



# Nb<sub>3</sub>Sn Thick Layers synthesized via Bronze Route

Approach from the Nb/Bronze Composite Wire Process

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# Collaborators



- Dr. Kensei Umemori
- Dr. Hiroshi Sakai
- Dr. Eiji Kako, and many others.



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- Mr. Yasunari Mizuta
- Dr. Taiji Mizuta



TAKEFU SPECIAL STEEL CO., LTD.

- Mr. Tsubasa Tsubokawa

# Diffusion pass for synthesis of Nb<sub>3</sub>Sn

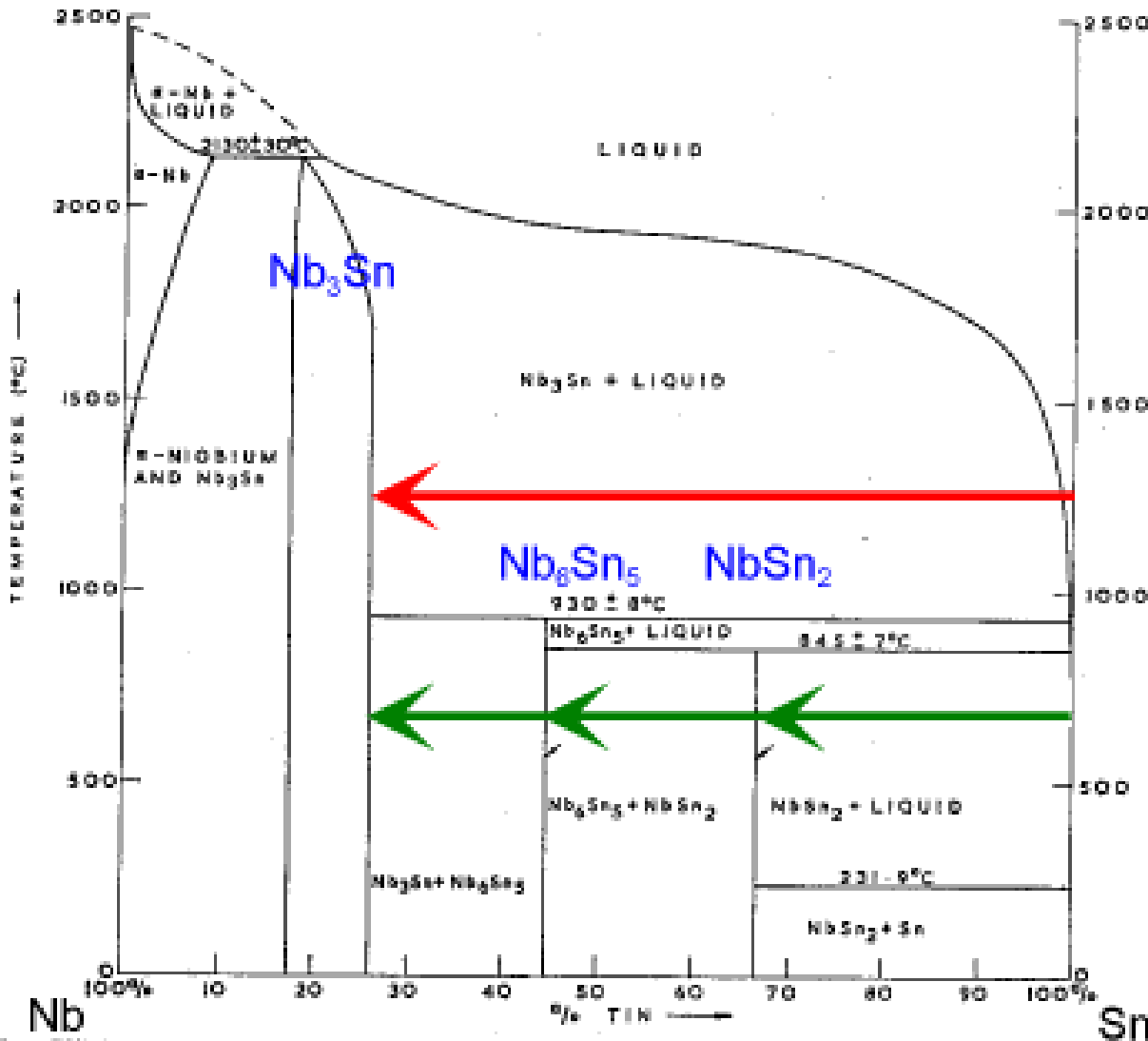


Figure 5 Niobium-tin equilibrium diagram.

## High Temperature Reaction

Single Nb<sub>3</sub>Sn phase can be obtained. But, Nb<sub>3</sub>Sn grain is very large. Since the grain boundary acts as the flux pinning center, its critical current density, J<sub>c</sub> is very low.

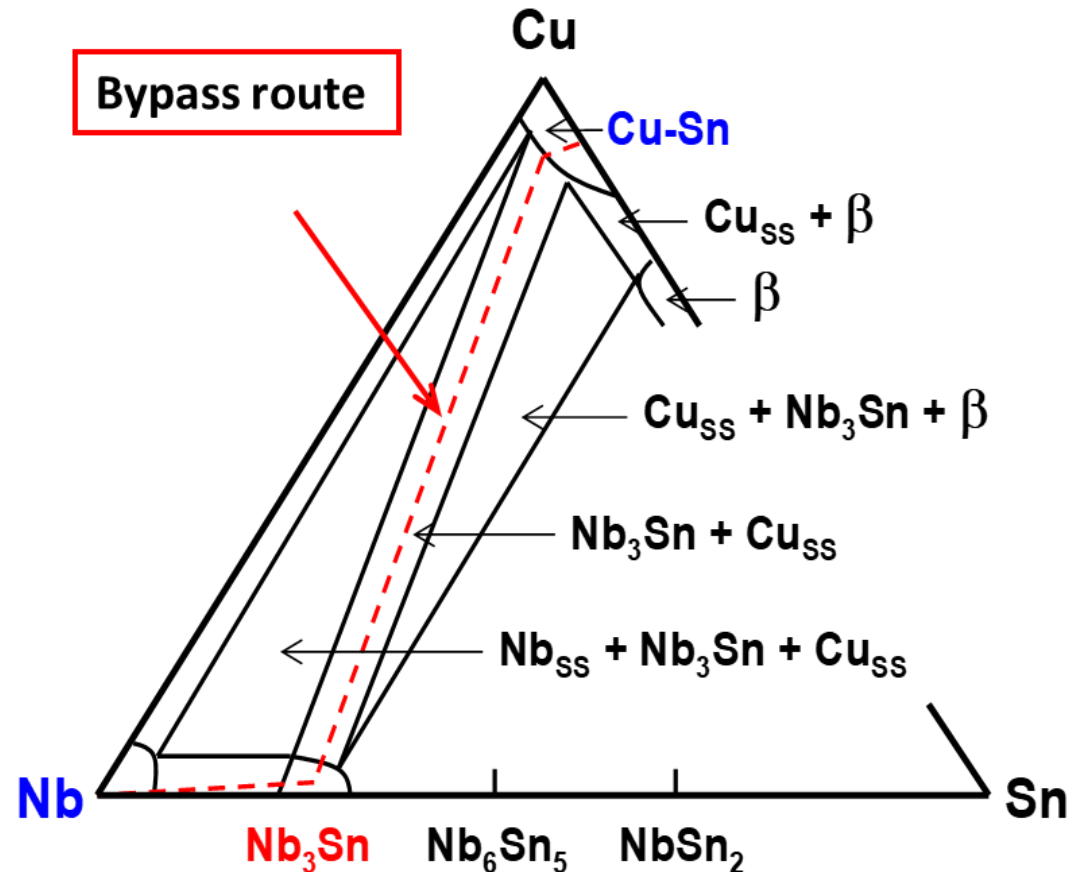
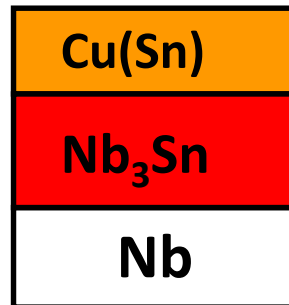
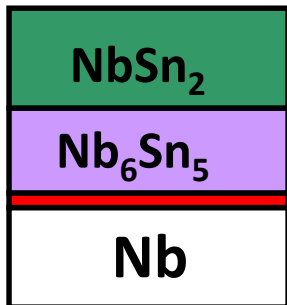
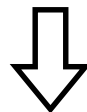
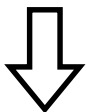
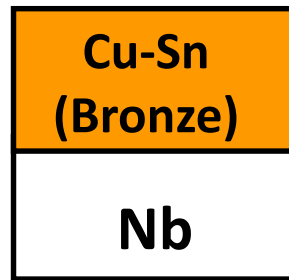
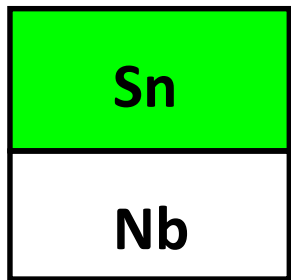
## Low Temperature Reaction

Non superconducting NbSn<sub>2</sub> and Nb<sub>6</sub>Sn<sub>5</sub> intermediate phases dominantly form with a small amount of Nb<sub>3</sub>Sn phase. As a result, J<sub>c</sub> is still very low.

# Discovered Effect of Cu in 1967



Bypass Route for Direct Forming of  $\text{Nb}_3\text{Sn}$  Phase at low temperature diffusion reaction. (*Tachikawa Method*)



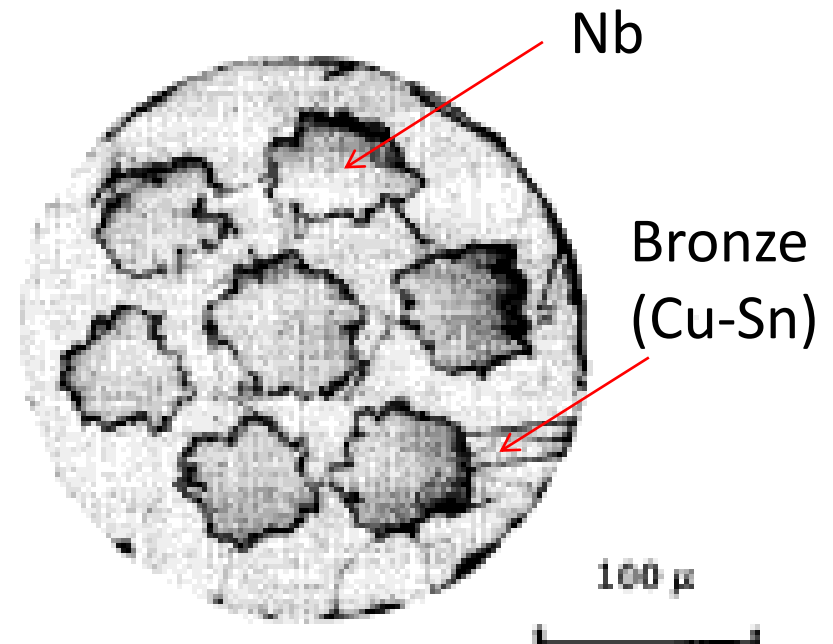
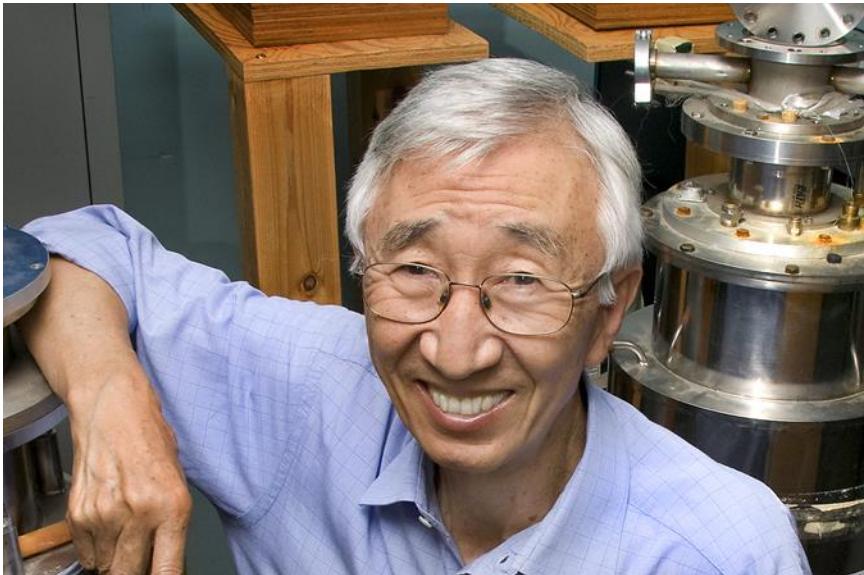
# Nb<sub>3</sub>Sn Multifilamentary Wires (1972)

## Superconducting Properties of Multifilamentary Nb<sub>3</sub>Sn Made by a New Process\*

M. Suenaga and W. B. Sampson†

*Metallurgy and Materials Science Division, Brookhaven National Laboratory, Upton, New York 11973*  
(Received 6 January 1972; in final form 13 March 1972)

A new processing method for producing multifilamentary A-15 superconducting composite wires in a ductile matrix is described. Nb<sub>3</sub>Sn made in this way was found to have a critical temperature of 17.5 K and a critical current density of  $7.6 \times 10^3$  A/cm<sup>2</sup> at 40 kG when suitably heat treated. The composite conductor could be bent with a radius of 2 cm or greater without appreciable degradation of its current-carrying capacity.

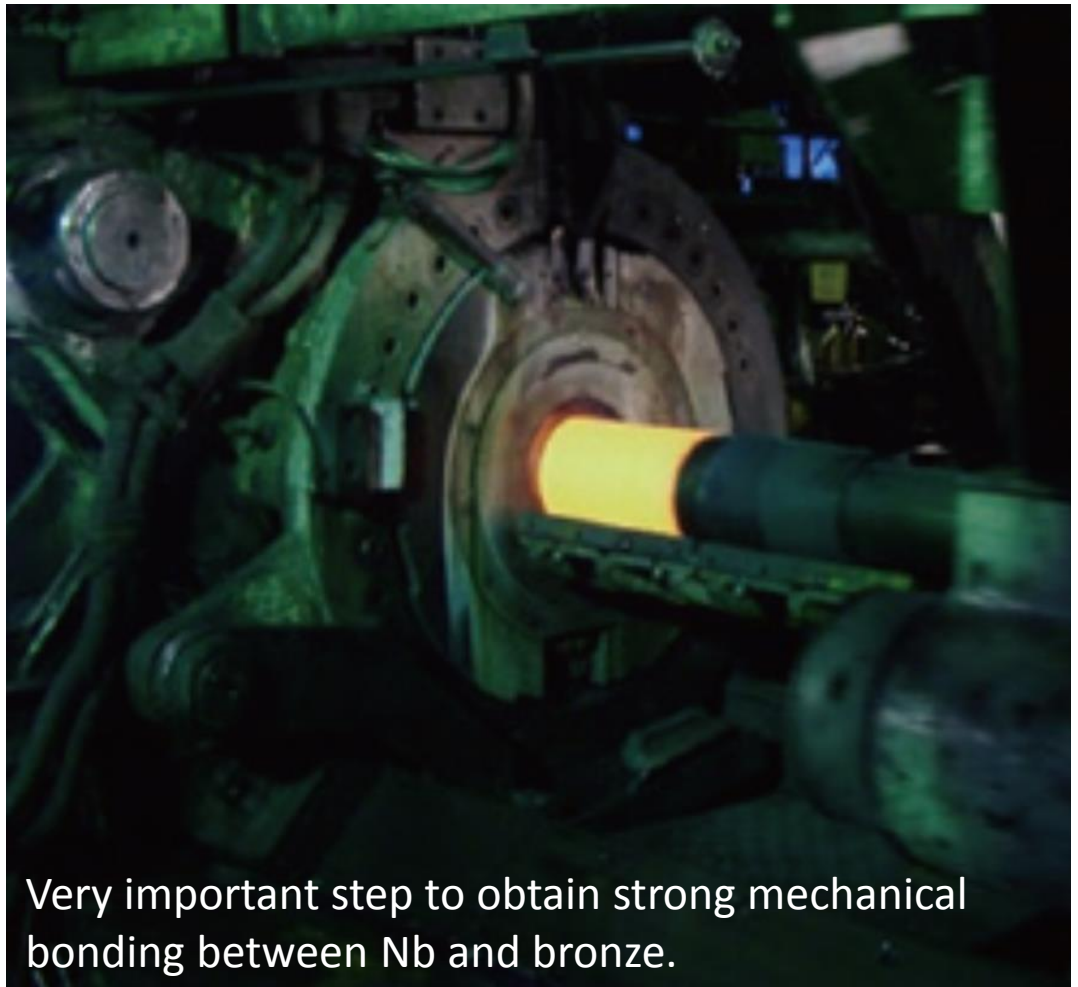


# Production Scheme of Nb<sub>3</sub>Sn wires

Billet Assembly  
(with EB welding)



Hot Extrusion



# Production Scheme of Nb<sub>3</sub>Sn wires

Cold-Die-Drawing

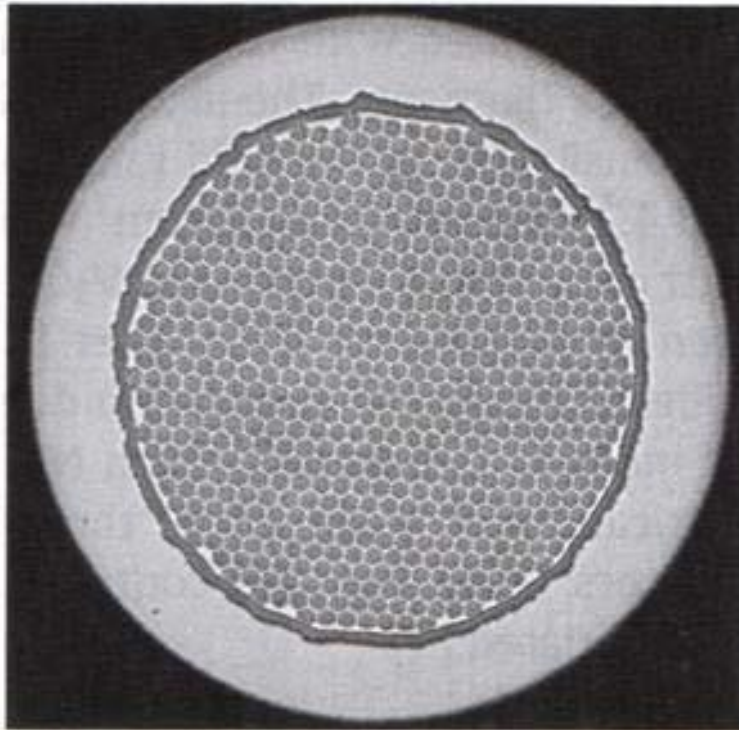


Intermediate annealing

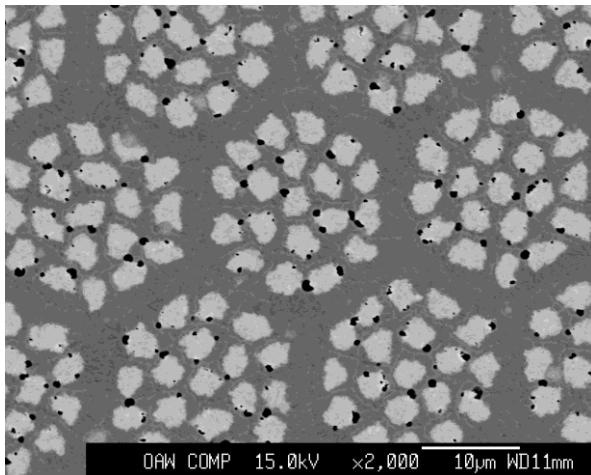


Bronze shows significantly work hardening. In order to keep well-balanced hardness between Nb and bronze, frequent intermediate annealing is needed in cold die drawing.

# Typical Nb<sub>3</sub>Sn Multifilamentary Wires



Wire diam. (with Cr plating)	0.826 mm
Twist pitch	15.0 mm
Bronze composition (wt%)	Cu-15.5 Sn-0.3Ti
Filament composition (wt%)	Nb-1.0 Ta
Filament diameter	3.0 μm
No. Filament	19×583=11,077
Cu/non Cu ratio	1.0
Barrier material	Nb
non Cu J <sub>c</sub> @ 12 T, 4.2 K	820 A/mm <sup>2</sup>
n-value @ 12 T, 4.2 K	30.0
Hysteresis loss @ ± 3 T, 4.2	620kJ/m <sup>3</sup>

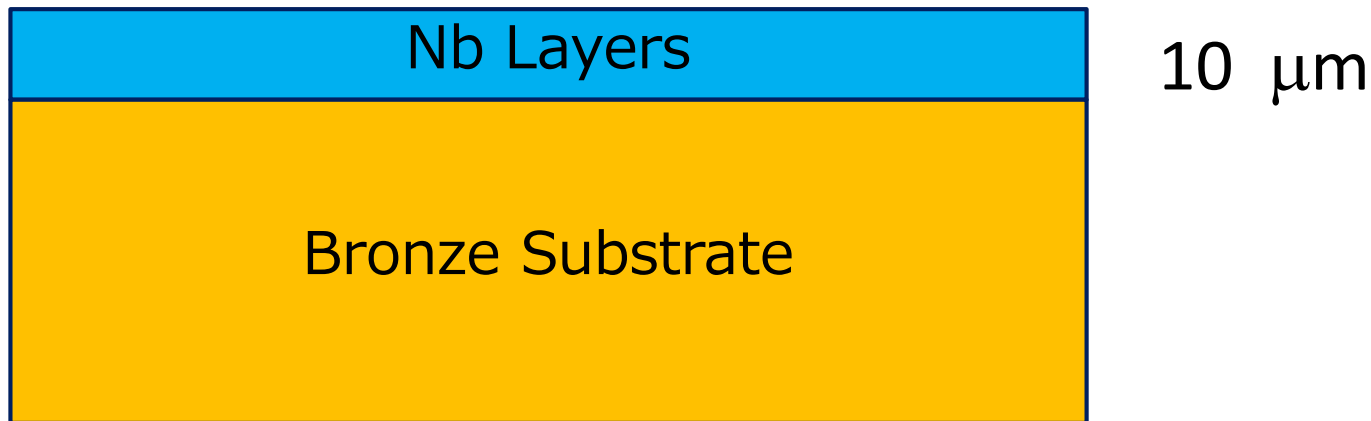


From the achievements on the fabrication of multifilamentary wires, we can say that it is possible to control the Nb thickness on the bronze substrate through the plastic deformation of Nb/Bronze composite.

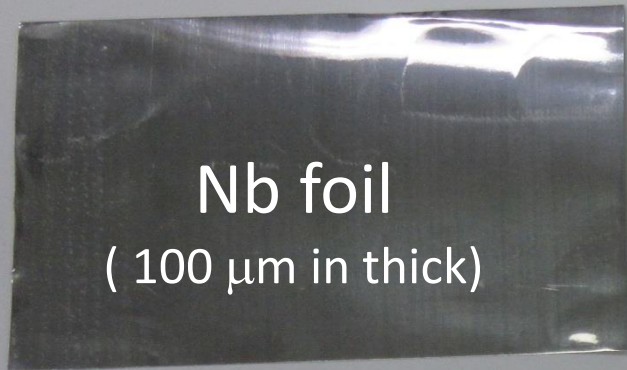


# 1. Combination of Hot Pressing and Cold Flat Rolling

(Fabrication of Nb/Bronze Composite Plate)



# Starting Materials



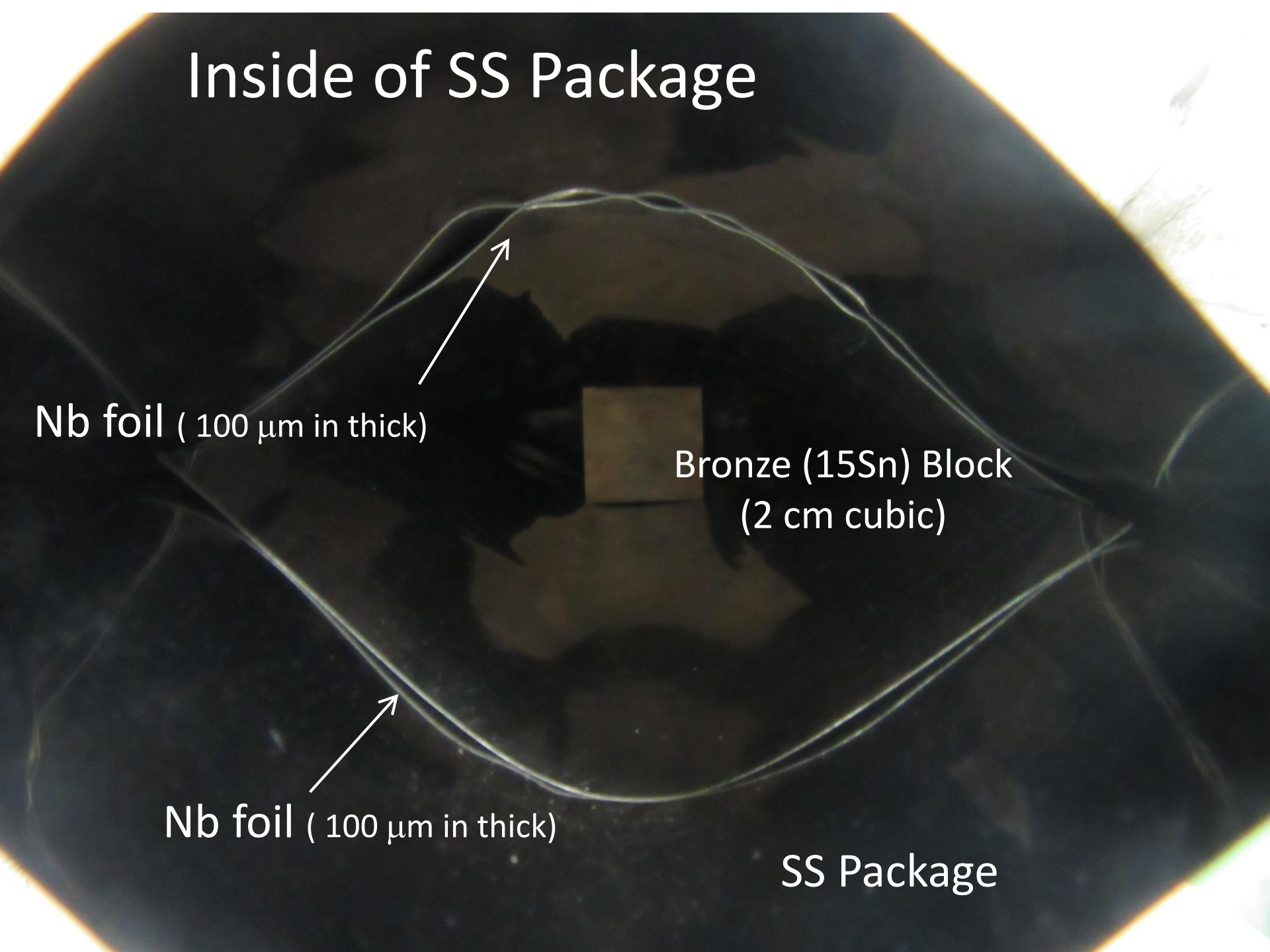
# Inside of SS Package

Nb foil ( 100  $\mu\text{m}$  in thick)

Bronze (15Sn) Block  
(2 cm cubic)

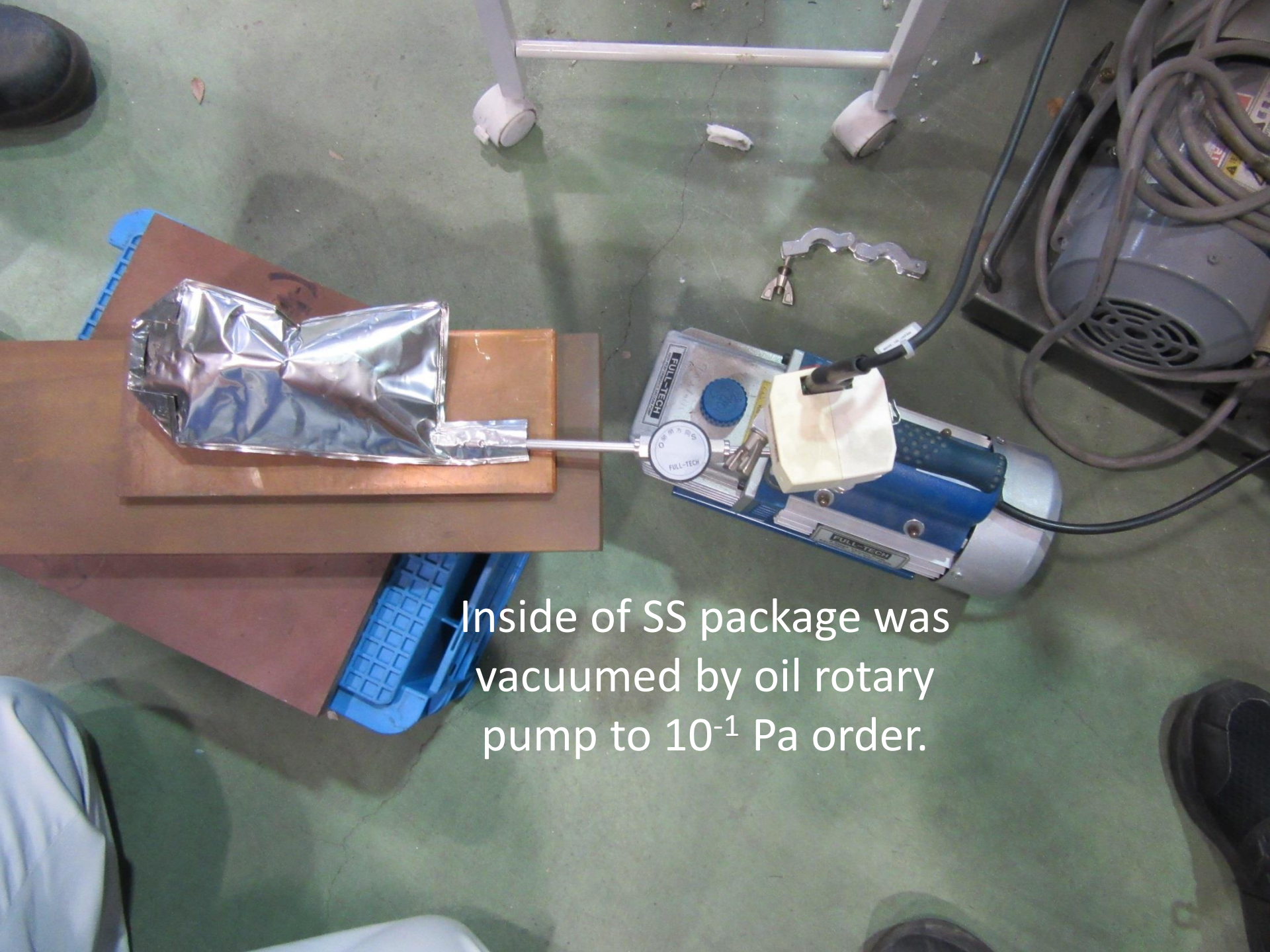
Nb foil ( 100  $\mu\text{m}$  in thick)

SS Package





The wide end of SS package was sealed by a spot welding .



Inside of SS package was vacuumed by oil rotary pump to  $10^{-1}$  Pa order.

# 300 ton Press at NIMS



The Package was heated up to 720 °C before pressing



# Cold Flat Rolling to Tape Shape



Cut to appropriate size

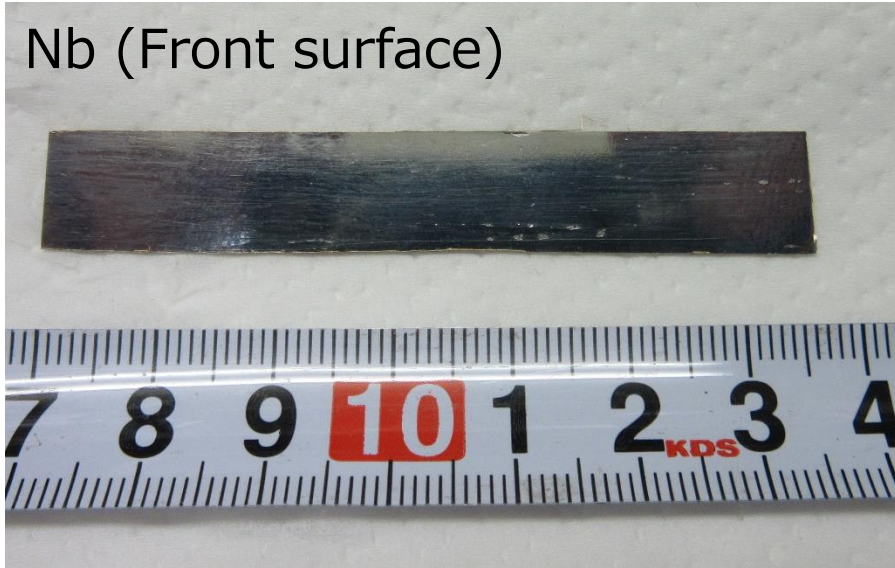


Flat rolling at room temperature

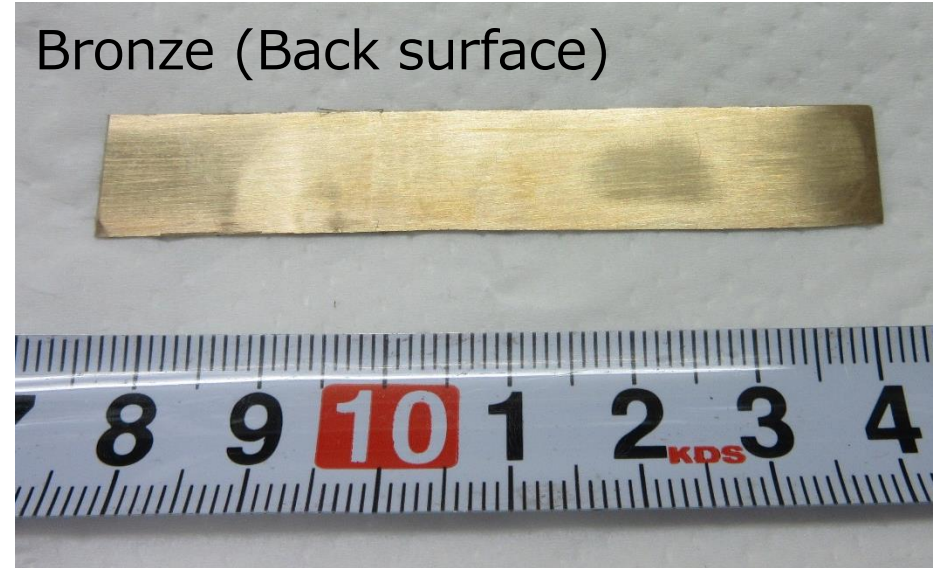


# 0.4 mm thick Nb/Bronze (Cu-15Sn) Clad Tape - Appearance -

Nb (Front surface)



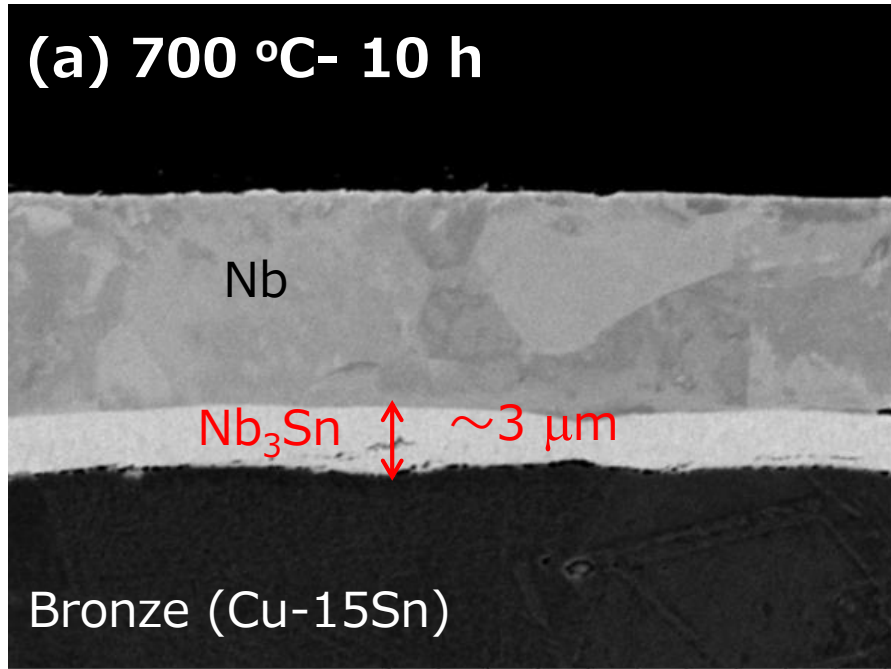
Bronze (Back surface)



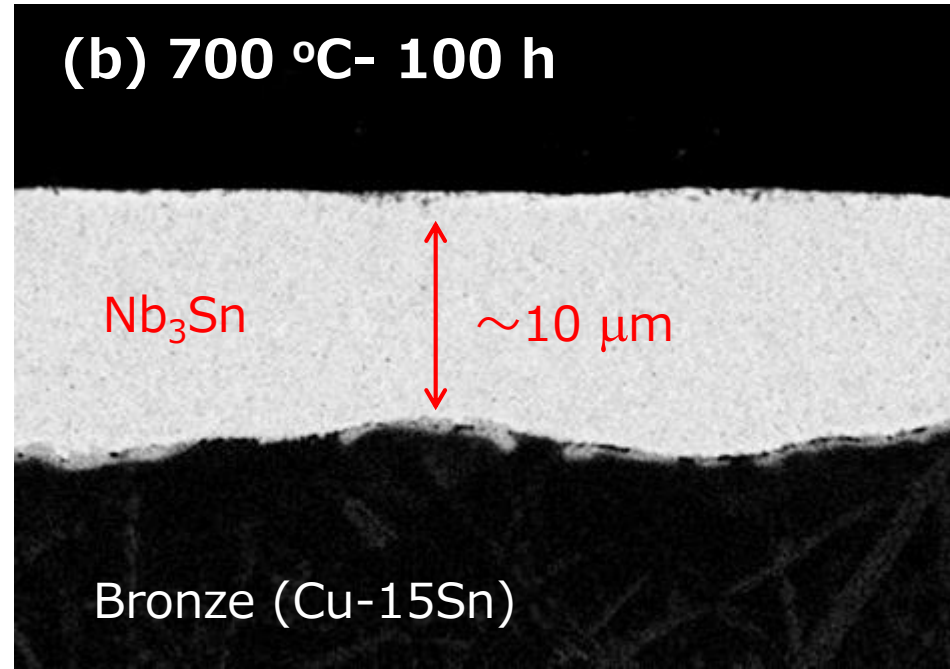


# Heat Treatment for Nb<sub>3</sub>Sn Formation

(a) 700 °C- 10 h



(b) 700 °C- 100 h

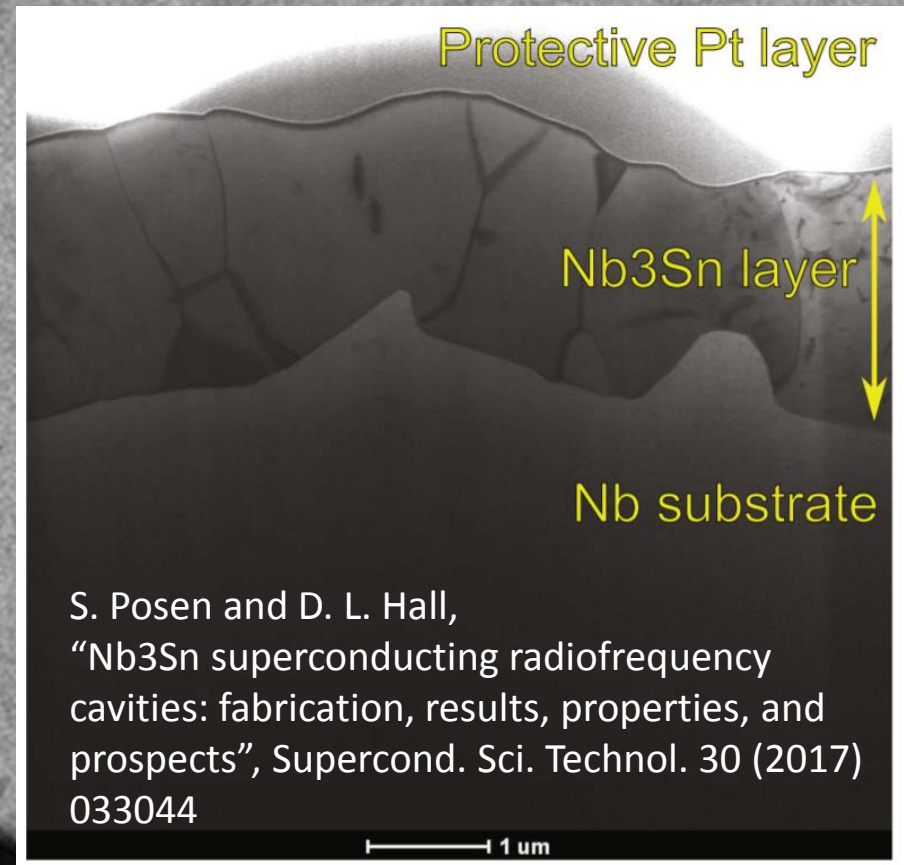


N D4.8 x3.0k 30 μm

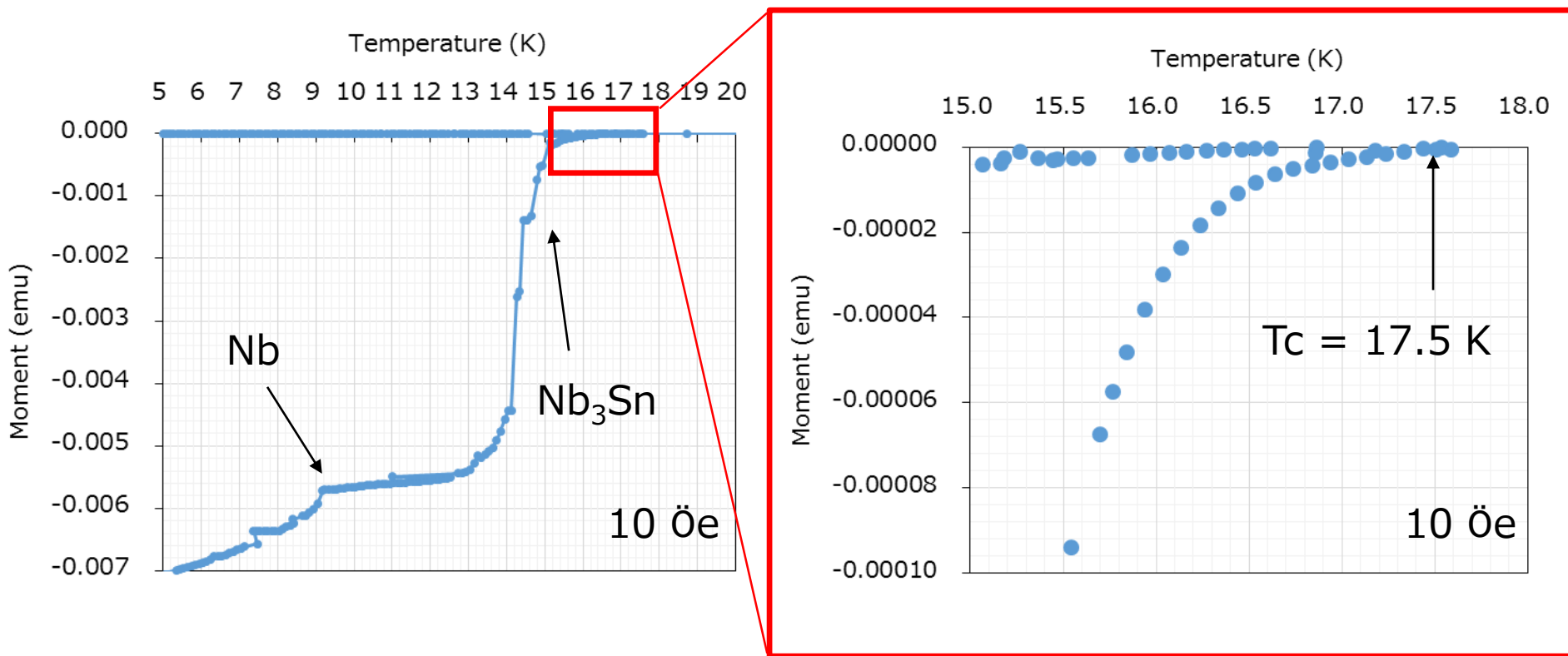
# Small Grain Size and Uniform Microstructure

**700 °C- 100 h**

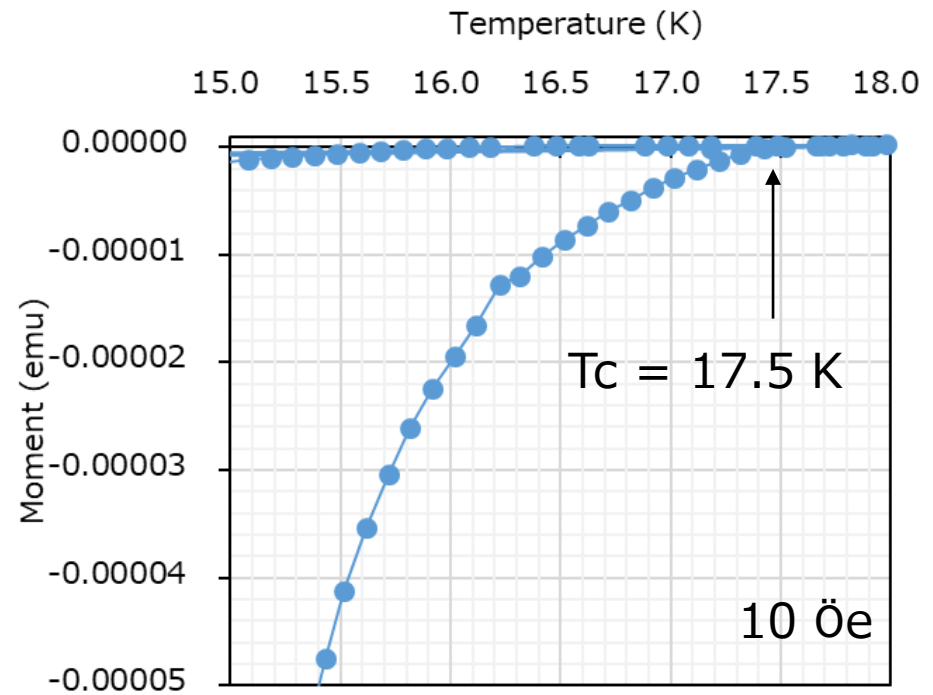
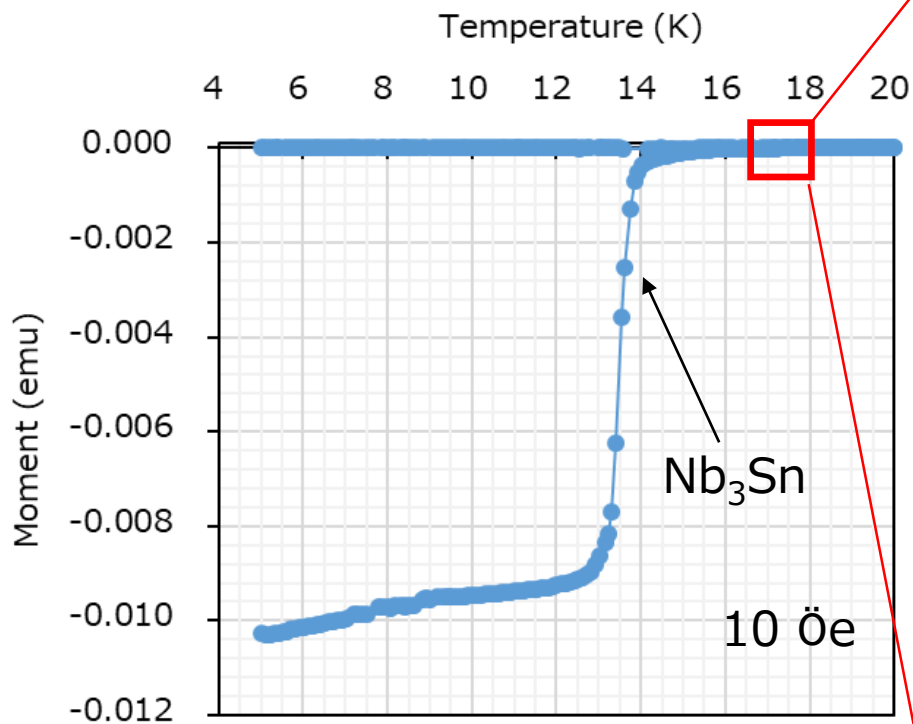
1  $\mu\text{m}$



# Partial Reacted at 700 °C for 10 h

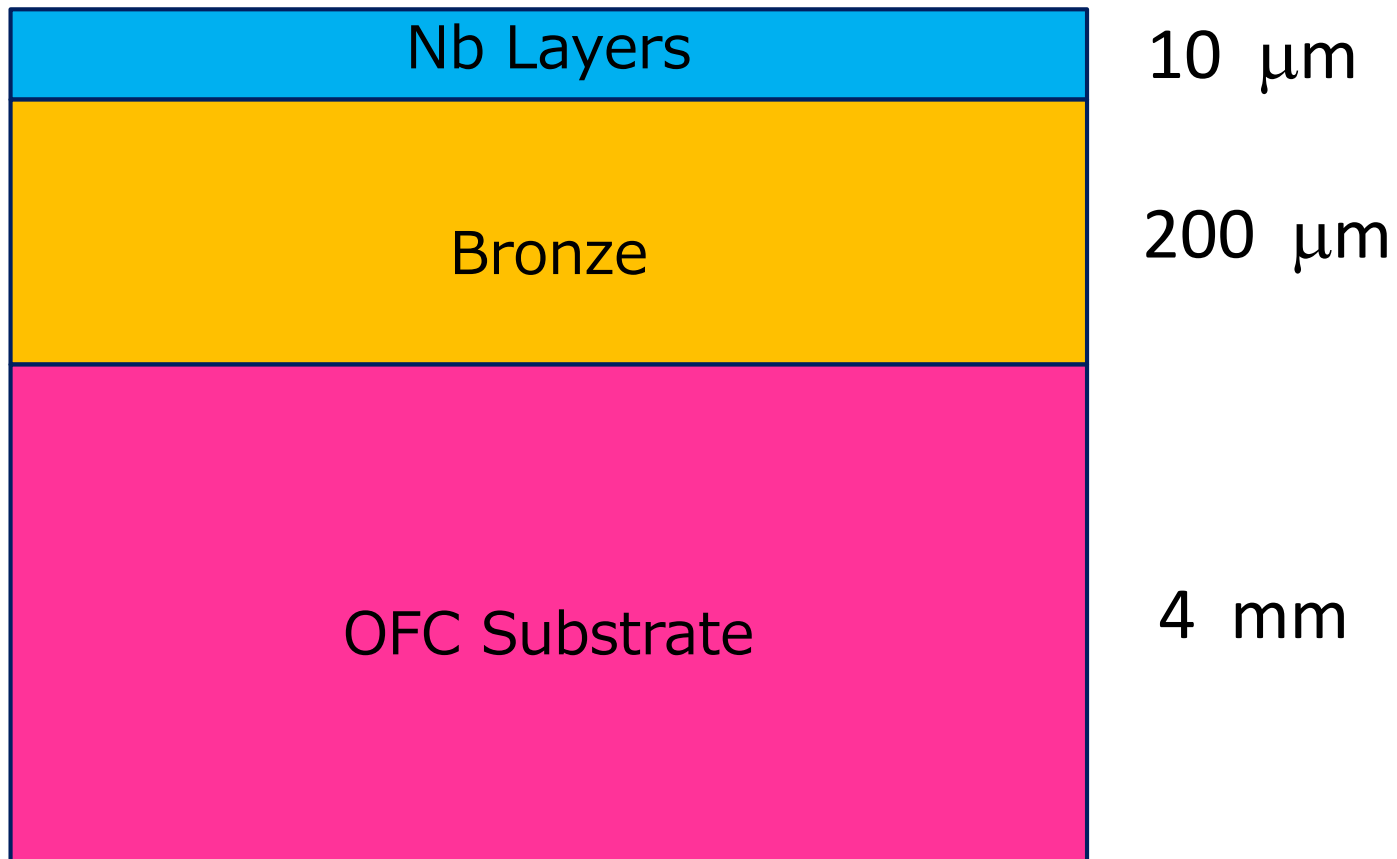


# Full Reacted at 700 °C for 100 h



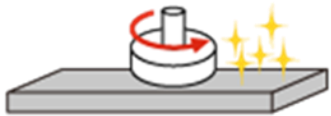
## 2. Combination of Hot Flat Rolling and Cold Flat Rolling

(Fabrication of Nb/Bronze/OFC Composite Plate)



# Production Scheme of Nb/Bronze/OFC Clad Plate

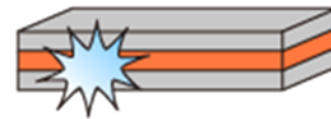
1. Surface Polishing



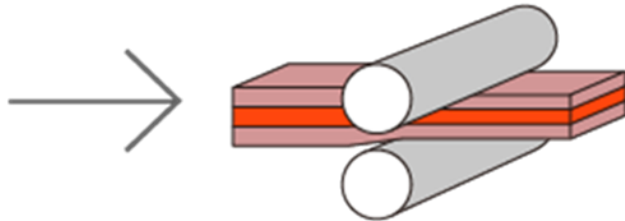
2. Assembling



3. EB Welding

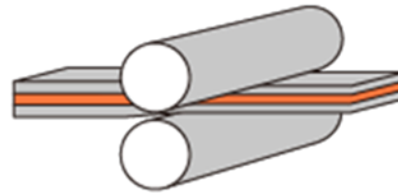


4. Hot Flat Rolling



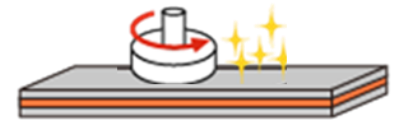
Hot Extrusion  
(for wire's process)

5. Cold Flat Rolling



Cold Die Drawing  
(for wire's process)

6. Mirror Polishing

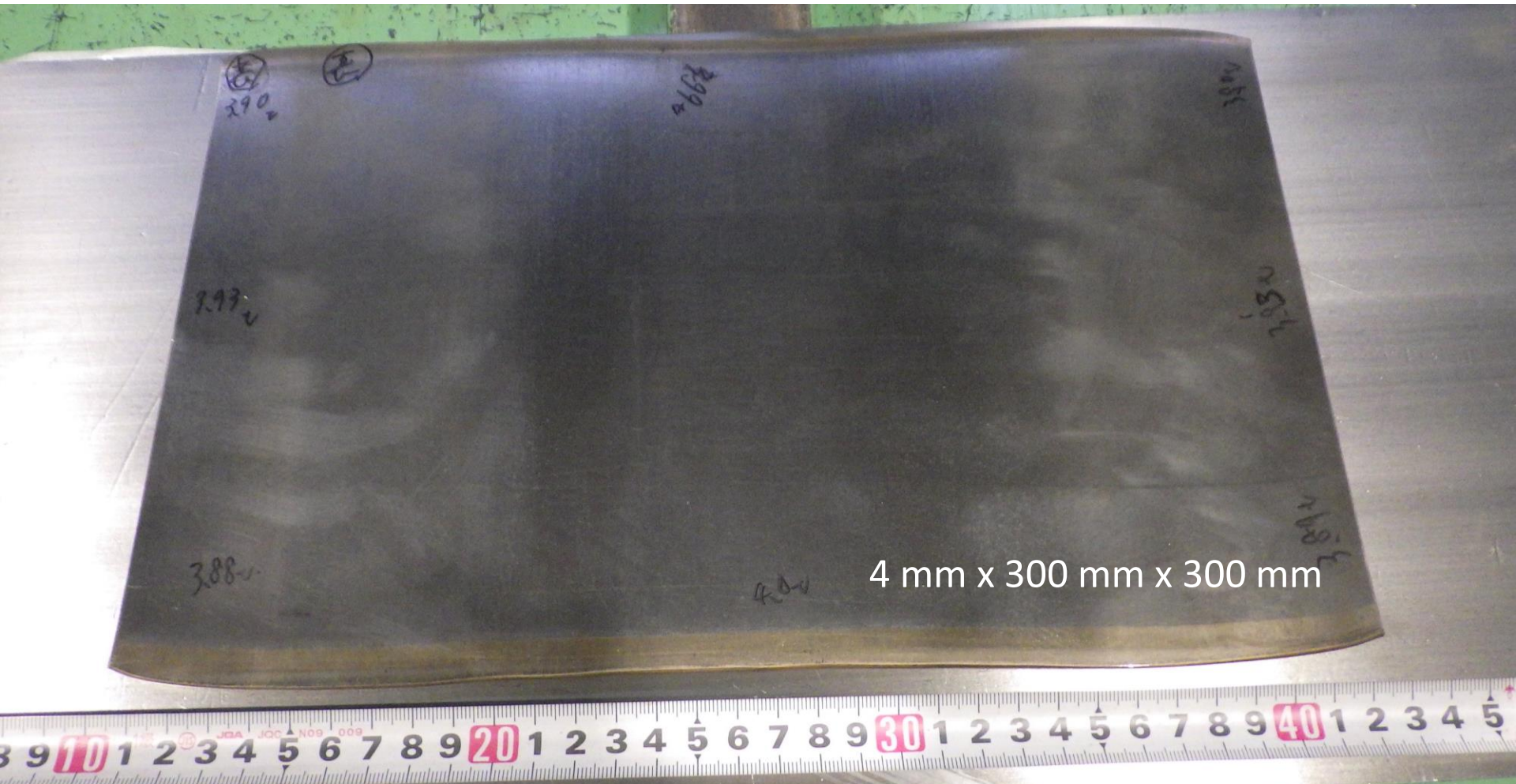


# Hot Flat Rolling for Mechanical Cladding



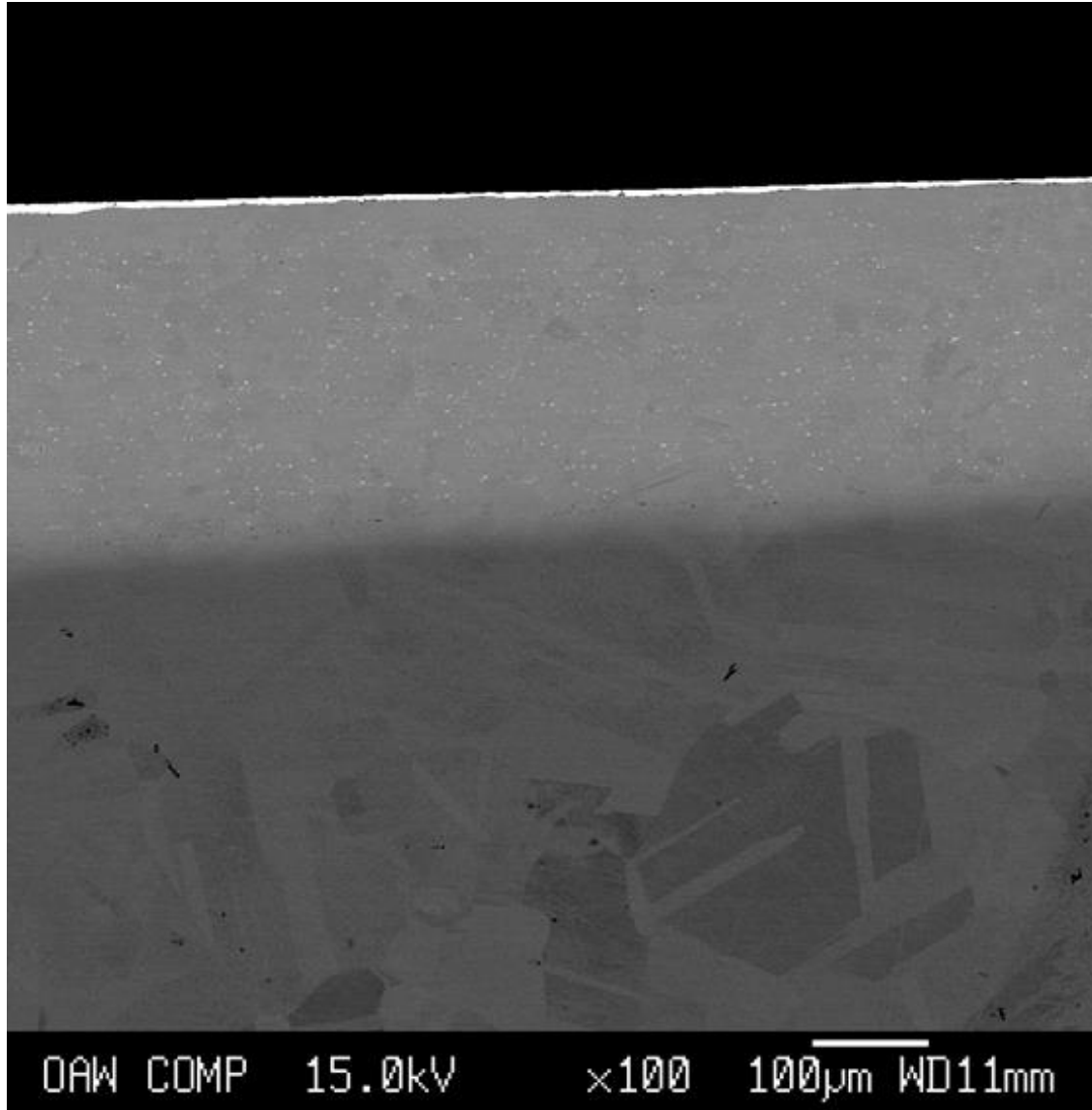
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# 4mm thick Nb/Bronze/OFC Clad Plate (Before Mirror Polishing)





# Nb/Bronze/OFC Clad Plate



Nb (10  $\mu\text{m}$ )

Bronze 14Sn (300  $\mu\text{m}$ )

OFC (4 mm)

# One estimation for Production of Cavity

Nb/Bronze/OFC  
Clad Tube

Hydro-Forming for Cavity



# Summary

- Nb/Bronze and Nb/Bronze/OFC composite plates could be fabricated by plastic deformation process, such as pressing and rolling.
- 10 microns thick and homogeneous Nb<sub>3</sub>Sn layers were easily synthesized through a bronze route with a low temperature diffusion reaction at 650 – 750 °C.
- Nb<sub>3</sub>Sn grain size (a few nm) is much smaller than that (a few microns) formed on the Sn vapor processed samples.
- Rs and other interesting measurements would be expected to perform by new collaborators in the near future.