

Nb₃Sn wire fabrication using rod-in-tube method with a diffusion couple of Nb and Sn alloy with co-addition of Cu and Zn

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Abstract—Large-scale devices, such as the next generation high energy particle accelerator project, demand the enhancement of the J_c and cost performance of Nb₃Sn wires. The cost performance is closely related to the design and drawability of the wires. This study aims to increase the hardness of the soft Sn core of the wires through the precipitation of fine compound particles, improve the hardness balance between the constituent materials (Nb, Cu, and Sn), and investigate the feasibility of this method for practical wire drawing. First, we investigated the variations in microstructure and hardness when Cu and Zn were co-added to Sn. A total addition of 50% Cu–Zn to the Sn alloy can triple the hardness, leaving the alloy ductile enough to sustain the entire deformation process. Subsequently, we manufactured tube-type multifilamentary wires using subelements containing different types of Sn alloys, with and without an intermediate layer of Cu between Sn and Nb. In the absence of Cu, we observed the formation of a large area of Nb₆Sn₅, which is responsible for the formation of a large coarse Nb₃Sn grain area. The presence of Cu suppresses the formation of the Nb₆Sn₅, demonstrating that controlling the Cu/Sn ratio at the subelement level is critical to suppressing the formation of coarse Nb₃Sn areas.

Index Terms— Hardness, Microstructure, Nb₃Sn, Sn alloy, Zn.

I. INTRODUCTION

LARGE-SCALE devices, such as next-generation high-energy particle accelerators and fusion reactors, require extremely large numbers of superconducting wires [1][2],[3],[4],[5],[6]. Therefore, the realization of these projects necessitates the enhancement of wire performance as well as cost effectiveness. Currently, high-performance Nb₃Sn wires are of the internal tin type[7],[8],[9],[10],[11], tube-type[12],[13], [14] and powder-in-tube type with internal oxidation processes[15],[16],[17],[18]. The performance of the internal tin and tube-type Nb₃Sn wires is approaching the required J_c for the Future Circular Collider (FCC) project (1500 A/mm² at 16T)[1],[2],[3]. Furthermore, the internal oxidation of Nb₃Sn wires exceeds the stipulated requirement for this project[19]. Meanwhile, the FCC magnets are designed to incorporate several thousand tons of Nb₃Sn wiring, amounting to thousands of millions of euros. Therefore, enhancing the cost

performance of the Nb₃Sn wires by a small percentage would lead to cost savings of tens of millions of euros.

Cost performance is closely related to the wire design and drawability. Good drawability can mitigate the risk of wire breakage and improve the yield rate. Furthermore, a good hardness balance would contribute to the maintenance of a good cross-sectional shape. The Nb₃Sn precursor composite comprised Cu, Nb, and soft Sn. The hardness of Sn is less than one-sixth of that of Cu or Nb. As the composite is deformed, the difference in hardness increases. Empirically, this hardness imbalance is often considered to be the primary cause of irregular deformation during the drawing process.

In this study, various Sn alloys were fabricated by the co-addition of Cu and Zn. Their microstructures, compositions, and hardnesses were investigated. Zn may have dispersed the compound precipitate. Tube-type multifilamentary wires are manufactured using Sn alloys. The feasibility of using hardened Sn alloys was investigated through microstructural observations of phase formation behavior and critical current measurements.

II. EXPERIMENTAL

A. Casting of Sn Alloy

The Sn alloys were cast in a normal induction-heating furnace in an Ar atmosphere. Cu, Zn, and Ti were used as additive elements. A mixture was prepared by combining predetermined amounts of each constituent material. For the addition of Ti, titanium powders (– 325 mesh) were utilized. The mixture was heated to a temperature range of 500 to 900 °C, which is above the melting point of Sn-alloys, and maintained for a sufficient duration to ensure the complete dissolution of Ti.

B. Microstructural and Microchemical Analysis

The microstructures of the samples were observed using a field-emission scanning electron microscope (FESEM), and composition analysis was performed using energy-dispersive X-ray spectroscopy (EDS) with the FESEM. The cross-section was first polished with polycrystalline diamond suspensions

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and then polished with non-crystallizing amorphous 0.05 and 0.02 μm colloidal silica suspensions (MasterMet, Buehler) in the final steps.

The hardness of the Sn alloys was measured using a Micro-Vickers hardness tester at a force of 0.025 kgf.

C. Wire Fabrication

Two configurations of the tube-type subelement were fabricated, distinguished by the presence of an additional copper layer between the Nb-alloy tube and the central Sn-alloy bar. In Configuration 1, the Sn-alloy bar, swaged to a diameter of 2.7 mm, was inserted into the Nb tube with an outer/inner diameter of 5.8/3.0 mm. This composite was then inserted into a Cu tube with an outer/inner diameter of 8.0/6.0 mm. This single-core composite was swaged and drawn into a hexagonal wire 1.0 mm in height. Nineteen pieces of the hexagonal single-core wires were stacked into a Cu tube with an outer/inner diameter of 8.0/5.3 mm and subsequently drawn into a wire 1.09 mm in diameter.

In Configuration 2, a thin Cu layer was incorporated between the Nb and the Sn alloy. The Sn alloys, swaged into a bar with a diameter of 8.5 mm, was inserted into a Cu tube with an outer/inner diameter of 10.0/9.0 mm. The Sn/Cu rod was then deformed to a final diameter of 2.7 mm. The subsequent steps for producing the subelement and the multifilamentary wire were the same as those followed for the Configuration 1. The final wire was a multifilamentary wire with 19 filaments and 1.09 mm in diameter.

Each sample was encased in a quartz tube filled with argon gas. The encased samples were heat treated at 650 $^{\circ}\text{C}/100$ h with a ramp-up time of 4 h for Nb_3Sn phase formation. To observe the temporal evolution of the microstructure, the samples were removed from the furnace at 400, 500, 600, and 650 $^{\circ}\text{C}$, as well as at 650 $^{\circ}\text{C}/20$ h and 650 $^{\circ}\text{C}/50$ h. At the extraction from the oven, the sample had been immediately quenched in water.

C. I_c Measurement

I_c was measured for short samples (4 cm in length) using the standard four-point probe method. The voltage tap distance was 10 cm and the I_c was determined by an electric criterion of 1 $\mu\text{V}/\text{cm}$. The non-Cu J_c was calculated by dividing the I_c by the non-Cu area.

III. RESULTS AND DISCUSSION

A. Microstructure and Hardness of Sn Alloys

The compositions of the cast Sn alloys are summarized in Table I, and the backscattered electron (BSE) images of the microstructures of typical Sn alloys are shown in Fig. 1. In Sn-2Ti, typically, Ti_6Sn_5 with a size of approximately 4 μm precipitates [20]. For a 20% Cu composition, the microstructure shows an island-like microstructure with tiny $\eta\text{-CuSn}$ particles dispersed. However, Sn-50Cu exhibited a dendritic microstructure with $\varepsilon\text{-CuSn}$ phase growth and became markedly brittle due to its excessive Cu content. In Sn-20Cu-30Zn-2Ti and Sn-10Cu-40Zn-2Ti, there was no dendritic

structure; however, a microstructure with dispersed compound particles was observed. EDS analysis indicated that these compounds are $\beta\text{-CuZn}$ and $\gamma\text{-CuZn}$ in Sn-20Cu-30Zn-2Ti and Sn-10Cu-40Zn-2Ti, respectively. According to the phase diagram of Cu-Zn, the composition ratios of Cu and Zn in these alloys are close to those of $\beta\text{-CuZn}$ and $\gamma\text{-CuZn}$, respectively. The melting points of these phases were approximately 900 and 830 $^{\circ}\text{C}$, respectively. Therefore, $\beta\text{-CuZn}$ and $\gamma\text{-CuZn}$ are stable in the Sn alloy; hence, they would precipitate as embryos during cooling. Notably, Zn does not dissolve in Sn. Thus, $\beta\text{-CuZn}$ and $\gamma\text{-CuZn}$ are presumed to have precipitated preferentially. The particle size ranged from approximately 10 to 20 μm .

In Sn-20Cu, the embryo is $\eta\text{-CuSn}$ as mentioned above. The particle size was less than 8 μm . If 5 at% of Zn is added, the particle size is further reduced to almost less than 5 μm , and the particles are more dispersed. EDS revealed that the precipitates originated from $\varepsilon\text{-CuSn}$ containing Zn. The Cu:Zn:Sn composition ratio is approximately 58:14:28. Zn appears to be effective in suppressing the growth of $\varepsilon\text{-CuSn}$. In Sn-10Cu-5Zn, the embryo was believed to be $\beta\text{-CuSn}$; Cu:Zn:Sn is approximately 53:38:9.

Fig. 2 summarizes the Vickers hardness values of the as-cast Sn alloys with those of Nb and Cu as references. The hardness of practically-used Sn-2Ti is approximately 11 HV, which is almost one-sixth that of Nb and Cu. The addition of large

TABLE I
COMPOSITION (AT%) OF CASTED SN ALLOYS

Name	Sn	Cu	Zn	Ti	Ductility
Sn-2Ti	98	0	0	2	Good
Sn-20Cu	80	20	0	0	Good
Sn-50Cu	50	50	0	0	Brittle
Sn-20Cu-30Zn-2Ti	48	20	30	2	Good
Sn-10Cu-40Zn-2Ti	48	10	40	2	Good
Sn-31Zn	69	0	31	0	Good
Sn-10Cu-5Zn	90	10	5	0	Good
Sn-20Cu-5Zn	75	20	5	0	Good

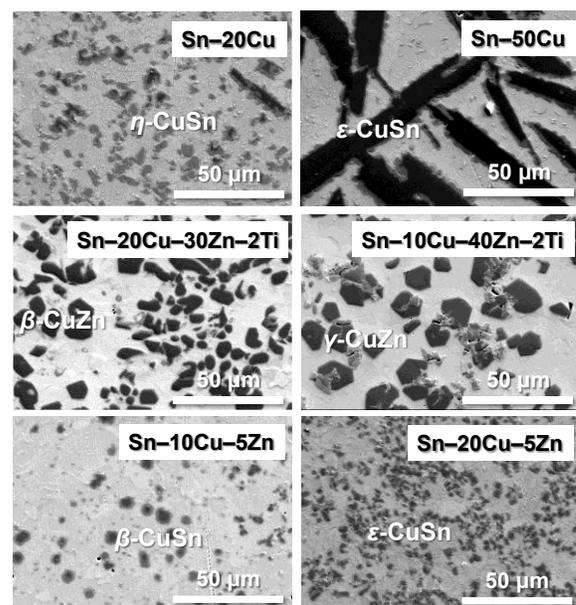


Fig. 1. BSE images of typical Sn alloys.

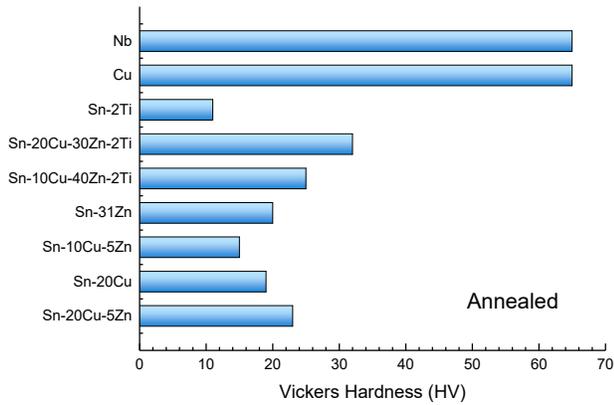


Fig. 2. Comparison of the Vickers hardness of Nb, Cu, and Sn alloys.

amounts of Cu and Zn can triple this hardness. After the drawing process (true strain = 8.14), the Vickers hardnesses of Nb, Cu and Sn-alloy were nearly saturated at 167, 133 and 32 Hv for Sn-20Cu-30Zn-2Ti wire, and 165, 136 and 33 Hv for Sn-10Cu-40Zn-2Ti wire, respectively.

B. Phase Formation Behavior

Cross-sectional views of the multifilamentary wires fabricated using Sn-20Cu-30Zn-2Ti are shown in Fig. 3. Drawability was good until the Sn core size was reduced to approximately 50 μm , at which point wire breakage often occurred. The size limitation may depend on the size of the precipitate.

Fig. 4 shows the phase formation at the interface between Nb and Sn-20Cu-30Zn-2Ti of the sub-element in the multifilamentary wires in Fig. 3(a). The precipitated $\beta\text{-CuZn}$ remained in the Sn alloy core at the temperature of 650 $^{\circ}\text{C}$ and NbSn_2 was formed at the interface (Fig. 4(a)). After 20 h at 650 $^{\circ}\text{C}$, the Nb_2Sn decomposed into Nb_6Sn_5 and Sn and a thin reaction layer of fine-grained (FG) Nb_3Sn layer formed at the reaction front (Fig. 4(b)). After 20 h at 650 $^{\circ}\text{C}$, the Nb_6Sn_5 decomposed into coarse-grained (CG) Nb_3Sn and Sn(Cu, Zn),

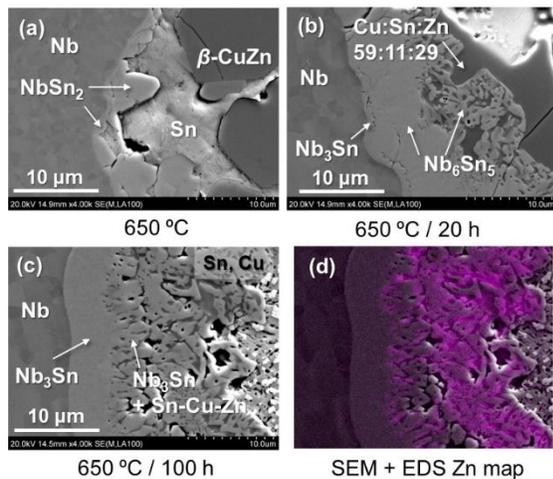


Fig. 4. Phase formation between Nb and Sn-20Cu-30Zn-2Ti of the subelement in the multifilamentary wire (configuration 1) at (a) 650 $^{\circ}\text{C}$, (b) 650 $^{\circ}\text{C}/20$ h and (c) 650 $^{\circ}\text{C}/100$ h, and (d) EDS Zn map.

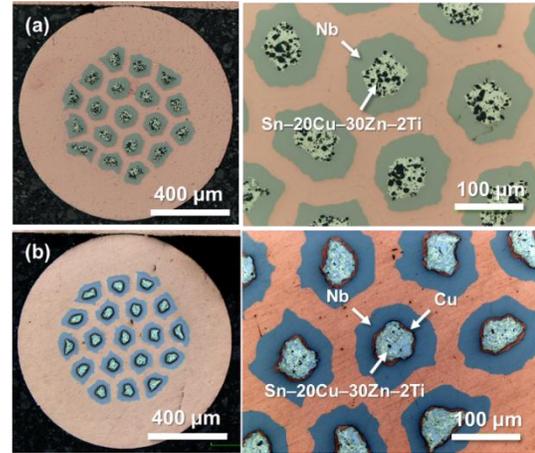


Fig. 3. Cross-sectional views of the multifilamentary wires using Sn-20Cu-30Zn-2Ti: (a) Configuration 1 (without Cu layer between Nb and Sn alloy) and (b) Configuration 2 (with Cu layer).

while the FG Nb_3Sn layer grew further (Fig. 4(c)). Zn does not dissolve in Nb_3Sn as reported in[21] (Fig. 4(d)). This phase formation behavior is similar to the case in which Sn activity is significantly high[22],[23],[24],[25]. In general, in Sn-Cu, the Sn activity increases with the Sn content in Sn-Cu. At Sn content ratios (Sn/(Sn + Cu)) greater than 25at%, reportedly, NbSn_2 and Nb_6Sn_5 forms before Nb_3Sn [23].

To reduce the Sn content, a thin Cu layer was inserted between Nb and the Sn alloy in the second configuration (Fig. 3(b)). Fig. 5 shows the phase formation in this wire when a thin intermediate Cu layer exists between Nb and Sn-20Cu-30Zn-2Ti. The Sn/(Sn + Cu + Zn) ratio decreased from 50at% to 33at%. The layer growth of Nb_6Sn_5 (decomposed from NbSn_2) was suppressed compared to the case without Cu (Fig. 5(a) and (b)). As reported by Xu, an increase in the Cu content resulted in an increase in the FG/CG ratio [22]. This accounts for the small Nb_6Sn_5 layer growth in configuration 2 (Fig. 5(c)). Nb_6Sn_5 did not transform into coarse Nb_3Sn ; instead, a Nb-Sn-Ti-Cu-Zn compound phase was formed (Fig. 5(d)). EDS

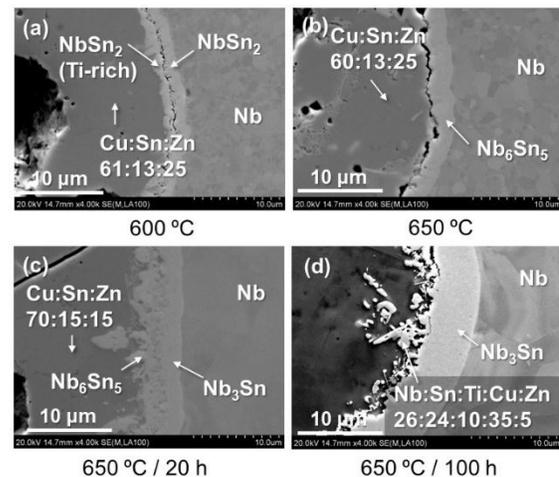


Fig. 5. Phase formation between Nb and Sn-20Cu-30Zn-2Ti in the presence of a thin intermediate Cu layer in the multifilamentary wire (configuration 2) at (a) 600 $^{\circ}\text{C}$, (b) 650 $^{\circ}\text{C}$, (c) 650 $^{\circ}\text{C}/20$ h, and (d) 650 $^{\circ}\text{C}/100$ h.

indicated a Nb:Sn:Ti:Cu:Zn composition ratio of 26:24:10:35:5. The formation of complex compound phases is often observed when Ti is doped to Sn[26].

Fig. 6 shows the grain morphologies of the FG area in configurations 1 and 2. The average grain size was calculated by dividing by the number of grains in a certain area containing more than 140 grains, which is a reasonable number from a statistical standpoint. The average grain sizes were 144 and 114 nm, respectively. Notably, the insertion of a Cu layer resulted in a finer grain morphology. In addition, at a high Sn content (configuration 1), finer grains were observed near the Nb side, whereas at a high Cu content (configuration 2), the grains tended to be finer near the Sn alloy core side.

C. Effect of Zn Addition

Fig. 7 compares the grain morphologies of the FG Nb₃Sn area with those of Sn-20Cu-30Zn-2Ti and Sn-10Cu-40Zn-2Ti in configuration 2. A reduction in the Cu-Zn ratio, as in Sn-10Cu-40Zn-2Ti, resulted in a thicker Nb₃Sn layer. Zn has been reported to be an effective element for promoting Nb₃Sn formation[27]. The increased Zn content is believed to enhance the formation of Nb₃Sn.

D. Non-Cu J_c Properties

Fig. 8 shows the non-Cu J_c properties of the multifilamentary wires fabricated using Sn-20Cu-30Zn-2Ti and Sn-10Cu-40Zn-2Ti. The non-Cu J_c properties were measured for configurations 1 and 2. The J_c values are considerably lower than those of industrial wires[22],[24],[28],[10], which may be because the heat treatment conditions were not optimized and Sn diffusion was not sufficient owing to the large sub-element diameter. Otherwise, Ti might not diffuse sufficiently in Nb₃Sn because of the presence of Nb-Sn-Ti-Cu-Zn or Nb₆Sn₅, as shown in Fig. 4 and 5. As shown in Fig. 8, the J_c of the Sn-10Cu-40Zn-2Ti sample was higher than that of the Sn-20Cu-30Zn-2Ti sample. This can be attributed to the thicker Nb₃Sn layer in the Sn-10Cu-40Zn-2Ti sample. The B_{Kramer} estimated

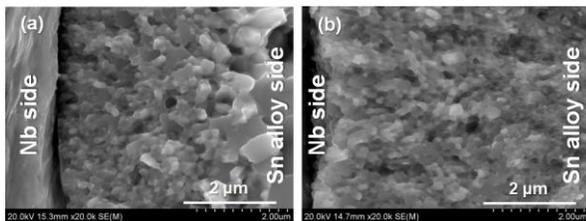


Fig. 6. Grain morphologies of the FG Nb₃Sn area in configurations (a) 1 and (b) 2 using Sn-20Cu-30Zn-2Ti. The average grain sizes are 144 and 114 nm, respectively.

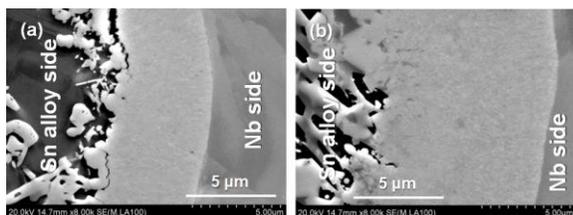


Fig. 7. Grain morphologies of the FG Nb₃Sn area for (a) Sn-20Cu-30Zn-2Ti and (b) Sn-10Cu-40Zn-2Ti in configuration 2.

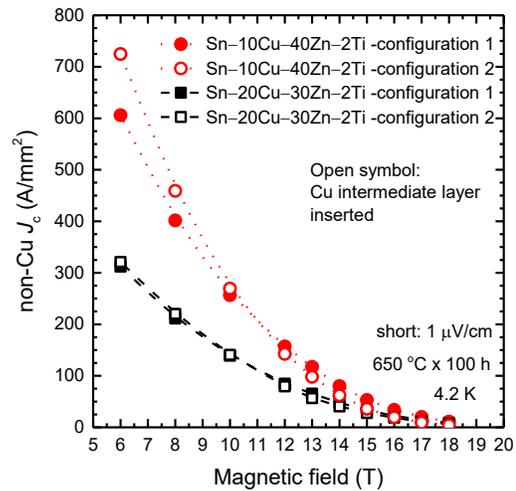


Fig. 8. Non-Cu J_c properties of multifilamentary wires fabricated with Sn-20Cu-30Zn-2Ti and Sn-10Cu-40Zn-2Ti, respectively. Closed symbols and open symbols indicate configuration 1 and 2, respectively.

by extrapolation of $J_c^{0.5}B^{0.25}$ were 20.6 and 21.1 T for Sn-20Cu-30Zn-2Ti wire with and without Cu intermediate layer, and 19.1 and 20.2 T for Sn-10Cu-40Zn-2Ti wire with and without Cu intermediate layer, respectively.

As shown in Fig. 8, J_c - B curves of the wires with and without Cu intermediate layer are crossed. That is presumably attributed to the difference of Ti diffusion to Nb₃Sn phase. As shown in Fig. 5, in the wires with Cu layer, a compound phase of NbSnTiCuZn is formed at the interface of Nb₃Sn and Sn-Cu core. Reportedly, this compound phase tends to trap Ti[26], which would decrease the Ti content in the Nb₃Sn, thereby slightly reducing the B_{c2} . Meanwhile, it is reported that increase in Ti addition to Nb₃Sn tends to slightly coarsen the grain morphology[29],[30], when Ti is doped into Cu-Sn in the precursor, which would decrease the low field J_c properties. Consequently, the J_c curves with and without Cu layer are thought to be crossed.

V. CONCLUSION

In this study, we presented the effect of hardness and microstructure evolution on Sn alloy fabricated through the co-addition of Cu, Zn, and Ti. This resulted in the precipitation of small compound phases, which increased the hardness of Sn by a factor of three, thereby maintaining drawability. When the composition ratio of Cu to Zn (Cu:Zn) is 20:30 or 10:40, Cu combines with Zn to form stable β -CuZn or γ -CuZn. Zn did not dissolve in the Sn. This would be the reason why dendritic structures do not grow in the Sn alloys.

Controlling the Sn/(Sn + Cu + Zn) ratio at the sub-element level by adding an extra Cu layer between the Sn alloy and the Nb tube is crucial to suppress the CG Nb₃Sn area. The consequent increase in Cu content results in a higher FG-to-CG ratio and a finer grain size. An increase in the Zn content tends to promote Nb₃Sn layer formation.

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