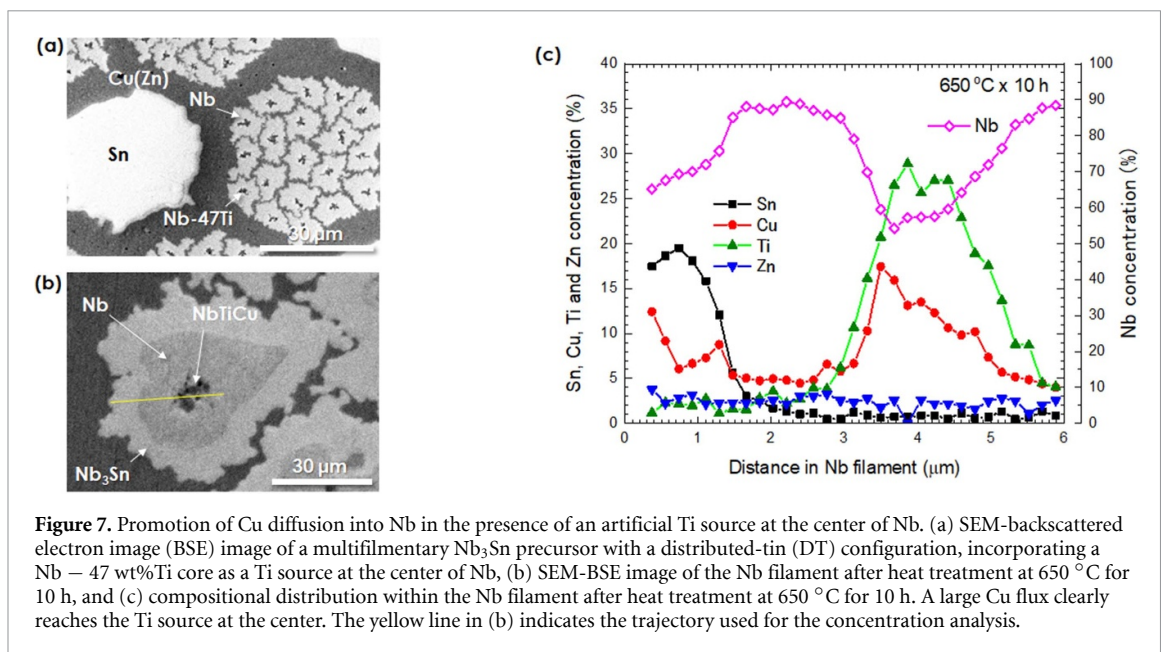
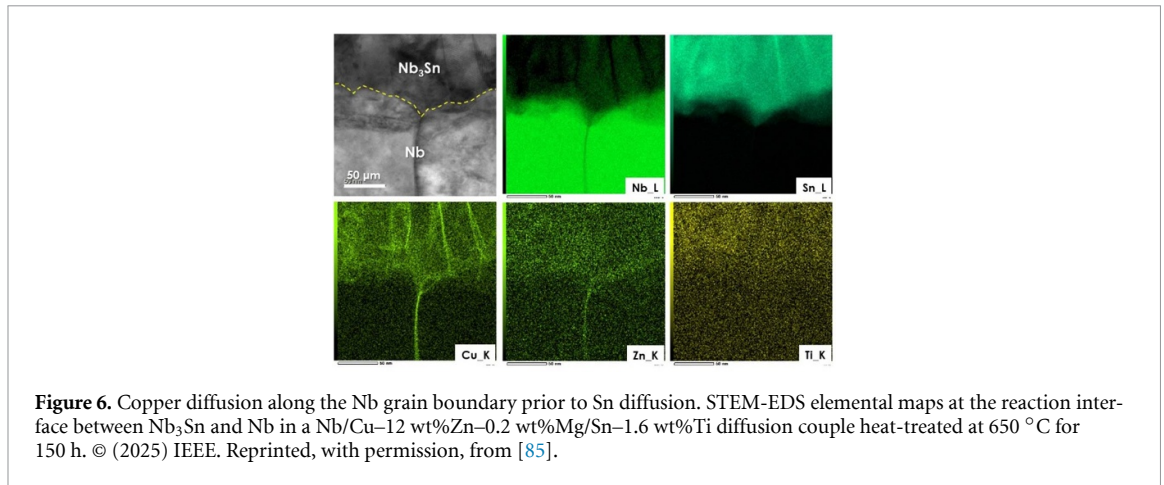
where J_i , C_i , M_i and μ_i denote the net flux, concentration, mobility, and chemical potential of component i , respectively [81]. This expression indicates that the diffusion flux is determined by both the chemical-potential gradient and the atomic mobility. Therefore, both an increased chemical-potential gradient and enhanced atomic mobility are expected to contribute to improved Sn transport and consequently to a more homogeneous Sn concentration profile within the Nb₃Sn layer.

2.3. Copper diffusion through Nb₃Sn and Nb grain boundaries

Previous elemental analyses of grain boundaries using atom probe tomography (APT) and scanning transmission electron microscopy (STEM) coupled with energy-dispersive x-ray spectroscopy (EDS) have revealed the presence of Cu at the grain boundaries after the reaction [68, 82–84]. This suggests that Cu also migrates towards the reaction front along with Sn through Nb₃Sn grain boundaries, thereby influencing Nb₃Sn formation at the reaction interface. However, the precise stage at which Cu diffuses remains a fundamental question, as a limited number of studies have thoroughly investigated Cu diffusion.

STEM-EDS analysis at the reaction interface between the Nb and Nb₃Sn layers clearly reveals the migration of Cu. Figure 6 shows a STEM-EDS elemental map of the reaction front between Nb and Nb₃Sn in a Nb/Cu–12 wt%Zn–0.2 wt%Mg/Sn–1.6 wt%Ti diffusion couple heat-treated at 650 °C for 150 h [85]. These results demonstrate that Cu preferentially diffuses along Nb grain boundaries upon reaching the reaction front, followed by Sn diffusion. This observation supports the hypothesis that the prior presence of Cu at the reaction front reduces the chemical potential of the subsequently arriving Sn, thereby suppressing Nb₂Sn and Nb₆Sn₅ formation while promoting Nb₃Sn nucleation. These findings provide valuable insights into Cu diffusion within the Nb₃Sn layer. Regarding the grain boundary diffusion of Sn and Cu, the Sn chemical potential at the Nb₃Sn grain boundary is schematically represented by the dotted line in figure 4.

Furthermore, in Nb filament structures where a Ti source, such as Nb – 47 wt%Ti, is positioned at the Nb center (figure 7(a)), Cu diffusivity increases due to mutual diffusion with Ti. Figures 7(b) and (c) show SEM images of the Nb filaments and the corresponding compositional profiles after heat treatment at 650 °C for 10 h. Elevated Cu concentrations were detected within the NbTi core (details provided in section 3.6.), indicating that a large amount of Cu reaches the NbTi core more rapidly than the Sn diffusion rate permits. Notably, Cu was detected by EDS in the NbTi core even after heat treatment at 550 °C for 100 h. Copper and Ti reportedly undergo mutual diffusion in Cu-based NbTi wires during heat treatment at approximately 650 °C [86, 87]. This phenomenon suggests that Ti diffuses efficiently



within the Nb module, even in cross-sectional architectures such as RRP wires, where NbTi filaments are dispersed as the Ti source [88].

Why is Cu so special? The significance of Cu in Nb₃Sn layer formation is attributed to two primary characteristics:

1. Copper exhibits negligible solubility in Nb and
2. The solubility of Sn in Cu, including the formation of Cu–Sn compound phases, moderately adjusts the Sn chemical potential.

In addition to Cu, Ag also exhibits a comparable effect on phase formation behavior, as observed in the bronze method [89, 90]. Although an equilibrium phase diagram for the Nb–Ag system is currently unavailable, investigations into equilibrium reactions and Ag-matrix Nb₃Al wires indicate that Nb and Ag exhibit negligible mutual solid solubility [91, 92], suggesting that the Nb–Ag phase diagram is similar to that of the Nb–Cu system.

Cu diffusivity may depend on the chemical potential of Cu in the surrounding Cu–Sn matrix. Because the thermodynamic state of Cu differs among bronze-route, PIT, and internal-tin conductors, the driving force for Cu diffusion may also vary with conductor architecture. For example, the Cu-rich matrix in bronze-route conductors may provide a different chemical environment from that in PIT or internal-tin conductors. Such differences could influence the extent of Cu diffusion into the Nb₃Sn layer and thereby affect subsequent phase formation and microstructural evolution.

2.4. Nucleation and grain growth: grain refinement and formation of APCs

Nb₃Sn phase formation via diffusion reactions may be characterized as a thermodynamic nucleation process, wherein Nb₃Sn nuclei precipitate within the Nb matrix at the reaction front. In regimes dominated by grain boundary diffusion, refining the Nb₃Sn grain structure increases the Sn diffusion flux. This enhanced Sn transport accelerates growth kinetics.

Nucleation theory facilitates the understanding of Nb₃Sn formation and the precipitation of nanoparticles that serve as artificial flux pinning centers in superconducting materials, including Nb₃Sn and REBCO [29, 93–97]. In contrast to REBCO conductors, where APCs are widely utilized, flux pinning in conventional Nb₃Sn conductors is generally dominated by grain boundaries. Therefore, grain refinement remains one of the most effective approaches for enhancing J_c . Recent APC and internal-oxidation approaches have shown promising improvements in conductor performance; however, the extent to which oxide nanoparticles directly contribute to flux pinning remains under active investigation [98].

Generally, the magnetic flux pinning force is maximized when precipitate dimensions are commensurate with the coherence length. Furthermore, the optimal grain size is considered comparable to the magnetic flux lattice spacing, as this condition maximizes the magnetic flux pinning force. Assuming a square magnetic flux lattice [99–101], the lattice spacing is defined as $a = \sqrt{(\varphi_0/B)}$, where φ_0 is the magnetic flux quantum and B is the external magnetic flux density. For example, as a guideline, at $B = 5, 12$, and 16 T, a is estimated at approximately 20, 13, and 11 nm, respectively. Godeke correlated the maximum pinning force with the reciprocal grain size, demonstrating a universal, monotonic increase in the maximum pinning force down to a grain size of 35 nm [11].

Classic nucleation theory posits that nucleation occurs when the reduction in volume energy during phase precipitation exceeds the corresponding increase in surface energy [102]. The Gibbs energy change (ΔG) is expressed as $\Delta G = (-4\pi r^3/3)(\Delta g/v) + 4\pi r^2\sigma$, where, Δg , r , v and σ represent the molar free-energy change (i.e. the chemical potential difference driving phase formation), precipitate radius, precipitate molar volume, and surface energy density, respectively. The first term on the right-hand side of the equation represents the volume free energy, while the second term accounts for the surface free energy. This relationship yields a concave energy curve with a peak value (ΔG_c) occurring at the critical nucleus radius, $r_c = 2\sigma v/\Delta g$.

Although the classical homogeneous-nucleation model provides a useful framework for understanding the role of interfacial energy in nucleation, Nb₃Sn formation in practical conductors generally proceeds through heterogeneous nucleation at existing interfaces. Furthermore, the subsequent phase evolution and layer growth are largely governed by diffusion processes rather than by nucleation kinetics alone. Meanwhile, for oxide precipitation associated with internal oxidation, the thermodynamic driving force originates primarily from the corresponding chemical reactions, although interfacial energy remains an important factor governing nucleation.

As Δg increases, the critical radius decreases and the nucleation rate increases, leading to grain refinement. The chemical potential difference is influenced by the internal strain state and crystal defects, such as dislocations and grain boundaries, within the parent phase prior to precipitation, which serve as preferential nucleation sites. Hafnium additions to Nb exemplifies this effect. Figure 8 shows micrographs of the Nb₃Sn phase formed from a Nb–4at%Ta–1at%Hf matrix in the as-deformed condition and after preheating at 1000 °C [15, 103]. The recrystallization temperature of Hf-doped Nb exceeds the typical Nb₃Sn formation temperature of approximately 650 °C, allowing the retention of a fine microstructure during Nb₃Sn formation. Consequently, the Nb–Hf system accumulates internal strain energy, which provides an additional driving force for Nb₃Sn phase formation and subsequent grain refinement [103–106].

The temperature dependence of the nucleation rate follows an Arrhenius-type relationship, where the rate increases exponentially with temperature once the minimum activation energy threshold is surpassed [107, 108]. While elevated temperatures enhance diffusion rates and compositional uniformity, in Nb₃Sn layer formation dominated by grain-boundary diffusion, compositional homogenization within the grains is negligible over relevant timescales. This activation energy concept also governs grain growth, consistent with previous reports indicating that grain size varies exponentially with temperature [93, 109]. As reference data, the grain sizes reported for tube-type Nb₃Sn wires using Nb – 7.5 wt%Ta are approximately 80, 110, 140, and 180 nm at 615 °C, 650 °C, 700 °C, and 750 °C, respectively [93, 109]. This relationship is approximately expressed as $d_{GS} = 0.5918e^{0.0056T}$, where d_{GS} is the grain size (nm) and T is the temperature (K). Thus, balancing layer growth and grain growth through optimized heat treatment is therefore vital for enhancing the critical current density of Nb₃Sn.

The Zener pinning effect also significantly influences the formation of Nb₃Sn polycrystalline layers [110]. This effect has recently garnered significant attention, particularly in the Nb internal oxidation

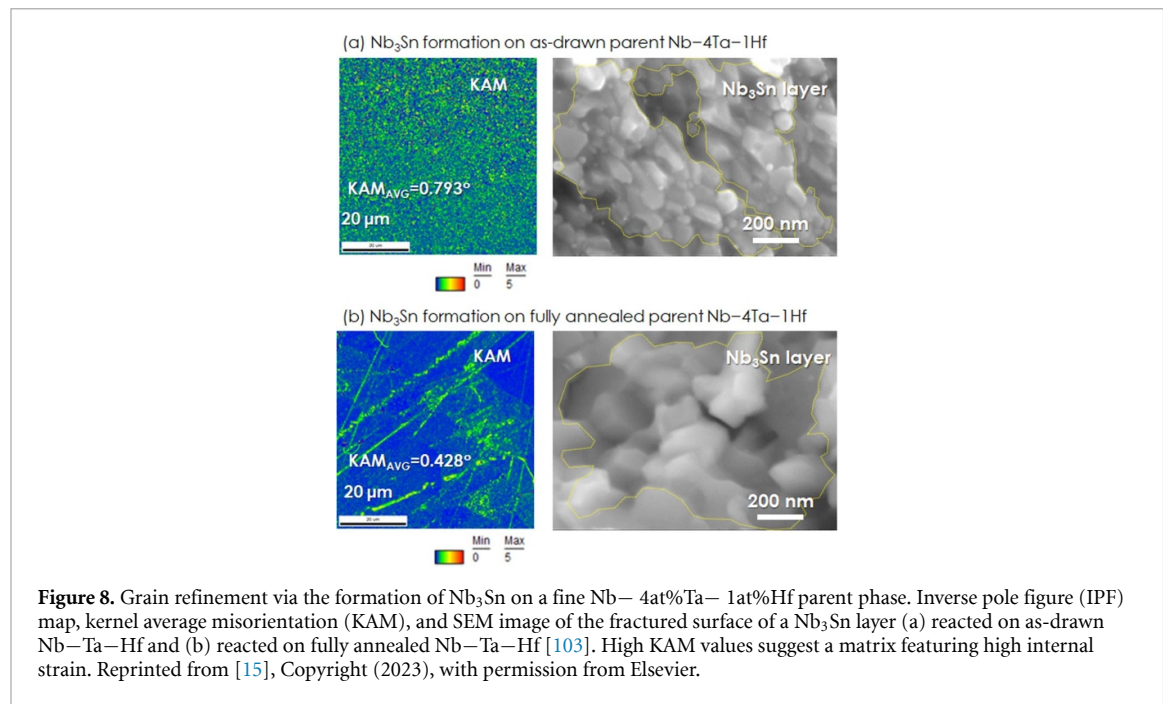


Figure 8. Grain refinement via the formation of Nb₃Sn on a fine Nb–4at%Ta–1at%Hf parent phase. Inverse pole figure (IPF) map, kernel average misorientation (KAM), and SEM image of the fractured surface of a Nb₃Sn layer (a) reacted on as-drawn Nb–Ta–Hf and (b) reacted on fully annealed Nb–Ta–Hf [103]. High KAM values suggest a matrix featuring high internal strain. Reprinted from [15], Copyright (2023), with permission from Elsevier.

technique for Nb₃Sn [29, 93, 106]. This effect occurs when finely dispersed, energetically stable second-phase particles within the reaction layer suppress grain boundary migration, thereby inhibiting grain coarsening (grain growth). The empirical relationship can be expressed as $R \cong 4/3 \cdot r/f$, where R is the final grain size, f is the fraction of the total volume occupied by inclusions, and r is the size of the second-phase particles [111–113]. Precipitates that induce this effect are not limited to oxides. Santra reported the existence of β -(Ti, Nb) particles at grain boundaries and noted that these particles induce the Zener pinning effect [84]. Similarly, the solute drag effect, characterized by the segregation (or concentration) of specific solute elements at grain boundaries, further hinders grain boundary migration [114–116].

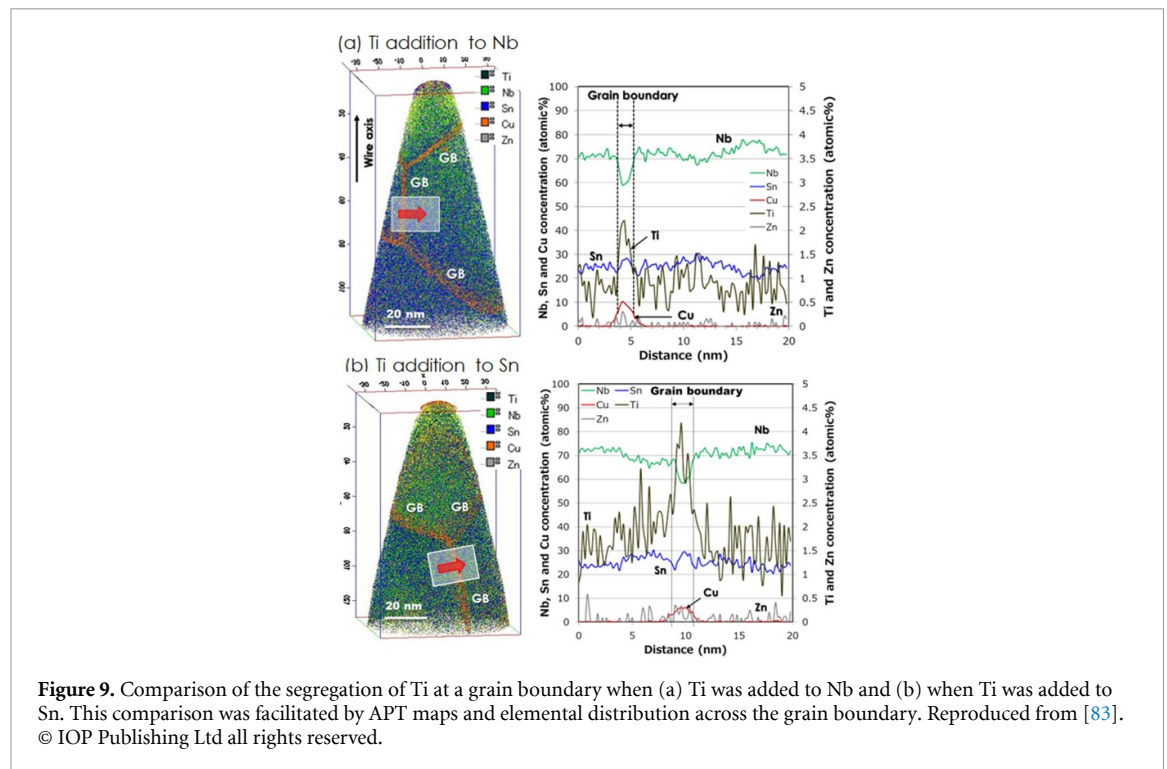
3. Effect of elemental additions

During the formation of Nb₃Sn layers via solid-state diffusion, elemental additions influence polycrystalline Nb₃Sn layer formation and the overall phase formation, including that of the matrix materials surrounding the superconducting filaments. Based on the fundamental principles of thermodynamics and growth kinetics outlined above, this section discusses the effects of elemental additions on the formation of Nb₃Sn layers via complex diffusion reactions using several examples and incorporating our recent research results.

The internal oxidation approach reported by Xu *et al* is also relevant to the present discussion of elemental-addition strategies because it involves the addition of oxygen-gettering elements such as Zr or Hf to Nb [106]. Therefore, a brief overview of its underlying mechanism is provided here. In this approach, nanoscale oxide precipitates (e.g. ZrO₂ or HfO₂) formed during internal oxidation suppress grain growth through the Zener pinning effect, resulting in significant refinement of the Nb₃Sn grain structure [13, 29, 93, 106, 109, 117]. In addition, these oxide nanoparticles have been reported to provide an additional contribution to flux pinning. Detailed descriptions of the internal oxidation approach and APC conductors can be found in the review by Xu *et al* [13, 118, 119]. The present review therefore focuses primarily on the thermodynamics and growth kinetics through which elemental additions influence phase formation and microstructural evolution.

3.1. Variation in the Nb₃Sn phase formation behavior depending on the Ti addition site

Ti and Ta are widely recognized as additive elements that improve B_{c2} in Nb₃Sn [17, 120, 121]. At 4.2 K, Ti or Ta doping increases B_{c2} by 3 and 4 T relative to undoped samples, reaching approximately 27 T. Within the dirty-limit ($l \ll \xi_0$, where l is the electron mean free path and ξ_0 is the BCS coherence length) approximation of the Ginzburg–Landau–Abrikosov–Gor’kov theory [122–124], $B_{c2}(0)$ can be expressed as $B_{c2}(0) = 3.11 \times 103 \rho_n \gamma T_c$, where ρ_n is the residual resistivity of the normal state, γ is the



electronic specific heat coefficient, and T_c is the critical temperature. The disproportionately large change in B_{c2} , as compared to T_c , is attributed to a significant increase in normal-state resistance caused by the shortening of the mean free path resulting from increased electron scattering [16, 125–127]. The optimal Ti addition amount is approximately 1.5–2 at%, whereas Ta requires approximately double the concentration of Ti to achieve a comparable effect. The extended x-ray absorption fine structure findings by Tarantini, which indicate that Ti preferentially substitutes at Nb sites more readily than Ta [88], may explain this discrepancy. APT analyses have further revealed that Ti does not form a complete solid solution within the Nb_3Sn phase; instead, trace amounts of Ti segregate at the grain boundaries [82, 83].

The site of the elemental additions in the precursor wires is a critical consideration for Nb_3Sn layer formation via diffusion reactions. In the internal-Sn process, Ti dissolves into the Nb_3Sn phase regardless of whether it is added to a Nb, Cu matrix or Sn core. Conversely, Ta exhibits minimal dissolution, except when added to Nb. This behavior is attributed to the lower diffusion driving force of Ta in the Sn–Ta phase.

Figure 9 compares the elemental concentration distributions at grain boundaries obtained via APT for Ti doping with Nb and Ti doping within Sn cores [83]. The addition of Ti to the Sn core results in higher Ti concentrations. Santra reported the presence of β -(Ti, Nb) at the grain boundaries in bronze-processed wires, noting that the amount of β -(Ti, Nb) is likely higher when Ti is doped into the Cu–Sn bronze than when it is added to Nb [84]. Santra further highlighted the contribution of β -(Ti, Nb) to grain refinement via the Zener pinning effect. Popova reported the presence of fine Ti_6Sn_5 particles within Nb_3Sn grains when Ti doped to Nb, which may purify the grain boundaries and induce local grain coarsening near these particles [128]. Thus, the site of Ti doping can influence both the Nb_3Sn grain size and Sn diffusivity within the Nb_3Sn layer.

In the internal Sn method, the site of Ti addition significantly influences the interfacial reaction. The addition of Ti to the Sn cores represents the simplest case and is employed in JASTEC and KAT wires [25, 31]. When Ti is added to Sn cores, a distinct multinary Nb–Sn–Cu–Ti compound layer develops at the interface [129].

Figure 10 shows micrographs comparing the interfacial reaction behavior at 685 °C when Ti is added to a Sn core, the Cu intermediate layer, or the Nb matrix within a Nb/Cu/Sn single-core wire [129]. When Ti is added to Nb, no compound layer is observed. However, adding Ti to either the Sn core or Cu layer results in the formation of a distinct Ti-containing compound layer. Recent STEM-EDS analyses of Nb/Cu–15Zn/Sn–1.6Ti diffusion-couple samples indicated that the composition of this compound corresponds to the values listed in table 2 [85]. Considering only the Nb:Sn ratio, the compound appears stoichiometrically similar to Nb_6Sn_5 . This quaternary compound forms at approximately 500 °C

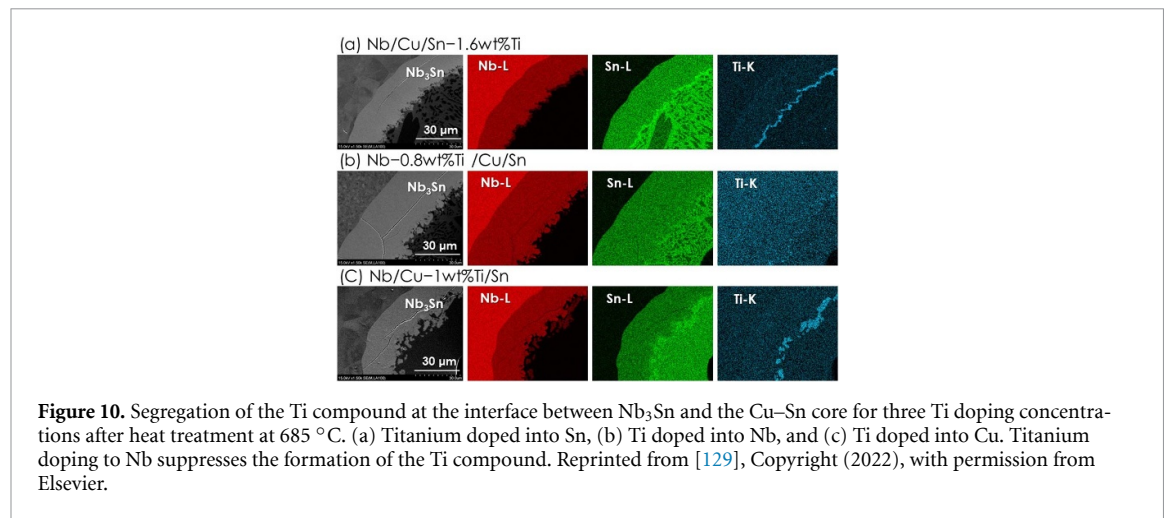


Table 2. Composition of the NbSnCuTi phase analyzed using STEM-EDS (at%).

Nb	Sn	Cu(Zn)	Ti
38.5	33.6	13.2	14.7

and decomposes at approximately 685 °C. Consequently, this quaternary compound clearly differs from nausite in both composition and formation temperature [61, 75, 77].

Within the Cu–Sn–Ti reaction system, a liquid phase and CuSnTi compound coexist at 572 °C [130]. At the Nb interface, the liquid phase facilitates Nb dissolution, which initiates the formation of NbSnCuTi compound phases. The deduced Sn chemical potential within this phase subsequently reduces the Sn diffusion driving force. As shown in figure 10, the thickness of the Nb₃Sn layer formed in the Sn–Ti core sample decreased to approximately two-thirds of the thickness observed in the Nb–Ti sheath sample, suggesting that the NbSnCuTi compound phase functions as a diffusion barrier for Sn. In contrast to the bronze process, the internal-tin method induces grain refinement in Ti-added Nb [85]. Furthermore, in multifilamentary wires, this compound phase persists as a diffusion barrier between Nb filaments within the Nb module, which serve as Sn diffusion pathways, thereby inhibiting the diffusion of both Sn and Ti to the module center. This results in compositional inhomogeneity within the final Nb₃Sn filament [25, 131].

3.2. Optimization of Ti doping position

From the perspective of Nb₃Sn layer formation, the internal-Sn method is optimized by locating the Ti source within the Nb module, a configuration currently implemented in RRP wires. In our laboratory-scale fabrications, a multifilamentary wire employing Nb alloy filaments containing 0.8 wt. % Ti exhibited an approximately 30% improvement in non-Cu J_c at 16 T, as compared with Ti additions to the Sn core (figure 11) [83]. Although these results were obtained from laboratory-scale samples, Ti addition to Nb represents a significant potential for further enhancing superconducting performance.

Furthermore, strategic placement of Ti sources within the Nb module may be achieved by substituting some Nb rods with NbTi rods, as demonstrated in the RRP design, or by artificially incorporating a Ti source directly into the Nb filaments, as illustrated by the structure shown in figure 7. This effect will be discussed in detail in section 3.6.

3.3. Enhancement of growth kinetics by Zn addition

Among various additive elements [132], Zn addition offers several unique advantages for microstructural modification not found in other elements, as follows:

1. During the Cu/Sn mixing process, β -CuZn or γ -CuZn phases form at the interface, which suppresses Cu diffusion into Sn and inhibits the formation of Kirkendall voids [83, 133].
2. In the Nb/Cu–Zn–Sn diffusion process, the Sn chemical potential increases, which promotes the growth of the Nb₃Sn layer [85, 134]

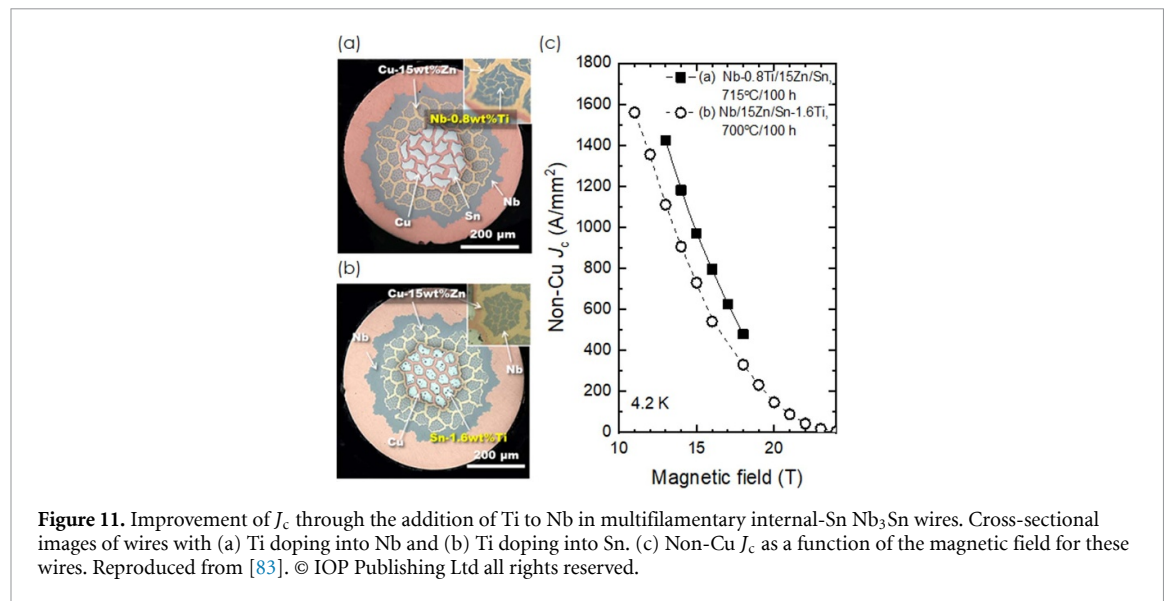


Figure 11. Improvement of J_c through the addition of Ti to Nb in multifilamentary internal-Sn Nb₃Sn wires. Cross-sectional images of wires with (a) Ti doping into Nb and (b) Ti doping into Sn. (c) Non-Cu J_c as a function of the magnetic field for these wires. Reproduced from [83]. © IOP Publishing Ltd all rights reserved.

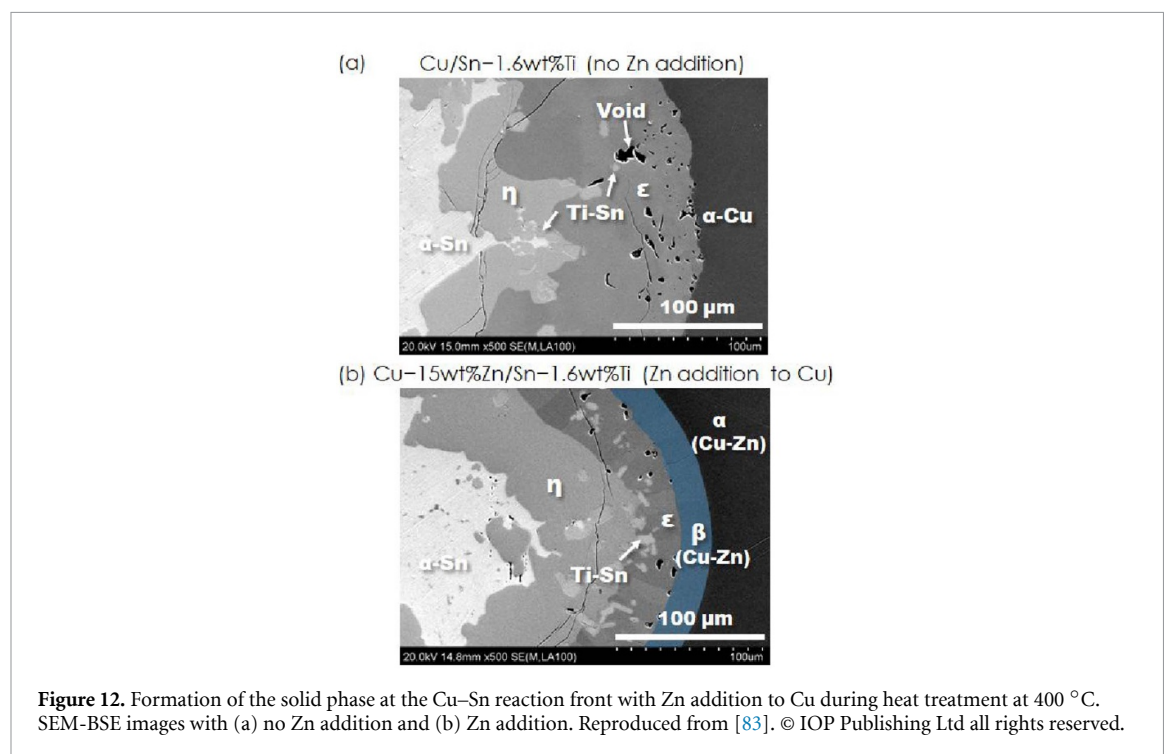
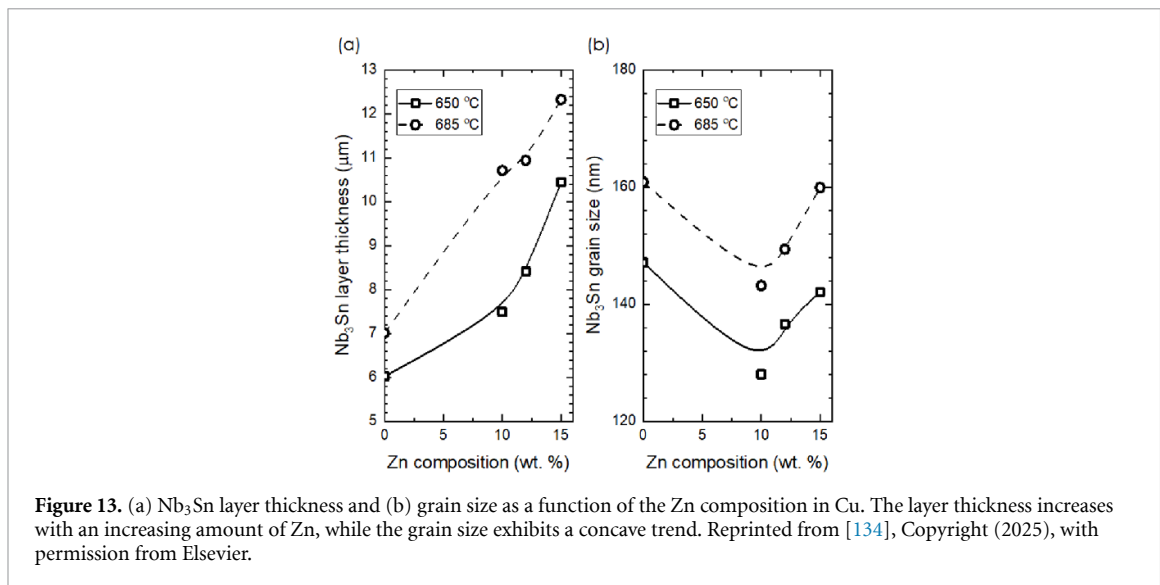


Figure 12. Formation of the solid phase at the Cu-Sn reaction front with Zn addition to Cu during heat treatment at 400 °C. SEM-BSE images with (a) no Zn addition and (b) Zn addition. Reproduced from [83]. © IOP Publishing Ltd all rights reserved.

3. Zinc remains primarily within the Cu matrix with minimal diffusion into the Nb₃Sn layer, resulting in solid-solution strengthening of the Cu matrix [45, 135, 136].

Figure 12 shows a micrograph illustrating the effects of Zn addition at the reaction interface after heat treatment at 400 °C in a Cu/Sn single-core structure [83]. Generally, during mutual solid-state diffusion in the Cu/Sn system, the faster diffusion of Cu in Cu-Sn induces the Kirkendall effect, resulting in interface migration and void formation [137]. Moreover, the formation of Cu-Sn compounds induces volume contraction, which exacerbates void development. As shown in figure 12(a), ϵ -Cu₃Sn forms at the reaction front between pure Cu and Sn at 400 °C, accompanied by large voids; the volume contraction associated with ϵ -phase formation is estimated to be approximately 5.3% based on density variations. Previous studies reported that at 500 °C, the δ phase predominates and large voids persist [138]. These voids can act as physical barriers to Sn diffusion, particularly in Nb modules with architectures comprising numerous fine filaments embedded in the matrix, such as the ultra-fine multifilamentary assemblies used in commercial wires. Conversely, the addition of Zn to Cu or Sn results in the formation of a dense CuZn phase at the interface without voids (figure 12(b)) [83, 133].



3.4. Variation in the pinning force dependent on elemental concentration at the grain boundary

Recent research regarding the optimization of Zn addition have yielded intriguing results, indicating that Zn addition influences both Nb_3Sn layer formation and the elemental pinning force at grain boundaries [134]. An overview of these results is provided below. The experimental samples consisted of single-core wires with a Nb/Cu/Sn diffusion-couple structure, where 10–15 wt. % Zn was added to the Cu intermediate layer. A wire featuring a pure Cu intermediate layer was also prepared as a reference. To eliminate the formation of unnecessary compound layers and isolate the effect of the Zn addition, Ti was not introduced. The sample designations were:

1. N-10Z-S: Nb/Cu-10 wt%Zn/Sn,
2. N-12Z-S: Nb/Cu-12 wt%Zn/Sn,
3. N-15Z-S: Nb/Cu-15 wt%Zn/Sn, and
4. N-C-S: Nb/Cu/Sn.

For BSE cross-sectional images at the interface following heat treatment at 650 °C, the reader is referred to [134]. Figure 13(a) shows the dependence of the Nb_3Sn layer thickness on the Zn content, as obtained through image analysis. The Nb_3Sn layer thickness increased monotonically with an increasing Zn addition, suggesting that Zn enhances the Sn diffusion driving force. Conversely, the Nb_3Sn grain size, as shown in figure 13(b), did not monotonically change with an increasing Zn addition; instead, it exhibited a convex trend with a minimum observed at 10 wt. %.

Figures 14(a) and (b) show the layer J_c and flux pinning characteristics per superconducting volume, respectively, derived from the magnetization curves measured using a vibrating sample magnetometer and analyzed using the critical state model [134, 139, 140]. As the Zn content increases, B_{c2} tends to increase, resulting in an enhanced J_c and pinning force under high magnetic fields. A particularly significant improvement in high-field J_c characteristics occurs at 15 wt. % Zn. Microstructural observation of the interface confirmed that Nb_6Sn_5 formed during the reaction only in the 15 wt. % Zn sample. The formation of Nb_6Sn_5 , as detailed in section 2.1, suggests an increased Sn diffusion driving force; consequently, the 15 wt. % Zn concentration represents a threshold at which the reaction behavior changes rapidly. Conversely, the maximum pinning force exhibited a monotonic decrease with an increasing Zn content (figure 14(b)). When compared to the convex relationship between the grain size and Zn concentration shown in figure 13(b), these results indicate that the pinning characteristics do not correlate directly with grain boundary density. This suggests that the elemental pinning force at grain boundaries is influenced by additional factors.

Examination of the Sn concentration ratio distribution within the Nb_3Sn layer (figure 15(a)) [134] shows that the Sn concentration approaches stoichiometric compositions with an increasing Zn content. This stoichiometric progression may account for the observed improvement in the high-field J_c characteristics at higher Zn contents. Conversely, the average Cu concentration decreased at 10 wt. % Zn and subsequently increased with further Zn additions, as shown in figure 15(b) [134]. Figure 16

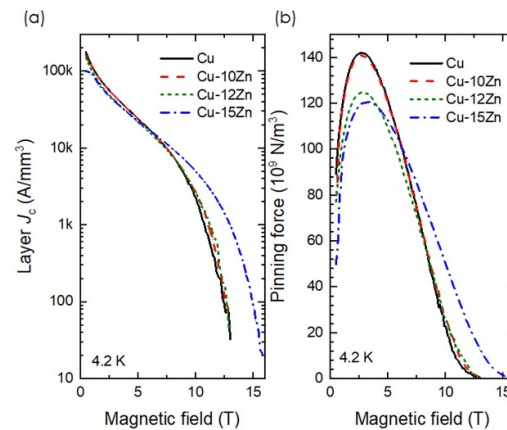


Figure 14. (a) Layer J_c and (b) pinning force per superconducting volume as a function of the magnetic field with respect to the Zn composition. The high-field layer increases with an increasing amount of Zn, while the maximum pinning force decreases. The pinning force values reported in [134], contain a scaling error due to a coefficient error in the calculation; the values shown in the present figure have been corrected accordingly. The relative trends and overall conclusions are unaffected. Reprinted from [134], Copyright (2025), with permission from Elsevier.

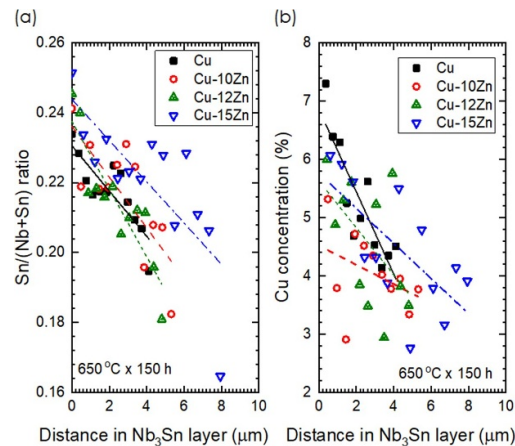
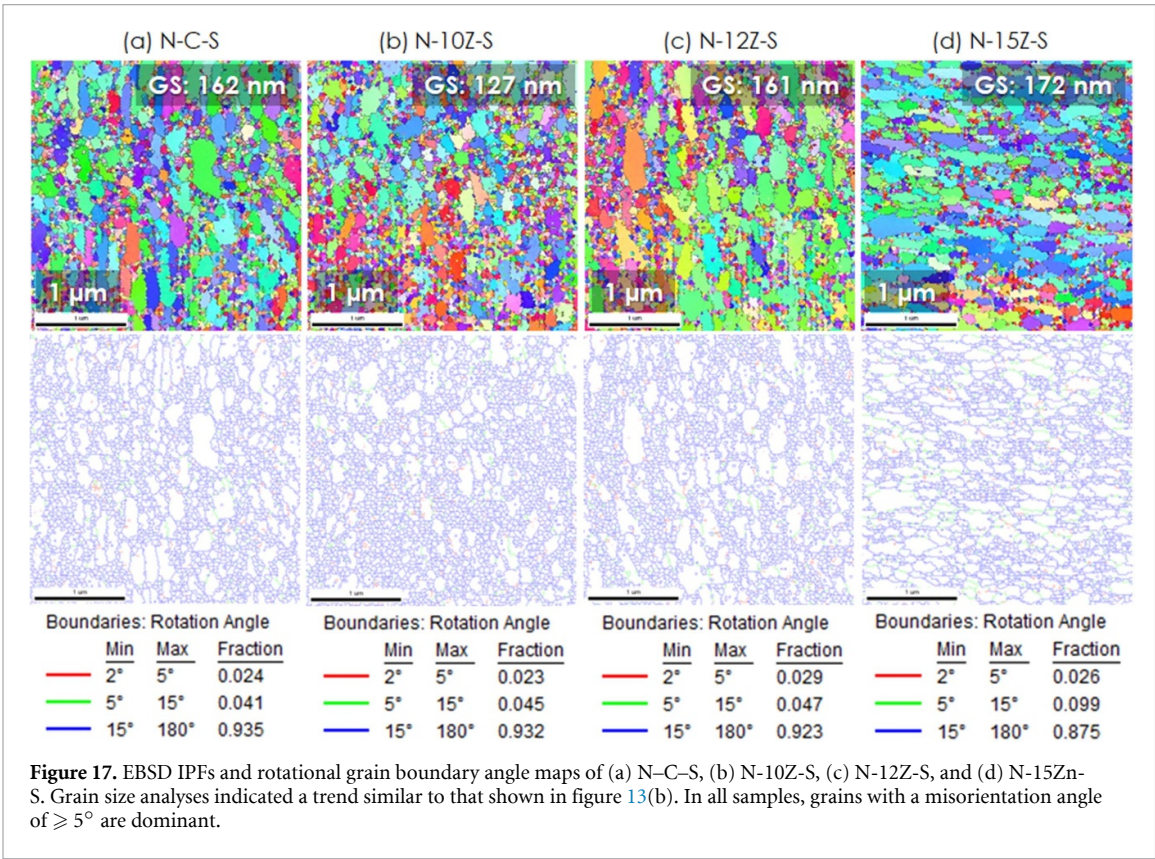
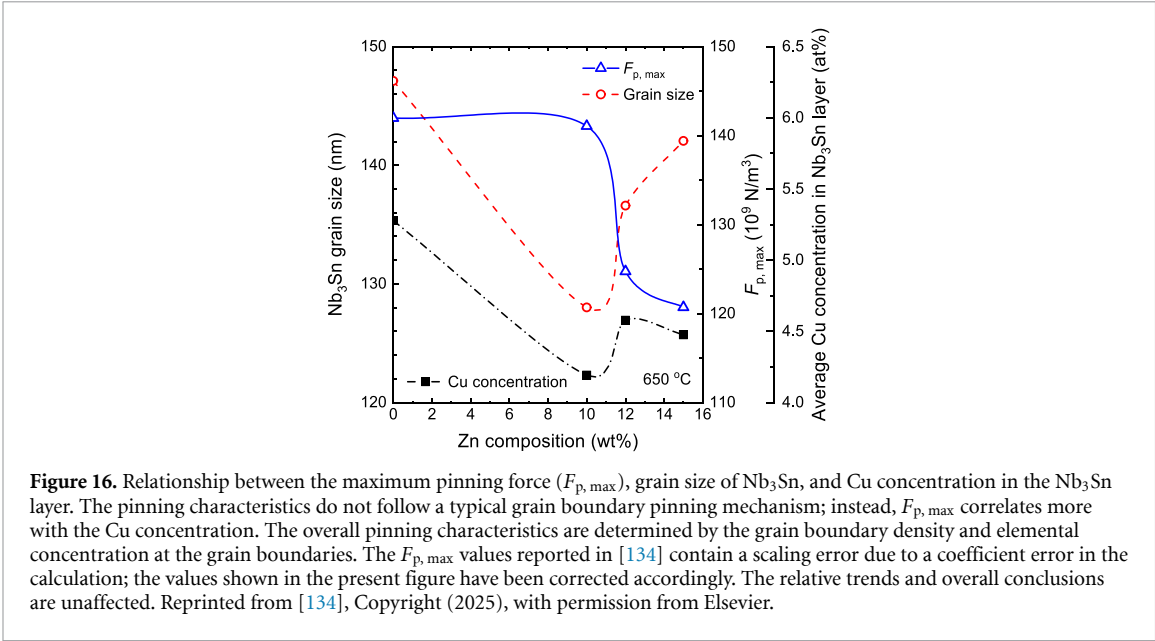


Figure 15. (a) Tin concentration ratio and Cu concentration distribution in the Nb_3Sn layer [134]. The Sn concentration increases with an increasing amount of Zn, while the Cu concentration does not exhibit a linear trend. Reprinted from [134], Copyright (2025), with permission from Elsevier.

summarizes the dependence of the maximum magnetic flux pinning force on both the grain size and Cu concentration [134]. The relationship between grain size and Zn content, obtained from electron backscatter diffraction (EBSD) analyses, also exhibits convex characteristics, as further detailed in figure 17. These results clearly indicate that the decrease in maximum flux pinning force with an increasing Zn addition correlates more significantly with Cu concentration than with grain size. APT [82–84] and STEM-EDS results [85] indicated that Cu preferentially segregates at grain boundaries. Therefore, an increase in the average Cu concentration within the Nb_3Sn layer suggests a corresponding enrichment of Cu at the grain boundaries. Based on these results, it is hypothesized that the elemental pinning force at the grain boundaries is dependent on the Cu concentration at the grain boundaries.

Grain boundary misorientation is another factor affecting the elemental pinning force at grain boundaries. Figure 17 shows the IPF maps and rotational grain boundary angle maps of the Nb_3Sn layer on each sample obtained via EBSD. Cross-sectional specimens were prepared for the EBSD analysis through mechanical polishing using a polycrystalline diamond slurry, followed by mechanochemical polishing using colloidal silica. Although the grain size approaches the resolution limit of the EBSD equipment (approximately 50 nm), which makes complete noise removal difficult, the overall trend indicates that all samples were dominated by high-angle grain boundaries with misorientation angles of approximately 15° , and no significant differences were observed among the samples. Accordingly, the contribution of grain boundary misorientation to the variation in pinning force can be excluded. These results indicate that the anomalous elemental pinning force characteristics are primarily influenced by element concentrations at the grain boundaries.



In conclusion, the overall magnetic flux pinning force is governed by two synergistic factors, namely, grain boundary density and the elemental concentration at those boundaries. As shown in figure 16, the Cu concentration increases when the Zn addition exceeds 10 wt. %, while the maximum pinning force simultaneously decreases. This behavior likely reflects the dominant influence of grain coarsening at elevated Zn concentrations.

Figure 16 further suggests a strong correlation between grain size and Cu concentration, where larger grain sizes occur at higher Cu concentrations. As discussed earlier, Cu diffuses along the Nb_3Sn grain boundaries alongside Sn toward the reaction front, where Nb_3Sn subsequently precipitates. The Cu concentration at the grain boundaries is expected to influence the localized Sn chemical potential. An enrichment in Cu reduces the Sn diffusion driving force, which subsequently increases the critical radius of Nb_3Sn nuclei. These experimental results demonstrate that Zn addition modifies the Cu concentration

at the grain boundaries and serves a crucial role in both grain boundary pinning and regulation of the Nb₃Sn grain morphology.

Previous studies have demonstrated that elemental segregation at grain boundaries affects both the grain size and magnetic flux pinning force [84, 128, 141]. However, to the best of our knowledge, few studies have comprehensively examined the specific influence of elemental concentration. It remains possible that the Ti concentration at Nb₃Sn grain boundaries, as shown in figure 9, affects the elemental pinning force at grain boundaries. While grain refinement is traditionally the guideline for improving J_c , these results provide new insights indicating that the elemental composition at grain boundaries also influences the J_c characteristics. Further detailed investigations are required to elucidate this mechanism.

3.5. Multiple elemental additions

Multiple elemental additions do not necessarily result in a simple superposition of their effects. For example, Mg is reportedly effective for grain refinement [142–144]. Wu employed high-resolution Auger spectroscopy to analyze bronze-processed Nb₃Sn wires and reported that Mg preferentially diffuses into Nb₃Sn grains rather than segregating at grain boundaries [145], although the underlying refinement mechanism has not yet been fully elucidated. Conversely, within the internal-Sn process, the addition of Ti to the Sn core may suppress the grain-refinement effect of Mg in specific instances [85]. Our group reported [85] that heat treatment of a single-core wire with a diffusion couple, comprising a Nb filament, a Cu intermediate layer co-added with 12 wt. % Zn and 0.2 wt. % Mg, and a Sn core doped with 1.6 wt. % Ti, at 650 °C resulted in the formation of a highly stable multinary compound phase at the interface. Magnesium was entrapped within this compound phase, thereby preventing its contribution to grain refinement. Similarly, although Ge doping in bronze-route Nb₃Sn wires reportedly induces Nb₃Sn grain refinement [146, 147], in the internal Sn process, the addition of Ti to the Sn core reportedly leads to the formation of a Ge–Ti-rich multinary compound phase at the interface [132]. To avoid such effects, it is necessary to carefully determine the additive location by considering the Gibbs energies of the resulting phases, thereby ensuring that no stable, undesirable compound phases are formed.

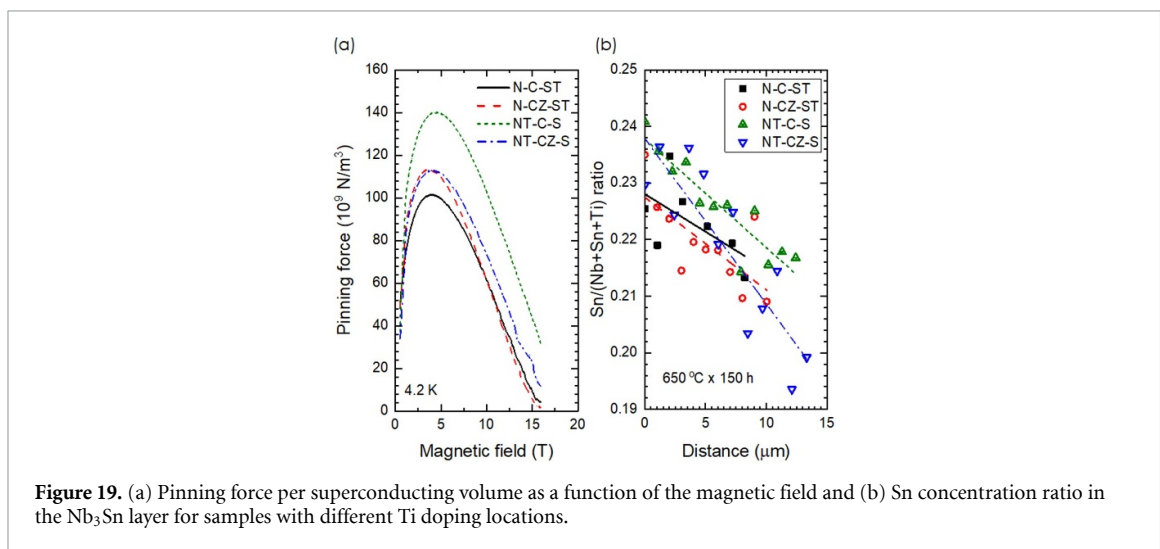
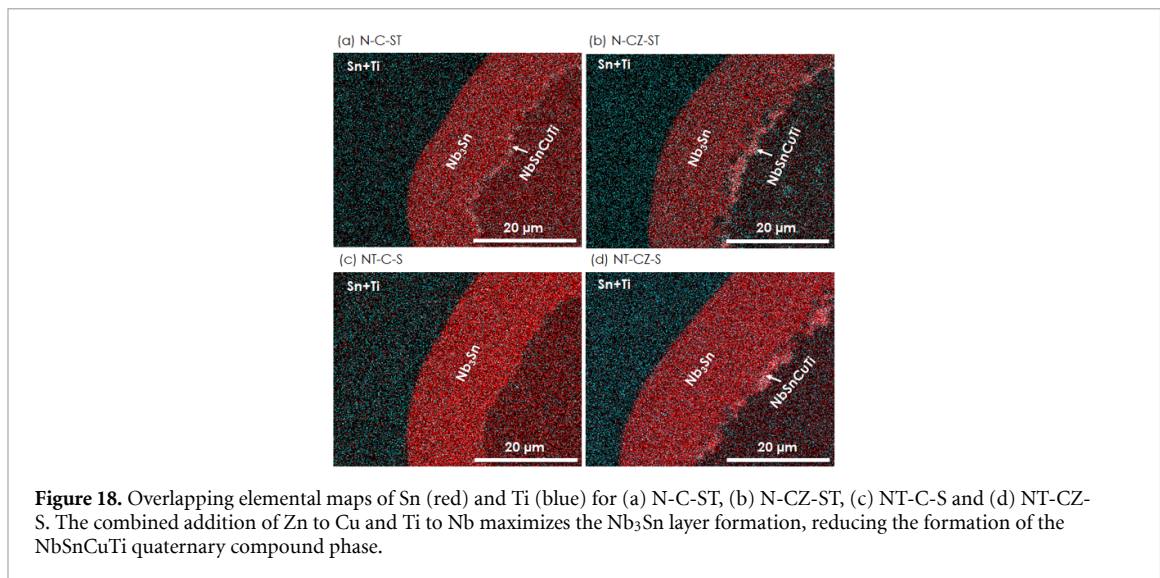
This section discusses the effective co-addition of Zn and Ti. Based on the preceding discussion, the combined addition of Zn to Sn and addition of Ti to Nb appears to be an optimal strategy for suppressing interfacial compound phase formation while maximizing Nb₃Sn layer formation. Accordingly, the effect of this co-addition was demonstrated using a diffusion-couple structure consisting of Nb – 0.8 wt%Ti, Cu – 15 wt%Zn and Sn (NT-CZ-S). The following four single-core samples, including a reference sample [85], were prepared:

1. N-C-ST: Nb/Cu /Sn – 1.6 wt%Ti,
2. N-CZ-ST: Nb/Cu – 15 wt%Zn/Sn – 1.6 wt%Ti,
3. NT-C-S: Nb – 0.8 wt%Ti/Cu /Sn, and
4. NT-CZ-S: Nb – 0.8 wt%Ti /Cu – 15 wt%Zn/Sn.

The wires had a diameter of 0.92 mm and consisted of seven modules, each containing a single Nb/Cu/Sn diffusion couple. The Sn/(Cu + Zn + Sn) compositional ratio, an important parameter governing the driving force for Sn diffusion, was designed to be approximately 26 at% to avoid the formation of Cu–Nb–Sn phases at the diffusion interface [148, 149]. In addition, the Nb layer was intentionally made sufficiently thick to prevent complete reaction of the Nb during heat treatment.

Figure 18 compares the SEM-EDS elemental maps (Sn and Ti overlay) of the Nb₃Sn layers for each sample after heat treatment at 650 °C. The addition of Zn to the Cu intermediate layer combined with Ti alloying in Nb maximizes the Nb₃Sn layer thickness, even though the isolated quaternary Ti-containing compound phase remains at the Nb₃Sn/Cu–Sn interface (approximately Nb:Sn:Cu(Zn):Ti = 25:20:50:5).

Figures 19(a) and (b) show the pinning force (F_p) per superconducting volume derived from the magnetization curves and the Sn compositional ratio distribution in the Nb₃Sn layer, respectively. Compared with N-CZ-ST, NT-CZ-S exhibited a higher B_{c2} and improved high-field J_c ; however, its F_p was lower than that of NT-C-S across the entire magnetic field range. Figure 19(b) shows that the Sn concentration gradient in NT-CZ-S was steeper than that in NT-C-S. This steeper concentration gradient is attributed to the reduced Sn diffusion driving force caused by the presence of the interfacial quaternary phase. Such a pronounced concentration gradient may contribute to the reduced F_p in NT-CZ-S. Meanwhile, the Sn concentration near the Cu–Sn/Nb₃Sn interface in NT-CZ-S remained comparable to that in NT-C-S. This observation suggests that differences in the chemical-potential gradient alone



may not fully account for the steeper Sn concentration gradient observed in NT-CZ-S. According to equation (1), diffusion flux is governed by both the thermodynamic driving force and atomic mobility. Therefore, the present result raises the possibility that Zn addition influences not only the thermodynamic driving force but also the effective mobility governing Sn transport, for example through Zn segregation at Nb₃Sn grain boundaries (figure 6). A more detailed analysis, including quantitative grain size evaluation, is currently underway, and the results will be reported in a future publication.

3.6. Arrangement of Ti source in Nb modules

In the internal-Sn method, the addition of Ti to Nb offers the potential to enhance the performance of Nb₃Sn wires. When introducing the Ti source into Nb modules, Ti may be uniformly alloyed with the Nb filaments; alternatively, isolated Ti sources may be distributed within the Nb module, as observed in RRP-type wire designs. Additionally, a double-layered structure for each Nb filament is conceivable, wherein a Ti source is artificially placed at the center of the filament. Here, two types of laboratory-scale multifilamentary wires with distinct Ti addition configurations were prepared. The sample featuring Ti uniformly alloyed with Nb was designated as Multi-NT-CZ-S, while the sample with Nb – 47 wt%Ti positioned at the Nb filament center was designated as Multi-NNT-CZ-S. Figure 20 shows cross-sectional SEM images of the precursor wires, and table 3 summarizes their specifications. The subsequent sections describe and discuss the changes in Nb₃Sn layer formation and the resulting superconducting characteristics. Both samples employed cross-sectional assemblies similar to those of DT-type wires, with 15 wt. % Zn added to the Cu matrix.

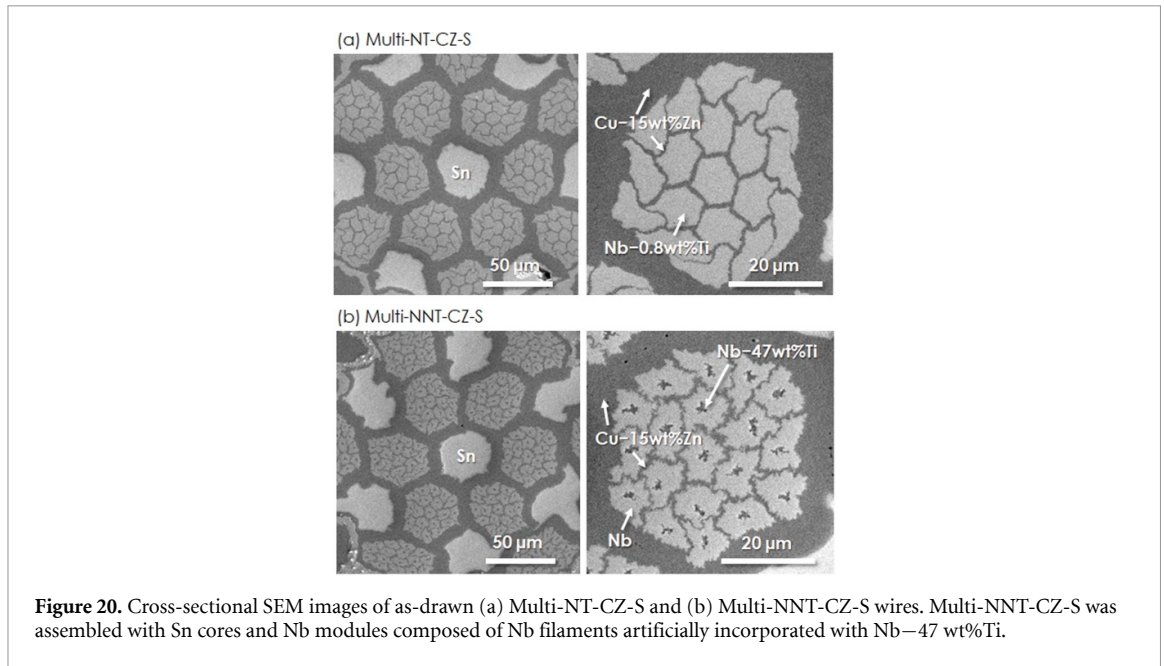


Figure 20. Cross-sectional SEM images of as-drawn (a) Multi-NT-CZ-S and (b) Multi-NNT-CZ-S wires. Multi-NNT-CZ-S was assembled with Sn cores and Nb modules composed of Nb filaments artificially incorporated with Nb–47 wt%Ti.

Table 3. Specifications of DT-type Nb₃Sn precursor wires with different Ti doping configurations to the Nb filament.

	Multi-NT-CZ-S	Multi-NNT-CZ-S
Wire diameter (mm)	0.7	0.7
Nb module diameter (μm)	49.7	49.7
No of Nb modules	12	12
No of Nb filament in the module	19	19
Nb filament diameter (μm)	9.3	9.3
No of Sn modules	7	7
Sn core diameter (μm)	42.2	42.2
Ti/(Nb + Ti) ratio (wt%)	0.8	1.62

Figures 21(a) and (b) show cross-sectional SEM images of the Nb₃Sn module following reaction at 650 °C. In both instances, the absence of multinary compound phases between the Nb filaments, which serve as Sn diffusion pathways, suggests that smooth Sn diffusion occurred. However, the reaction microstructure revealed significant differences. The Nb filaments with uniform Ti doping (Multi-NT-CZ-S) failed to undergo full reaction, leaving unreacted Nb at the center. In contrast, Multi-NNT-CZ-S received a sufficient Sn supply to the filament center. This phenomenon is attributed to the enhanced Sn diffusion kinetics driven by the mutual diffusion between Cu and Ti as shown in figure 6. Figures 21(c) and (d) plot the radial distribution of the Sn concentration ratio ($\text{Sn}/(\text{Nb} + \text{Sn} + \text{Ti})$) and the Ti concentration within the Nb₃Sn layer for each sample. EDS analysis identified the contrast observed at the center of the Multi-NNT-CZ-S sample as a multinary compound phase of Nb – Sn – Ti – Cu(Zn) resulting from the reaction between Sn and residual NbTiCu compounds. Multi-NNT-CZ-S seemingly achieved Sn concentration ratios closer to the stoichiometric composition, as compared with Multi-NT-CZ-S.

Figures 22(a) and (b) show the magnetic field dependence and the reaction temperature dependence of the non-Cu J_c for each sample heat-treated at 650 °C, respectively. Following heat treatment at 650 °C, Multi-NNT-CZ exhibited significantly higher J_c properties than Multi-NT-CZ-S. Given that the grain sizes evaluated from the fracture surfaces were approximately 125 nm for both Multi-NT-CZ-S and Multi-NNT-CZ-S, the observed difference in J_c is primarily attributed to differences in the stoichiometry of the formed Nb₃Sn. The comparable Nb₃Sn grain sizes in both samples imply that the Sn chemical potential at the Cu–Sn site was also similar. Consequently, the lower stoichiometry of the Nb₃Sn layer in Multi-NT-CZ-S results mainly from reduced Sn diffusion kinetics. This behavior is likely associated with the absence of Cu–Ti mutual diffusion. Additionally, the segregation of Ti-related compounds during the early stage of grain boundary diffusion as shown in figure 18(d) is considered to influence the Sn diffusion kinetics [84, 128].

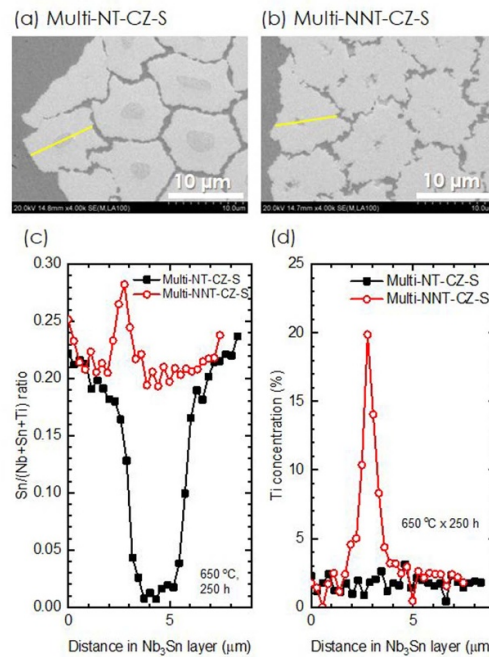


Figure 21. SEM-BSE images of the Nb modules in (a) Multi-NT-CZ-S and (b) Multi-NNT-CZ-S after reaction at 650 °C for 150 h. Concentration distributions of (c) Sn and (d) Ti in the Nb₃Sn filaments. The yellow line indicates the EDS analysis line.

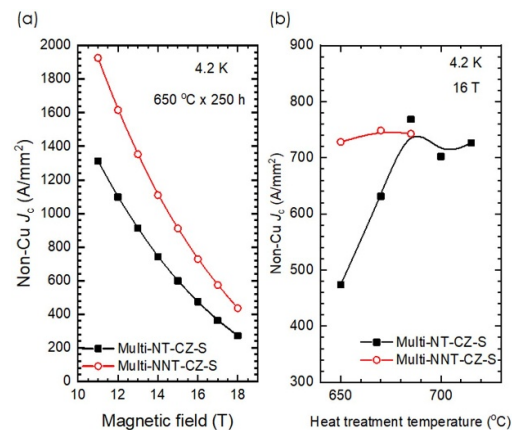
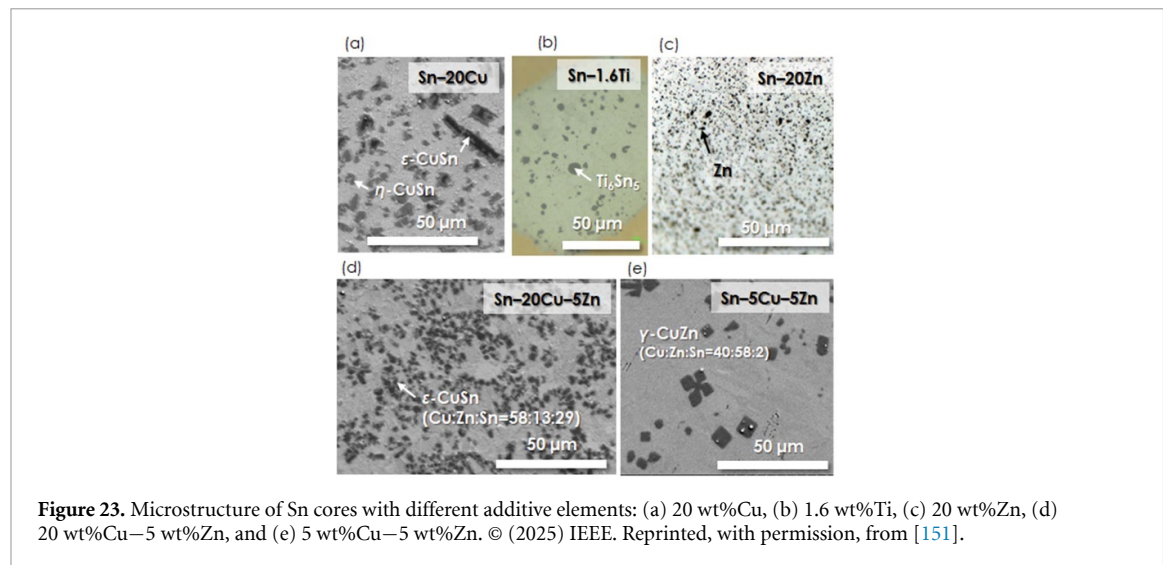


Figure 22. (a) Non-Cu J_c versus magnetic field of Multi-NT-CZ-S and Multi-NNT-CZ-S reacted at 650 °C for 250 h and 4.2 K and (b) temperature dependence of non-Cu J_c at 16 T for both wires.

3.7. Elemental doping to Sn core

In the internal Sn method, the necessity of drawing composite wires comprising materials with markedly different hardnesses constitutes a major challenge for industrial applications. One approach to enhancing the hardness balance involves increasing the hardness and homogeneity of the Sn core. Alloying elements such as Zn, Cu, Ti and Mg can be introduced to achieve a relatively uniform dispersion of precipitates during casting under near-thermodynamic equilibrium conditions [76, 133, 150, 151]. Copper readily forms intermetallic compounds with Sn, and the η and ε phases tend to grow during solidification; however, at concentrations up to approximately 20 wt. % Cu, a microstructure featuring a finely dispersed η phase can be obtained (figure 23(a)) [151]. Regarding Ti additions, stable Ti₆Sn₅ precipitates are dispersed throughout the core, although a tendency toward coarsening is observed (figure 23(b)). A notable characteristic of Zn addition is its complete immiscibility with Sn, which results in a uniform dispersion of Zn after melting and drawing (figure 23(c)). The co-addition of Cu is effective for further refining and homogenizing the dispersion (figure 23(d)). However, an increase in the relative Zn ratio is likely to increase the size of the precipitated particles (figure 23(e)). Copper preferentially bonds with Zn rather than Sn, leading to the formation of Cu–Zn compounds. Even at a total Cu + Zn content of 50 wt. %, these compounds form preferentially, yielding a dispersed microstructure devoid of significant precipitate



crystal growth [151]. From the perspective of homogenization, previous research indicates that atomized Sn–Cu–Ti powders can be consolidated via cold isostatic pressing [31].

The Vickers hardness of Sn is approximately 7.5 HV [152], whereas that of Sn–20at%Cu, Sn–3.9at%Ti (Sn–1.6 wt%Ti), Sn–31at%Zn (Sn–20 wt%Zn), Sn–20at%Cu–5at% Zn, and Sn–5at%Cu–5at%Zn increases to approximately 19.4, 11, 20.7, 22.6, and 11 HV, respectively. Ensuring a sufficient Sn content in the core is essential for promoting Nb₃Sn layer formation, and thus future studies should focus on detailed investigations of alloying elements and compositions that enable sufficient hardness enhancement with reduced addition levels.

4. Summary: perspective for high-performance Nb₃Sn wires

Table 4 summarizes the representative additive elements discussed throughout this review and their principal effects on Nb–Sn diffusion reactions.

In light of the FCC project and the recent move toward compact DEMO reactors, Nb₃Sn wires featuring superior J_c performance fabricated via the internal Sn method are essential for achieving superior coil magnetic field performance under space-limited conditions. Concurrently, these applications increasingly require enhanced mechanical strength to withstand extreme electromagnetic force environments. If hybrid designs employing both high-performance Nb₃Sn wires and bronze-route wires prove feasible for Nb₃Sn coils, sustained demand for high-strength bronze-route Nb₃Sn wires is anticipated [26, 43, 153].

Furthermore, applications requiring fast magnetic field ramping, including the FCC-hh and tokamak magnets, necessitate the mitigation of hysteresis losses. This can be realized through the further reduction of the effective filament diameter and lowering of low-field J_c [27, 154, 155]. From a stability perspective, increasing the specific heat of the conductor to mitigate temperature rise is also considered an effective strategy [156, 157].

Accordingly, the future development of Nb₃Sn wires demands not only the further enhancement of J_c but also increased multifunctionality. Achieving these goals requires a comprehensive approach to the microstructural control of all constituent materials within the conductor, rather than focusing exclusively on Nb₃Sn phase formation. Elemental additions constitute a primary strategy for comprehensive microstructural control and, when applied with a thorough fundamental understanding, are expected to facilitate the realization of high-performance Nb₃Sn wires with novel and advanced functionalities.

The key topics regarding the elemental additions discussed in this paper and their implications for future conductor development are summarized as follows:

1. Control of the Sn chemical potential through Cu addition.

→ Efficient Nb₃Sn layer formation requires a high Sn chemical potential while maintaining the Sn content in the Cu–Sn matrix below 25 at%, thereby suppressing the formation of Sn-rich Nb–Sn compounds and facilitating Sn transport toward the growing Nb₃Sn layer.

Table 4. Representative additive elements discussed in this review and their principal effects on Nb–Sn diffusion reactions.

Element	Remark
Cu	Reduces Sn chemical potential, destabilizes Sn-rich Nb–Sn intermediate phases, suppresses their formation, and facilitates Sn diffusion during Nb ₃ Sn layer growth.
Ti	Enhances B_{c2} of Nb ₃ Sn. Ti source location is critical because Ti additions on the Sn side promote interfacial Nb–Sn–Cu–Ti compound formation, while Ti additions on the Nb side suppress these phases and improve layer homogeneity.
Ta	Enhances B_{c2} of Nb ₃ Sn. Approximately twice the Ta concentration is required to achieve a comparable effect to Ti because Ta substitutes for Nb sites less readily. Owing to its limited diffusivity, Ta is generally added to the Nb precursor.
Zn	Increases the Sn diffusion driving force and alters grain-boundary composition, thereby modifying grain-boundary elemental pinning characteristics.
Zr	Forms oxide nanoparticles through internal oxidation, leading to grain refinement via the Zener pinning effect.
Hf	Increases the recrystallization resistance of Nb, promotes grain refinement, and contributes to higher J_c . Similar to Zr, Hf can also form oxide nanoparticles through internal oxidation, resulting in Zener-pinning-induced grain refinement.
Mg	Reported to promote Nb ₃ Sn grain refinement. Its effectiveness may depend strongly on conductor architecture because Mg can be incorporated into interfacial quaternary compounds when added together with Ti on the Sn side.

2. The Sn concentration gradient within the Nb₃Sn layer originating from the slow bulk diffusion of Sn.
→ Minimizing the Nb/Sn diffusion distance in the precursor is an effective strategy for reducing the Sn concentration gradient and improving Nb₃Sn layer homogeneity.
3. Copper diffusion at Nb₃Sn and Nb grain boundaries.
4. Changes in pinning force and grain size at Nb₃Sn grain boundaries induced by grain-boundary Cu concentration (Zn addition is discussed as a representative example).
→ Grain-boundary Cu concentration may be controlled through elemental-addition strategies, providing a potential route to optimize grain-boundary pinning and grain-size control.
5. Variation in interfacial compound phase formation depending on the Ti addition site.
→ Thermodynamic assessment of possible interfacial compound phases is essential prior to elemental addition, since such phases can significantly alter diffusion pathways, reaction kinetics, and Nb₃Sn layer formation. The Ti-addition example demonstrates that introducing Ti from the Nb-module side is preferable because it minimizes the formation of undesirable interfacial compound phases.
6. Effective strategies for multielement addition.
→ The Zn-addition example demonstrates that conductor optimization should not focus exclusively on the Nb₃Sn layer. Zn addition is also effective in suppressing void formation during the Cu–Sn interdiffusion stage. Comprehensive microstructural control of both the Nb₃Sn layer and the surrounding matrix is essential for achieving multifunctional conductor performance.
7. Fundamental theories of nucleation and grain growth.
→ Grain refinement can be promoted by increasing the thermodynamic driving force for nucleation. From this perspective, increasing the Sn chemical potential and introducing internal strain energy are promising approaches for enhancing nucleation frequency and refining the Nb₃Sn grain structure. In addition, the ability of additive elements to provide a Zener pinning effect should also be considered, since suppression of grain growth is equally important for achieving fine-grained microstructures.
8. Strengthening of the Sn core through elemental additions.
→ Improved wire drawability requires fine and homogeneous compound dispersions while minimizing the formation of coarse brittle phases.

Despite substantial progress in understanding Nb₃Sn layer formation, several important scientific questions remain unresolved.

First, the relationship between grain-boundary chemistry and elemental pinning behavior remains poorly understood. A quantitative understanding of this relationship may provide important guidelines for maximizing grain-boundary pinning and further enhancing J_c .

Second, the effects of elemental additions on Nb₃Sn layer formation, including diffusion driving force, diffusion kinetics, and microstructural evolution, require further clarification. Similar challenges

exist in the design of high-strength matrix materials. Additional beneficial elements may remain undiscovered, and the exploration of such effects is expected to benefit significantly from thermodynamic databases, CALPHAD-based calculations, diffusion simulations, and AI-assisted materials design.

Third, although APC conductors have demonstrated outstanding superconducting performance, further understanding of the underlying mechanisms remains desirable, including the respective roles of grain refinement and direct nanoparticle pinning, as well as the optimization of nanoparticle precipitation conditions, size, and density. Furthermore, whether APC concepts can be generalized beyond current internal-oxidation-based PIT conductors and successfully implemented in other Nb₃Sn conductor architectures remains an open question. The development of alternative routes for introducing nanoscale precipitates into various Nb₃Sn conductor architectures may represent a promising direction for future research.

Addressing these issues will be essential for the rational design of next-generation multifunctional Nb₃Sn conductors possessing simultaneously high J_c , high mechanical strength, and improved stability.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Author contribution

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Conceptualization (lead), Data curation (lead), Formal analysis (lead), Funding acquisition (lead), Investigation (lead), Methodology (lead), Project administration (lead), Writing – original draft (lead)

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