Physical Vapor Deposition of Bronze-Route Nb_3Sn for SRF Cavities

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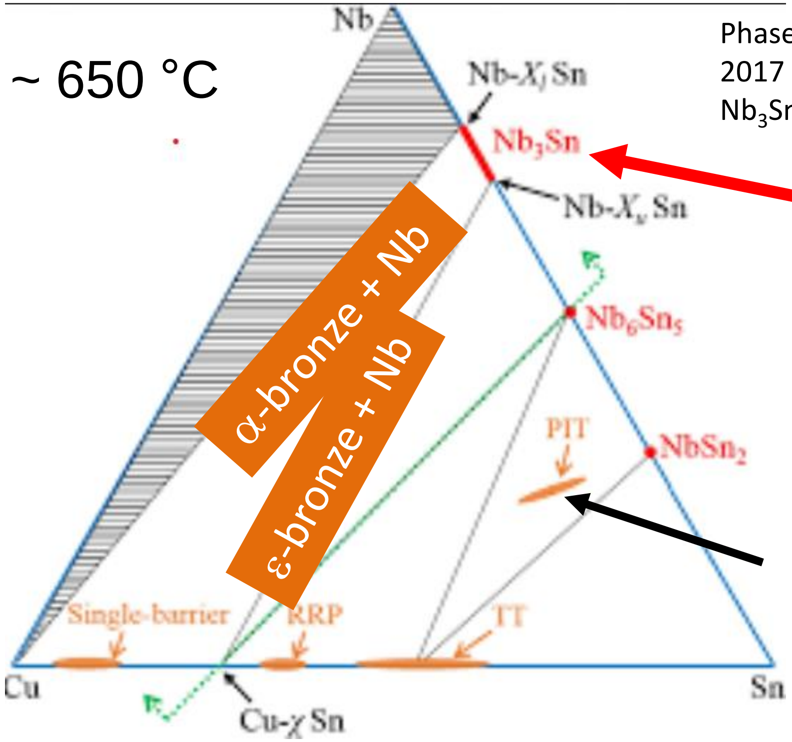
Why investigate bronze routes?

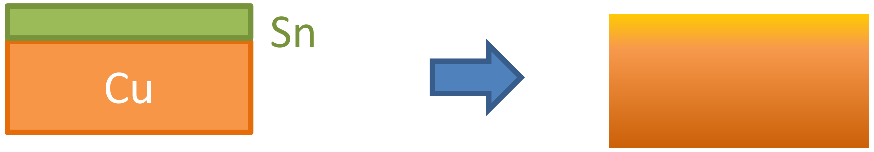
- Move reaction window from ~1100°C to ~700°C
  - Avoid the Nb_6Sn_5 and $NbSn_2$ phases by exploiting ternary Cu-Sn-Nb system
- Make the reaction compatible with cavity bodies made from copper; avoid bulk niobium (and)
  - Possible cost and formability advantages
  - Potential direct application with conduction cooling
    - Genesis of ideas traces back to founding of IARC at Fermilab
- Avoid Sn vapor, chlorides of Sn and Nb polishing chemistry (corrosive, toxicity).
- Tap into wealth of processing knowledge from composite wires.
- If desired, can push grain size to < 50 nm; very low roughness.

Cu-Sn-Nb pathways for SRF

- Apply Nb to bronze and convert entirely to Nb_3Sn
  - Also works for bronzed copper → utilize Cu cavities
  - More complicated geometries are possible, e.g. with diffusion barriers
- Apply bronze to Nb and react to form Nb_3Sn, then remove bronze post-process
  - Utilize Nb/Cu bimetal, e.g. hydroformed Nb/Cu cavities
  - Use Nb cavities

Phase diagram from Xingchen Xu, 2017 topical review of prospects for Nb_3Sn conductors, SuST 30 093001

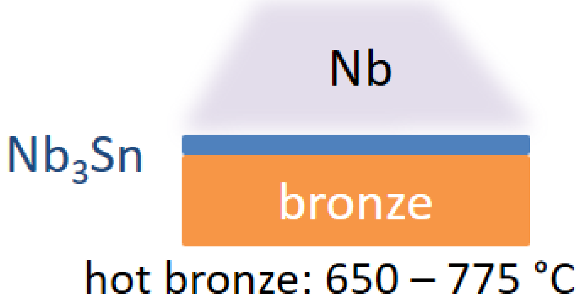




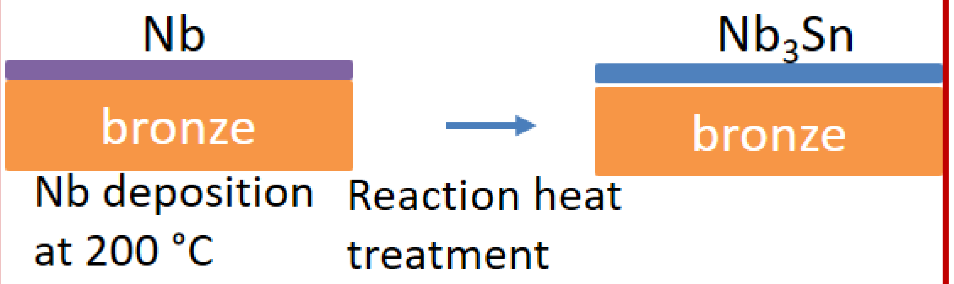
"Bronze" need not be bulk bronze (although bulk bronze is an easy starting point for studies) and can be bronzed Cu

First studies: Nb on α-bronze

- Substrates: Mechanically polished **zero phosphorous Cu-15%wt Sn** and **Cu-15%wt Sn- 0.3%wt Ti** bronzes.
  - Commercial Cu and bronze usually has P as a de-oxidizing agent. Ti was explored here as a getter.
- Substrates were cold-rolled and homogenized at 525°C prior to polishing.
- Nb film deposition conditions.
  - Background pressure ~5E-9 Torr
  - Processing gas 8 mTorr Ar
  - Deposition rate 22.4 nm per minute
  - Niobium film T_c 9.2 K, ΔT_c 0.1 K
- Both process routes took place in the deposition chamber; no vacuum break or exposure to ambient



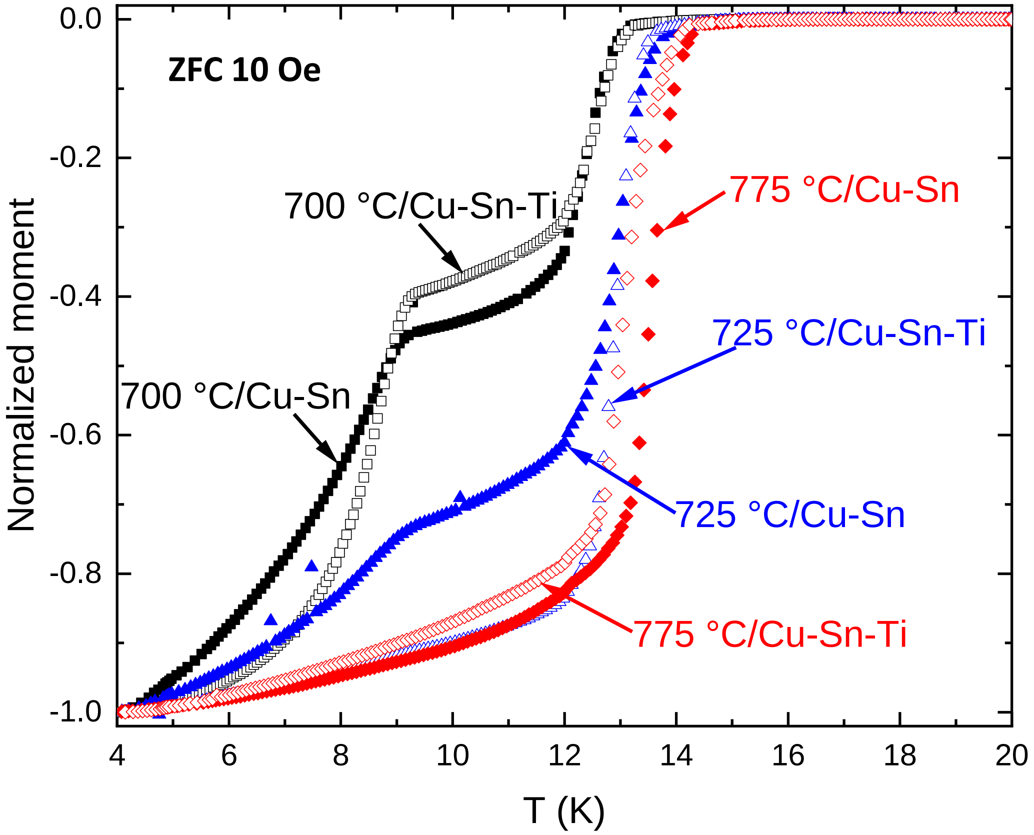
Route 1: Nb_3Sn forms concurrent with Nb deposition on hot bronze

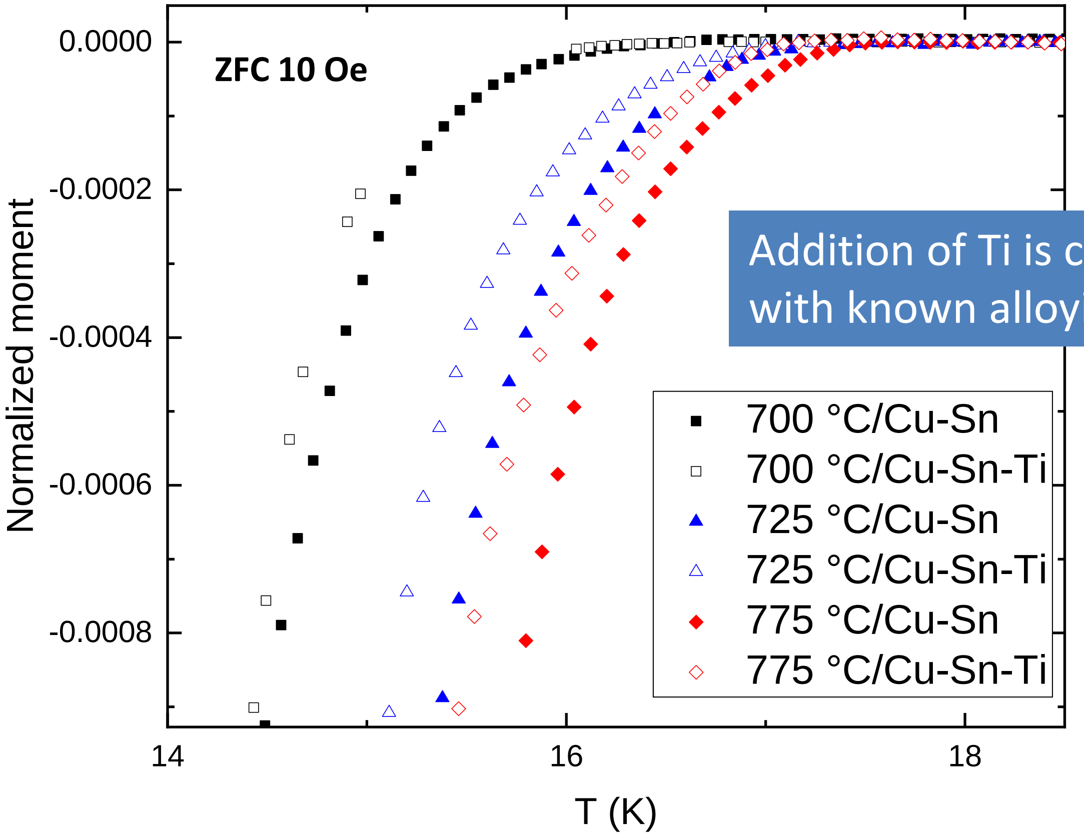


Route 2: Nb_3Sn forms during a diffusion reaction after deposition

Nb deposited on hot bronze achieves 10x faster reaction

650 nm film produced in 20-minute process!



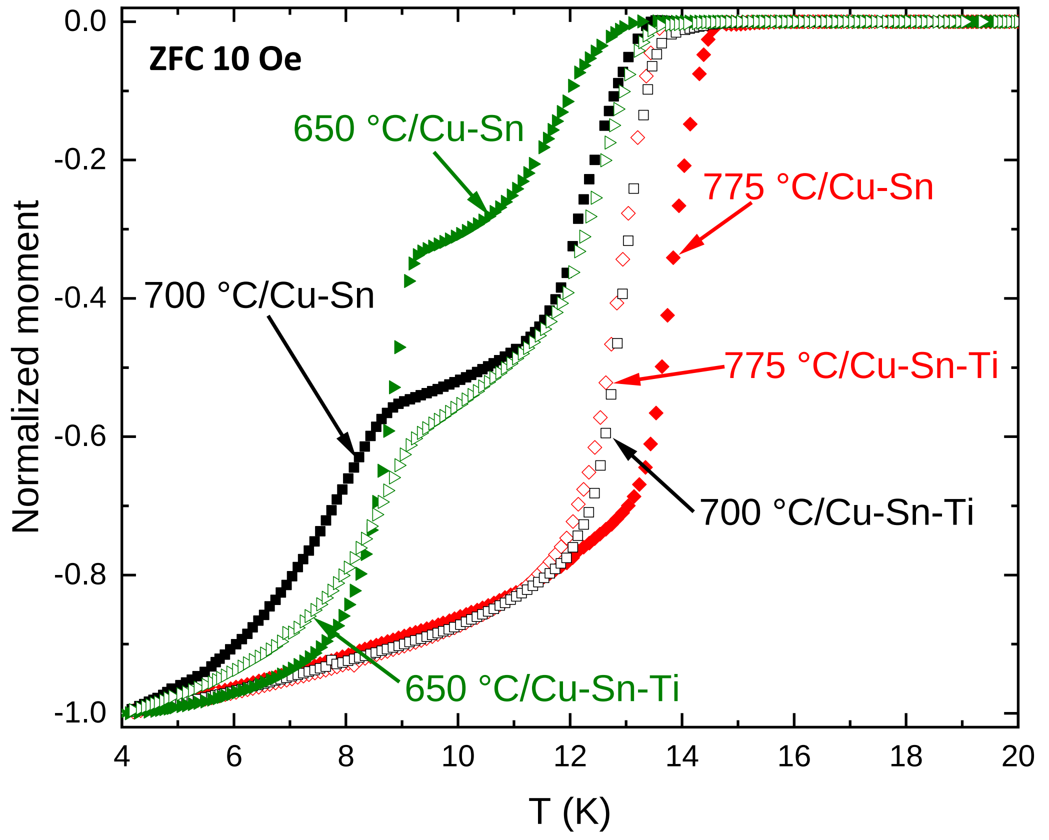


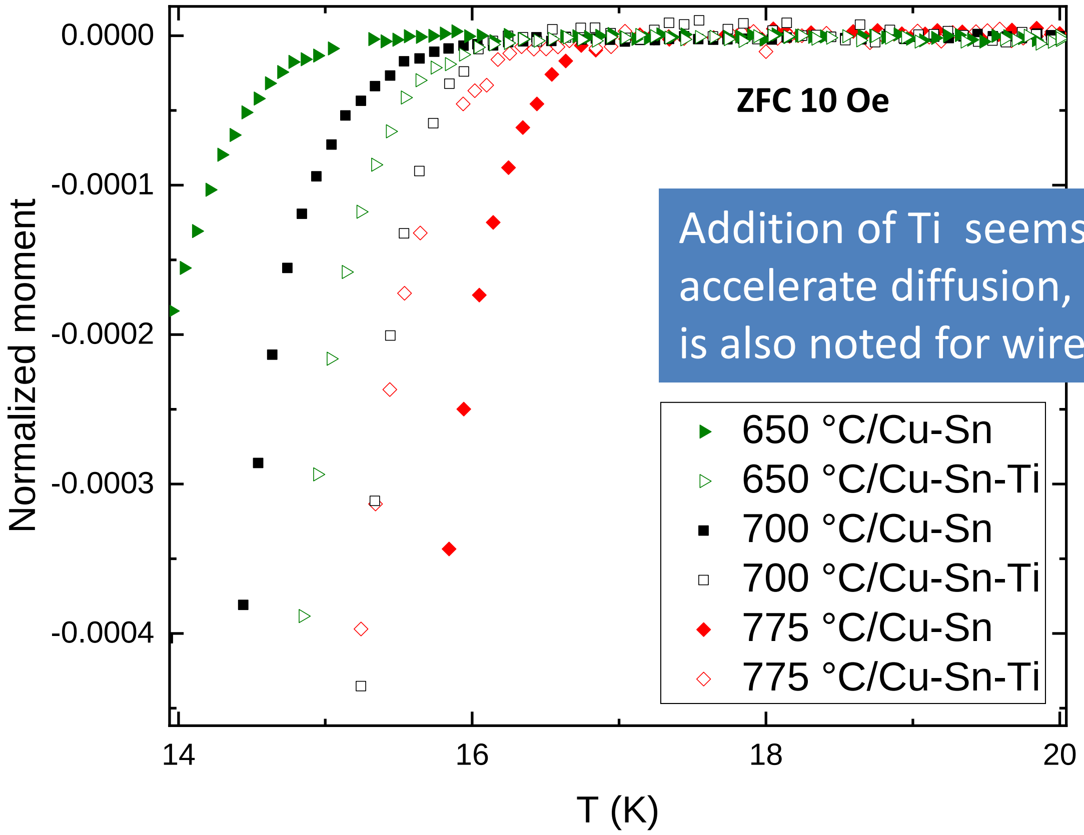
Addition of Ti is consistent with known alloying effects.

14-16 K T_c transition consistent with CTE mismatch between Nb_3Sn and bronze. Also seen by CERN for direct deposition of Nb_3Sn on copper

Nb on bronze + post-reaction behaves like processes in wires

6-hour heat treatments at > 700 °C were required to get full conversion of 500 nm



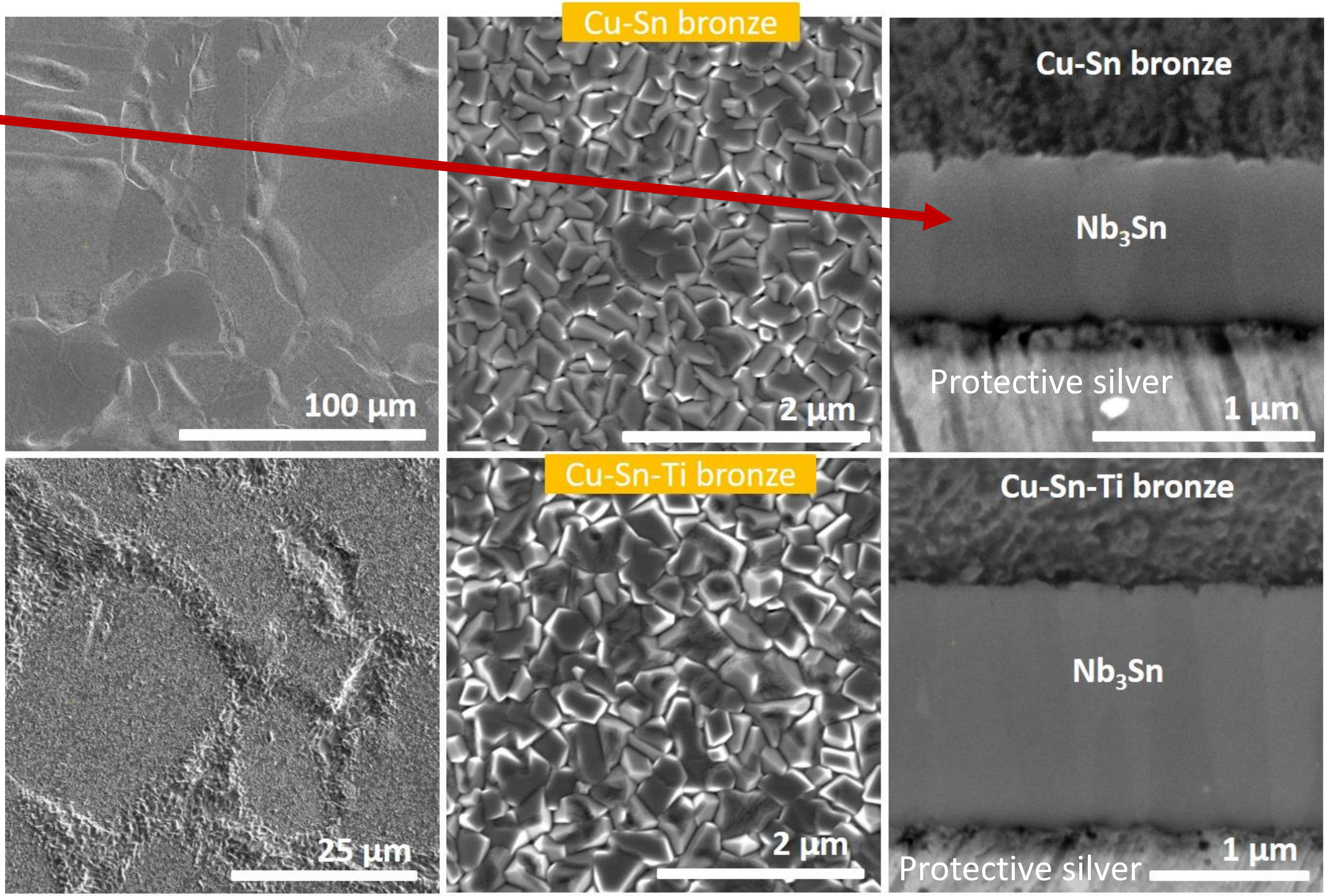


Addition of Ti seems to accelerate diffusion, which is also noted for wires.

Similar effect of CTE mismatch between Nb_3Sn and bronze as for route 1

Columnar grain structure produced by route 1 was never observed in bronze route

- **Columnar grains through entire thickness**
- Pipeline diffusion at bronze GBs → larger Nb_3Sn grains
- Bronze twin boundaries are evident beneath Nb_3Sn
- Very low roughness R_A ~ 7–10 nm in 100 μm^2.
- Sn content of Nb_3Sn layer is measured (EDX) at 25% or higher in both substrates.



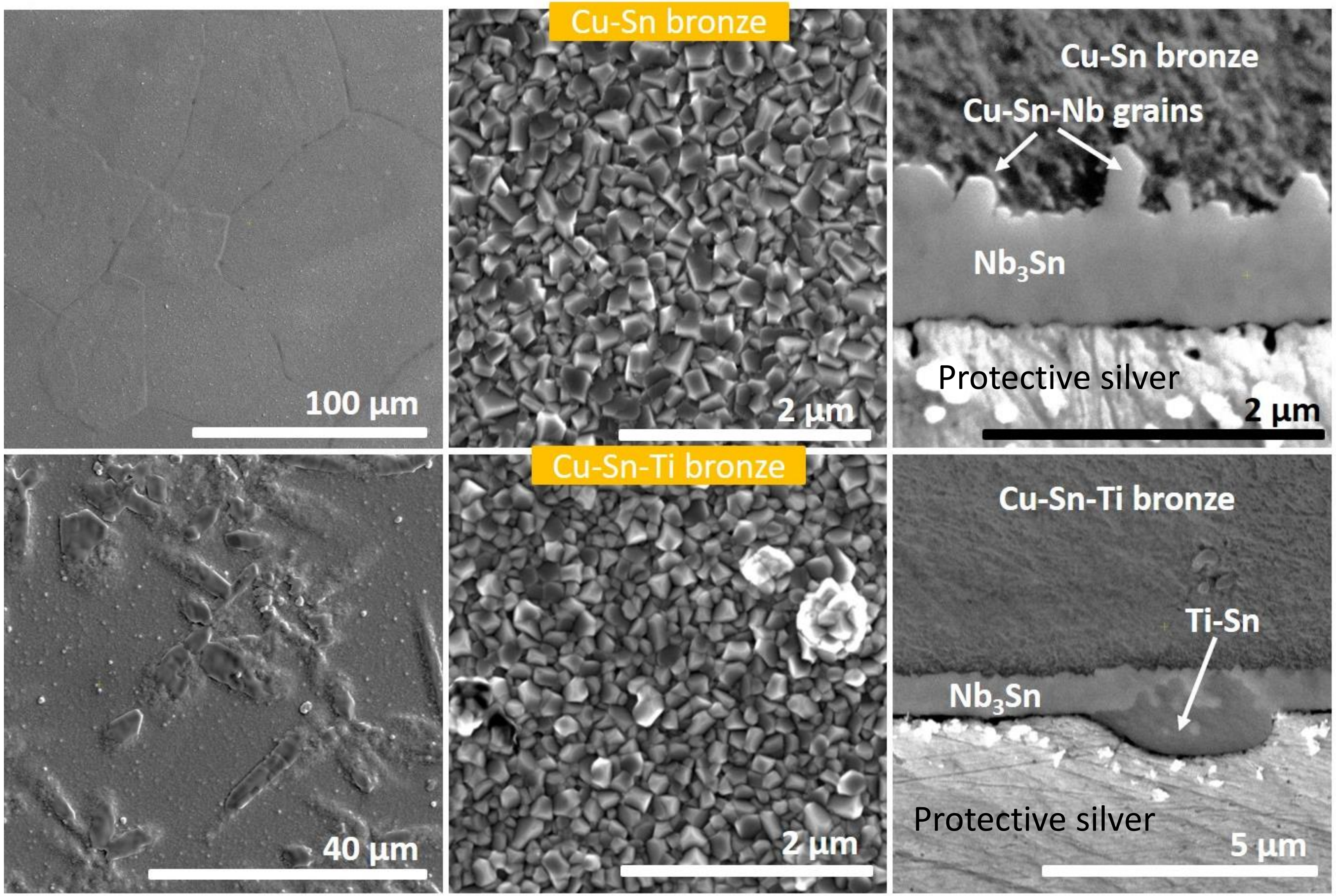
775 °C

Post-deposition reaction produces structures like those seen in wires

- Tendency toward high-angle equiaxed grains
- Evidence for pipeline diffusion along Ti alloyed bronze GBs, with enriched Sn and Ti.
- Very low roughness

$R_A \sim 10$ - 15 nm in $100\ \mu m^2$.

- Sn content of Nb_3Sn layer
- 22–23.5 % no Ti
- 24.5–25.5 % with Ti
- Large Ti_6Sn_5 grains in films on Ti alloyed bronze.



775 °C, 6-hour heat treatment

Discussion and summary

- Address the CTE mismatch issues, e.g. by engineering the copper
  - But 15 K is already sufficient to use conduction cooling for low R_{BCS}
- The hot bronze method may be better
  - Full tin content in Nb_3Sn
- What is the significance of the unique grain structure obtained by deposition of Nb onto hot bronze?
  - Hot bronze in vacuum has a tin-rich surface
    → high tin activity. Can this be exploited further?
  - Can Nb deposition properties give microstructure control?

    → HIPIMS and biasing schemes are available and not yet fully exploited

Discussion and summary

- While roughness is extremely low inside the underlying bronze grains, twin boundaries and grain boundaries affect the Nb_3Sn coating. Is it important to suppress twins and GB effects?
  - Alloying with Ti and other elements increases stacking fault energy, reduces twinning
- What is the significance of the very low roughness? Does this trade off with very high GB density?
- RF measurements are needed.
- Copper in GBs studies are needed.

Acknowledgment

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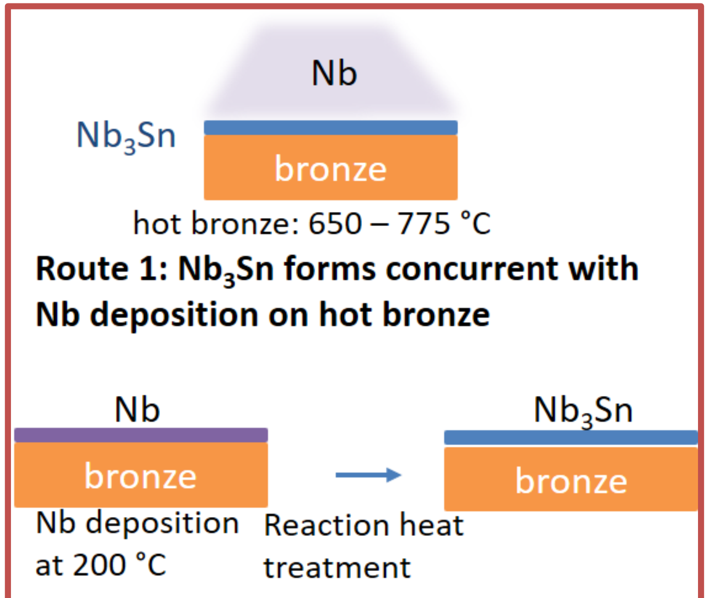
Backup Slides

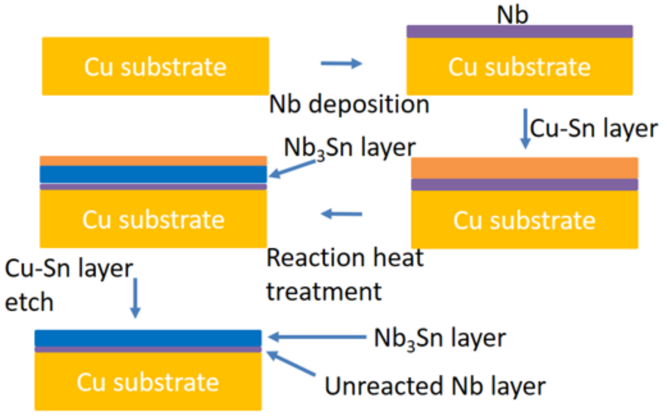
Bronze route Nb_3Sn films for SRF cavities

- Low temperature deposition allows bronze or Cu base cavity.
- Simplest case, Nb_3Sn coated bronze cavities.
- Significant material cost saving from switching Nb host cavity to a bronze or Cu host cavity.
- Easy fabrication and scaling.
- Cu at grain boundaries, Effect on RF?
- Low thermal conductivity, Engineering solutions?

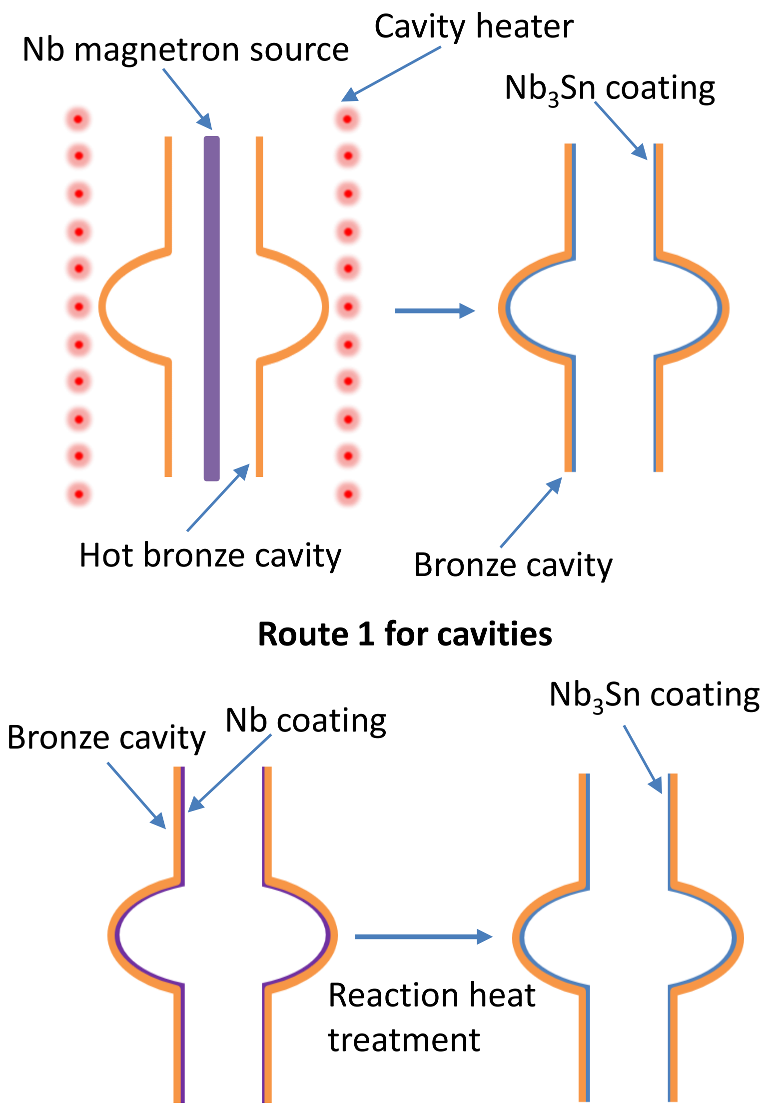


Route 1: Nb_3Sn forms concurrent with Nb deposition on hot bronze

Route 2: Nb_3Sn forms during a diffusion reaction after deposition



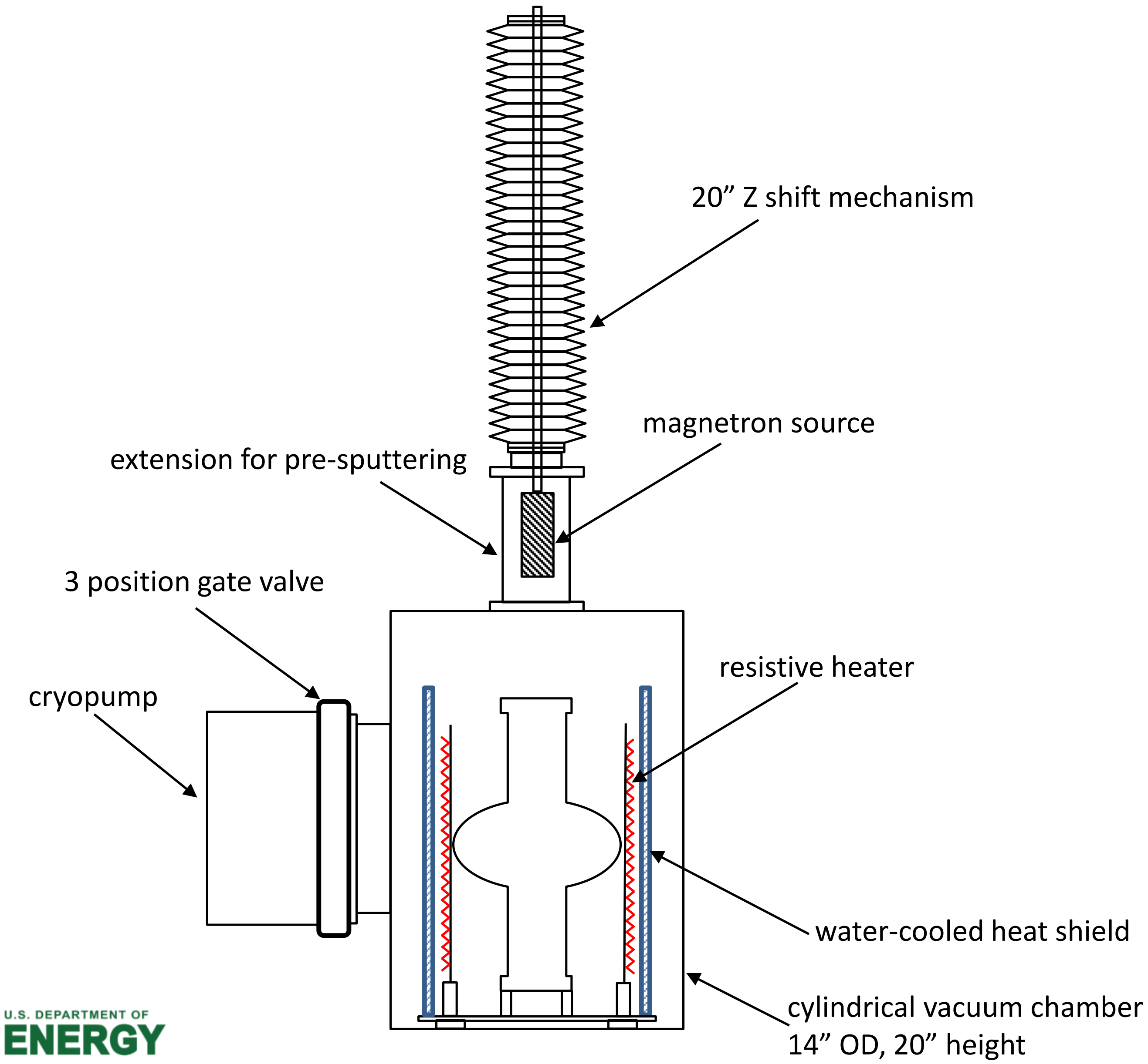
Bronze route for Cu substrates



Route 1 for cavities

Route 2 for cavities

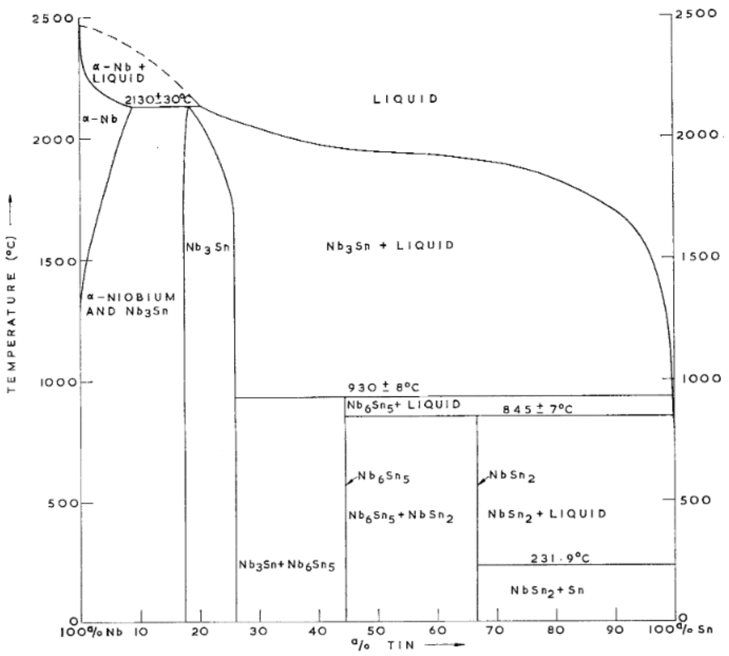
Proposed deposition chamber



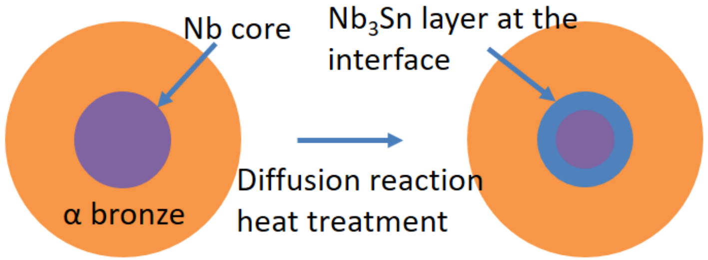
More introduction

- To synthesis pure Nb_3Sn by reacting Nb and Sn directly require temperatures above 930 °C.
- Eliminates the use of Cu substrate cavity in Nb – Sn direct reaction -> Significant material costs
- Require sophisticated furnace systems for Nb – Sn reaction.
- Bronze route is a well-established technique in Nb_3Sn wire fabrication.
- Bronze route guarantees only the pure Nb_3Sn phase at much lower temperatures (~600 – 800 °C).

Nb – Sn phase diagram

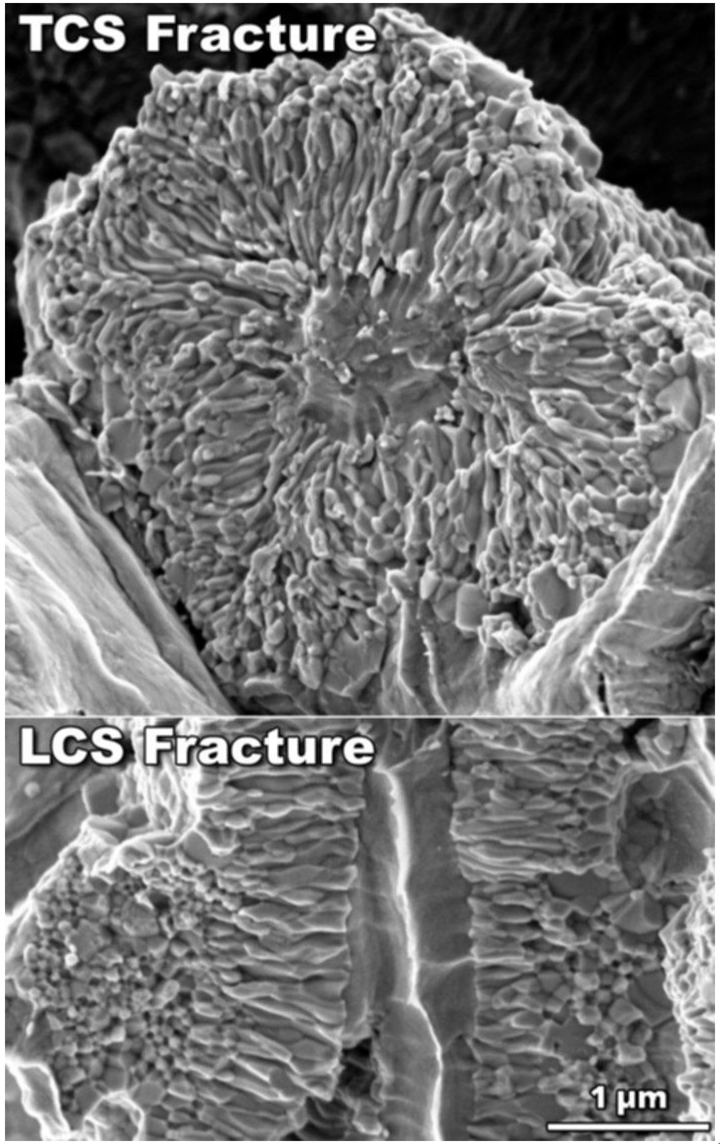


J. P. Charlesworth *et al J.* Mat. Sci.5, 580 (1970)



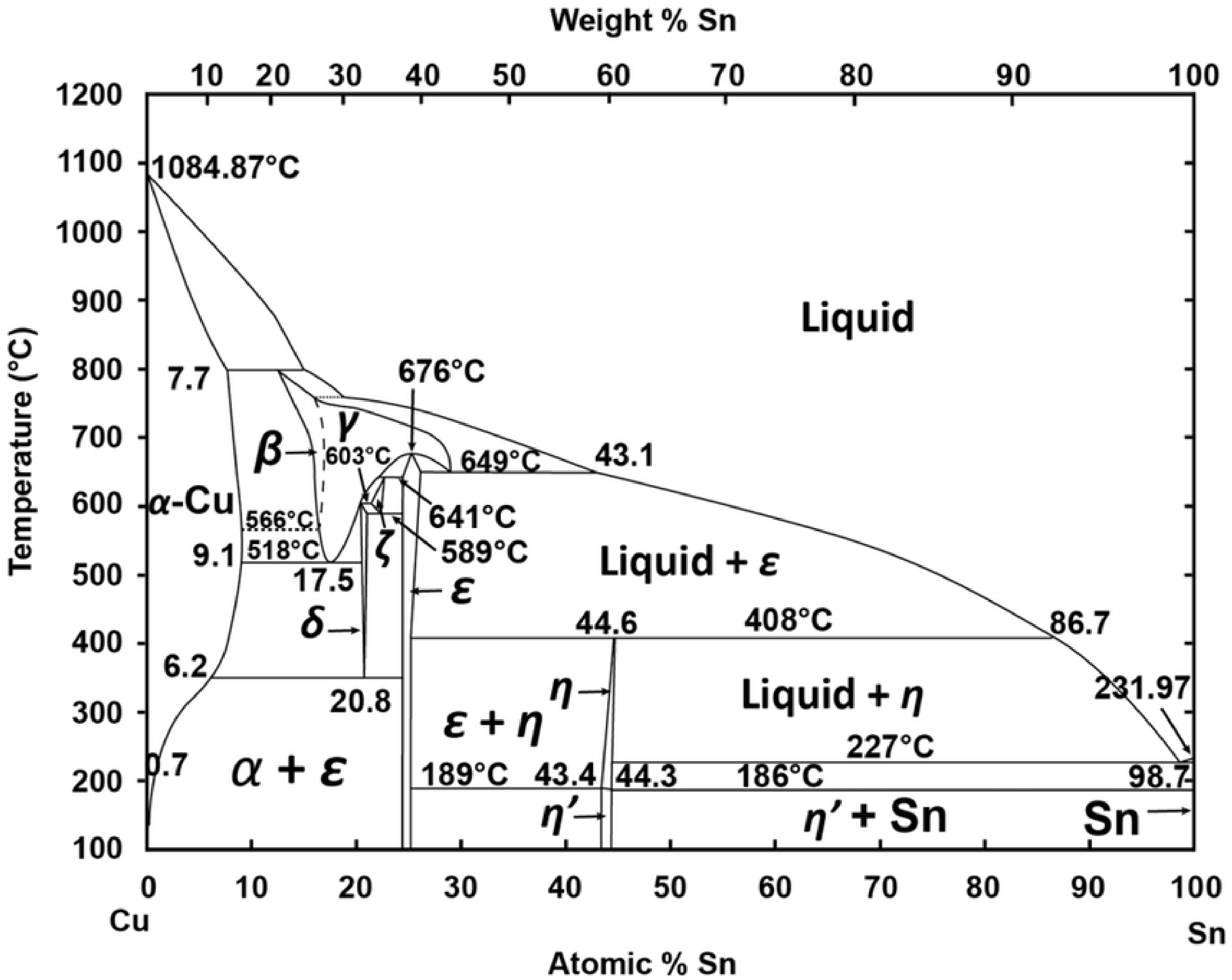
Bronze process

Fractographs of a bronze route Nb_3Sn filament

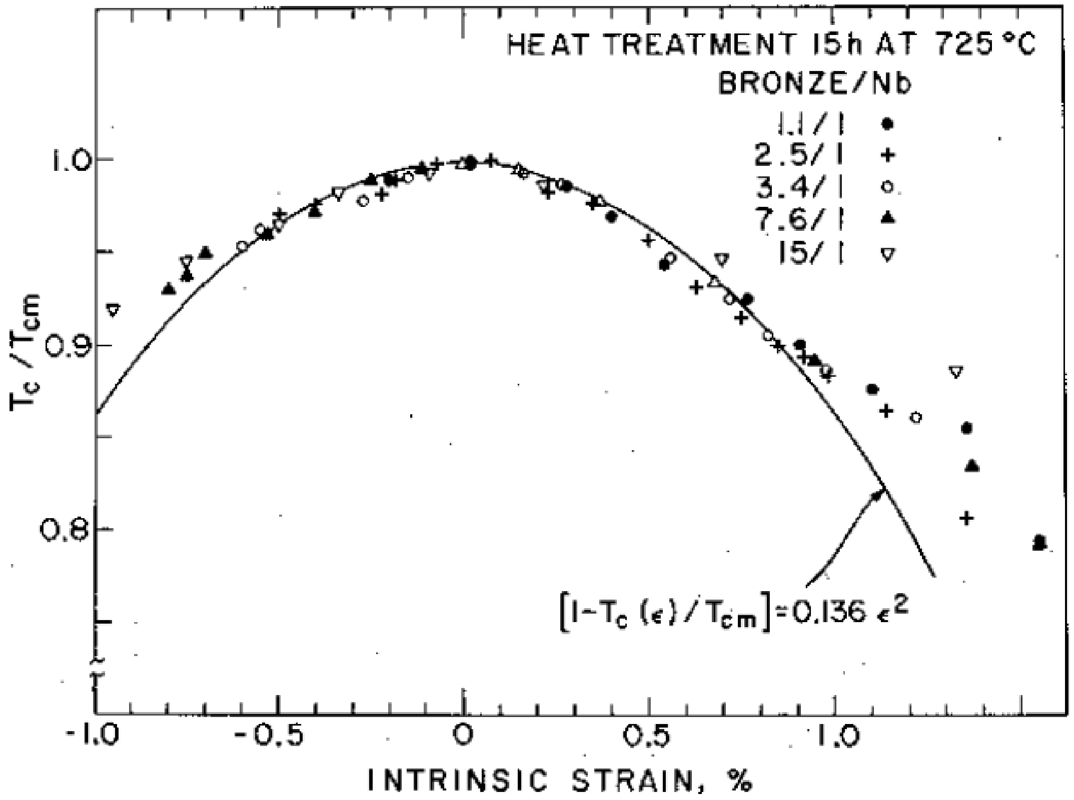


P.J. Lee, D.C. Larbalestier, *IEEE Transactions* 11 (2001)

Cu-Sn phase digram



CTE mismatch



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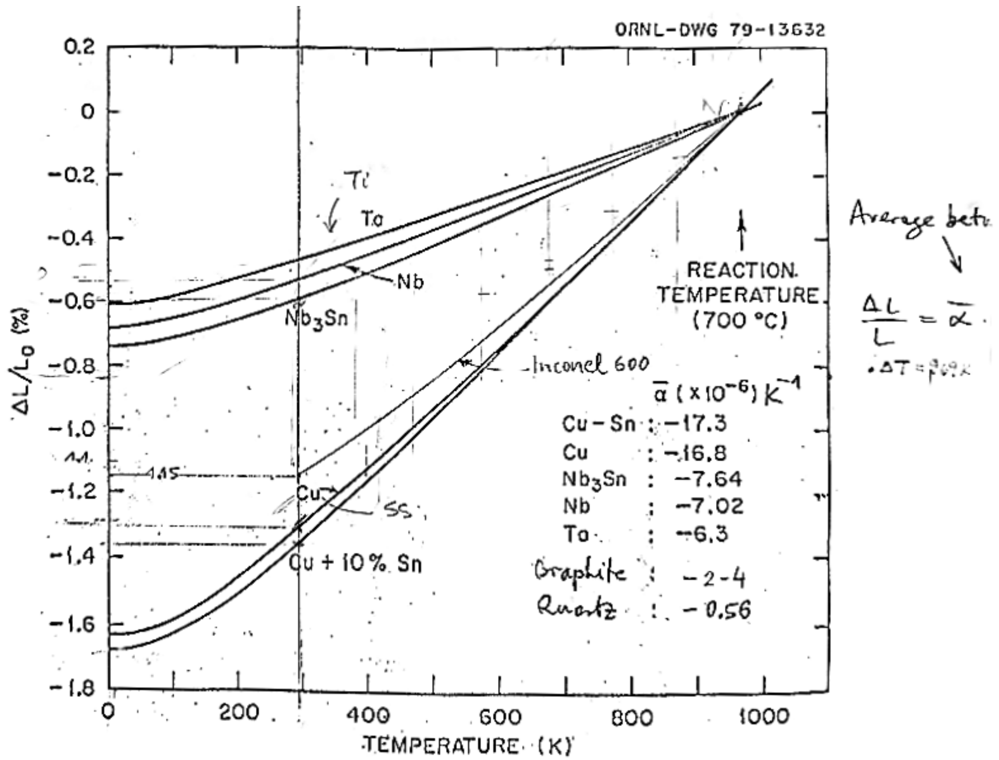

CTE mismatch between Nb_3Sn and common substrates