

# Development of Nb<sub>3</sub>Sn in Japan

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K. Miyashita, Y. Suzuki SH Copper Products.,  
K. Saito, S. Kawashima, Y. Fukumoto Kobe Steel and JASTEC  
H. Sakamoto, T. Fukushima, H. Shimizu Furukawa Electric

# Scope:

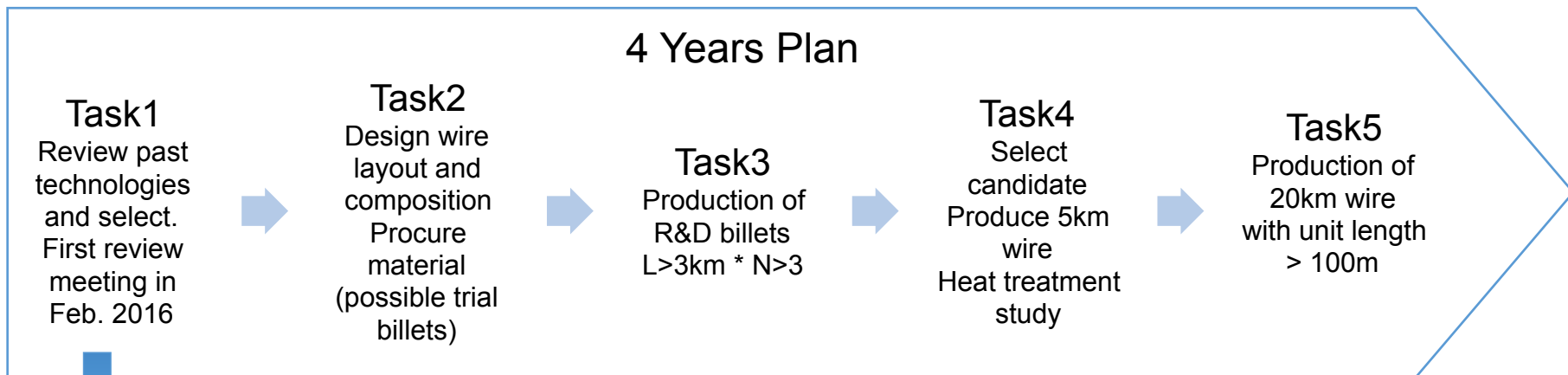
CERN, KEK and Tohoku university have jointly launched a R&D program

- The scope of the program is to develop, produce in representative lengths and characterize Nb<sub>3</sub>Sn wire with enhanced characteristics.
- The final goal is to achieve in representative unit lengths of material the development targets defined, on the basis of magnets performance, for the FCC Nb<sub>3</sub>Sn conductor:

# Final Target Properties

- A non-copper critical current density at 4.2 K and 16 T ( $J_c(4.2 \text{ K}, 16 \text{ T})$ ) of at least  $1500 \text{ A/mm}^2$ ;
- A wire diameter of not more than 1 mm;
- A fraction of stabilizer to superconductor in the wire of at least 1;
- An equivalent diameter of the superconducting  $\text{Nb}_3\text{Sn}$  filaments of less than  $50 \text{ }\mu\text{m}$ ;
- A low electrical resistivity of the copper stabilizer of the wire, i.e. a Residual Resistivity Ratio (RRR) of the copper after wire reaction of above 150.

# R&D Plan



**Workshop on High Jc Nb<sub>3</sub>Sn conductor development in Japan**

Monday 22 February 2016 - Wednesday 24 February 2016

KEK, Tsukuba, Japan  
**Programme**

## Tuesday 23 February 2016

**Tour to SH Copper Products (by KEK, CERN, and Tohoku Univ.) - (08:30-12:30)**

**Closed session discussion with each company: Closed session discussion with each company - 102-1F, Ni-Go-Kan Bldg. (13:30-15:40)**

time	[id] title	presenter
13:30	[10] SH Copper Products	
14:40	[11] Furukawa Electric	

**Discussion for Development Strategies - 102-1F, Ni-Go-Kan Bldg. (16:00-18:00)**

**Closing - 102-1F, Ni-Go-Kan Bldg. (18:00-18:10)**

## Monday 22 February 2016

**Registration - 102-1F, Ni-Go-Kan Bldg. (13:00-13:15)**

time	[c] title	presenter
13:00	[12] Information	

**Introduction - 102-1F, Ni-Go-Kan Bldg. (13:15-15:00)**

time	[c] title	presenter
13:15	[1] Welcome and workshop scope	Prof. OGITSU, Toru
13:30	[2] FCC CERN-KEK development program	Dr. BALLARINO, Amalia
14:00	[3] Possible contribution from Tohoku Univ.	Prof. AWAJI, Satoshi
14:30	[4] Test plan	Dr. SUGANO, Michinaka Dr. OGURO, Hidetoshi

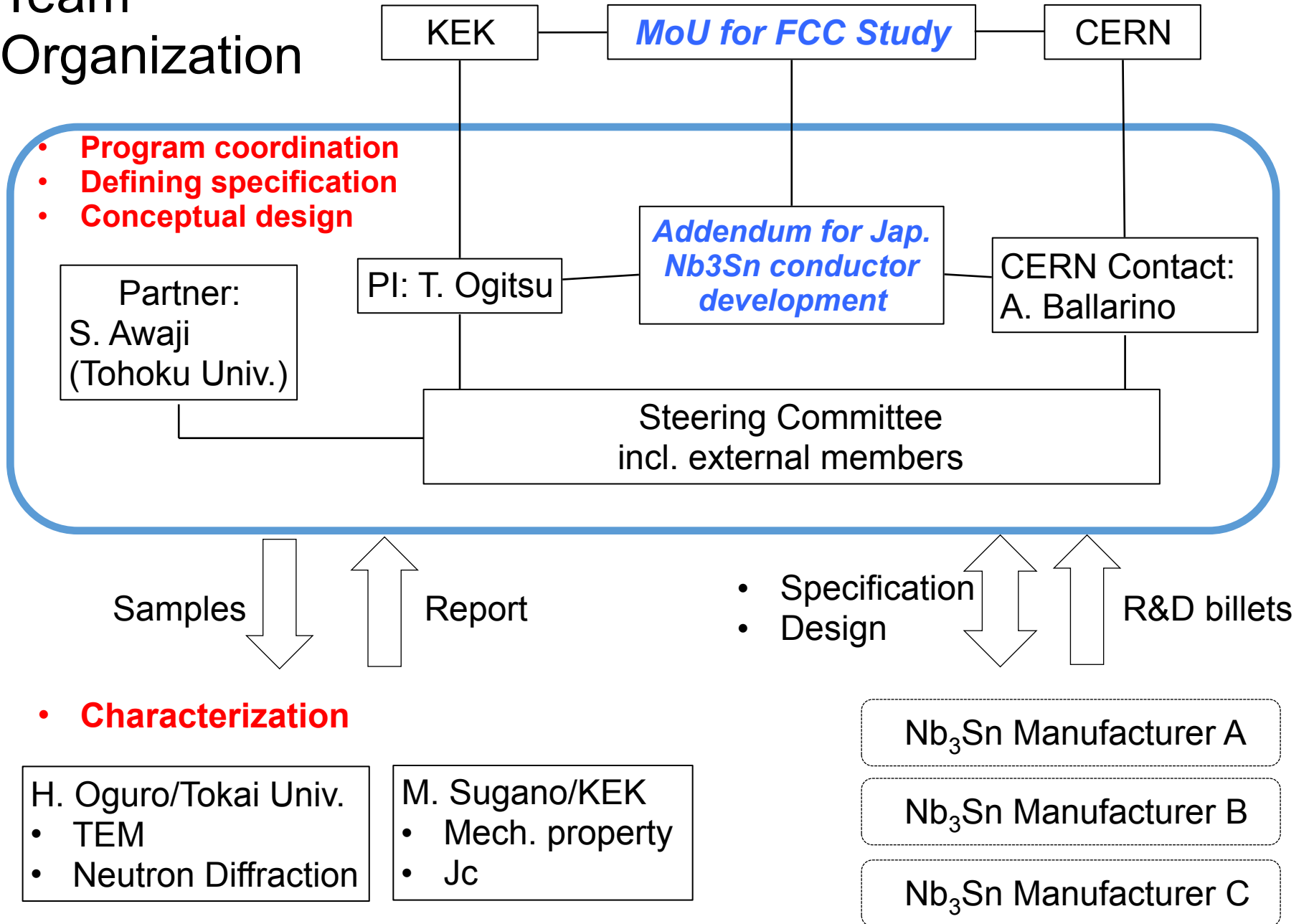
**Development status and plan at companies - 102-1F, Ni-Go-Kan Bldg. (15:15-16:50)**

time	[c] title	presenter
15:15	[5] Development at SH-Copper	
15:40	[6] Development at Furukawa	
16:05	[7] Development at JASTEC/Kobelco	
16:30	[8] Discussion	

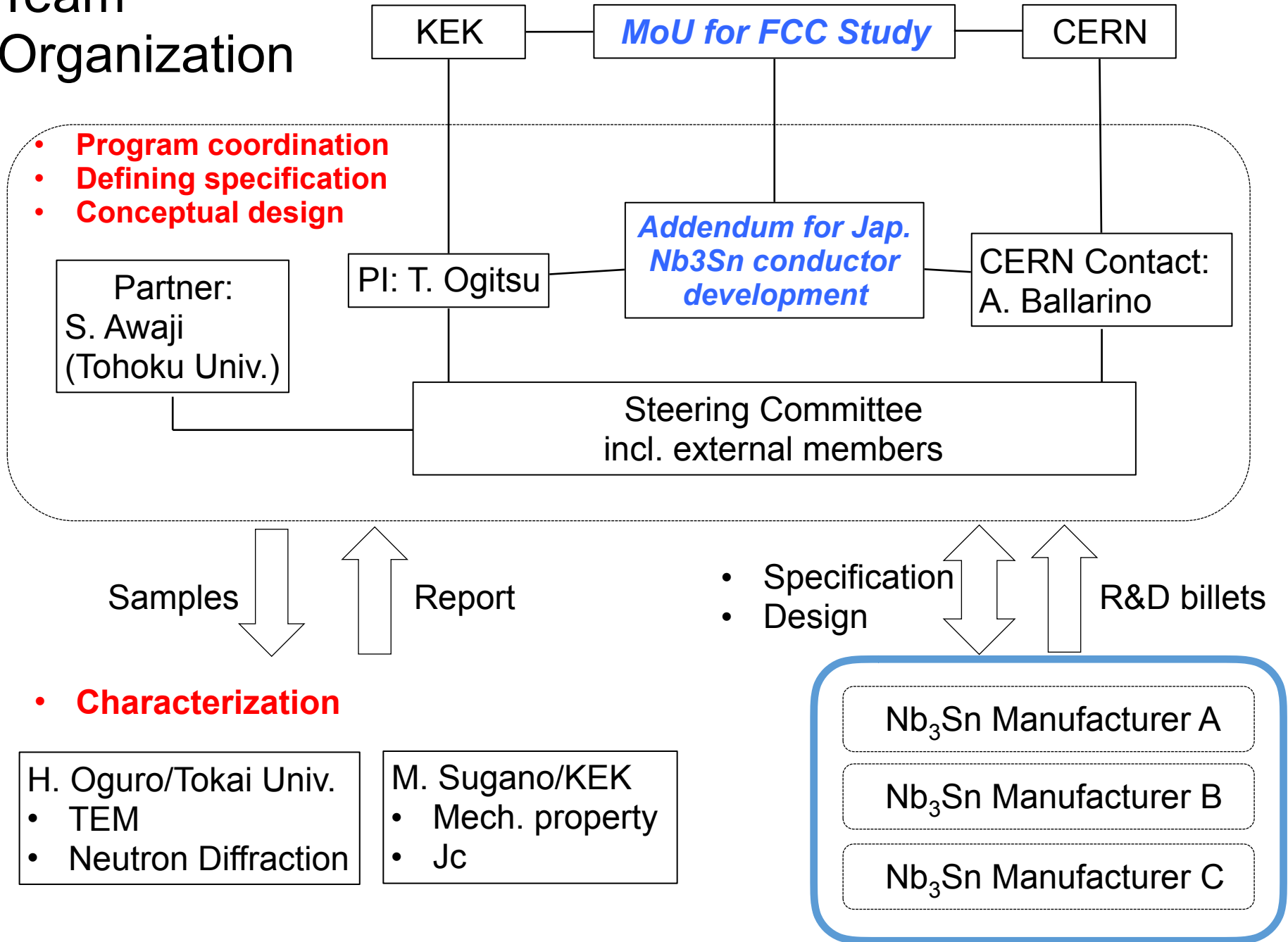
**Closed session discussion with each company - 102-1F, Ni-Go-Kan Bldg. (17:00-18:00)**

time	[c] title	presenter
17:00	[9] JASTEC/Kobelco	

# Team Organization

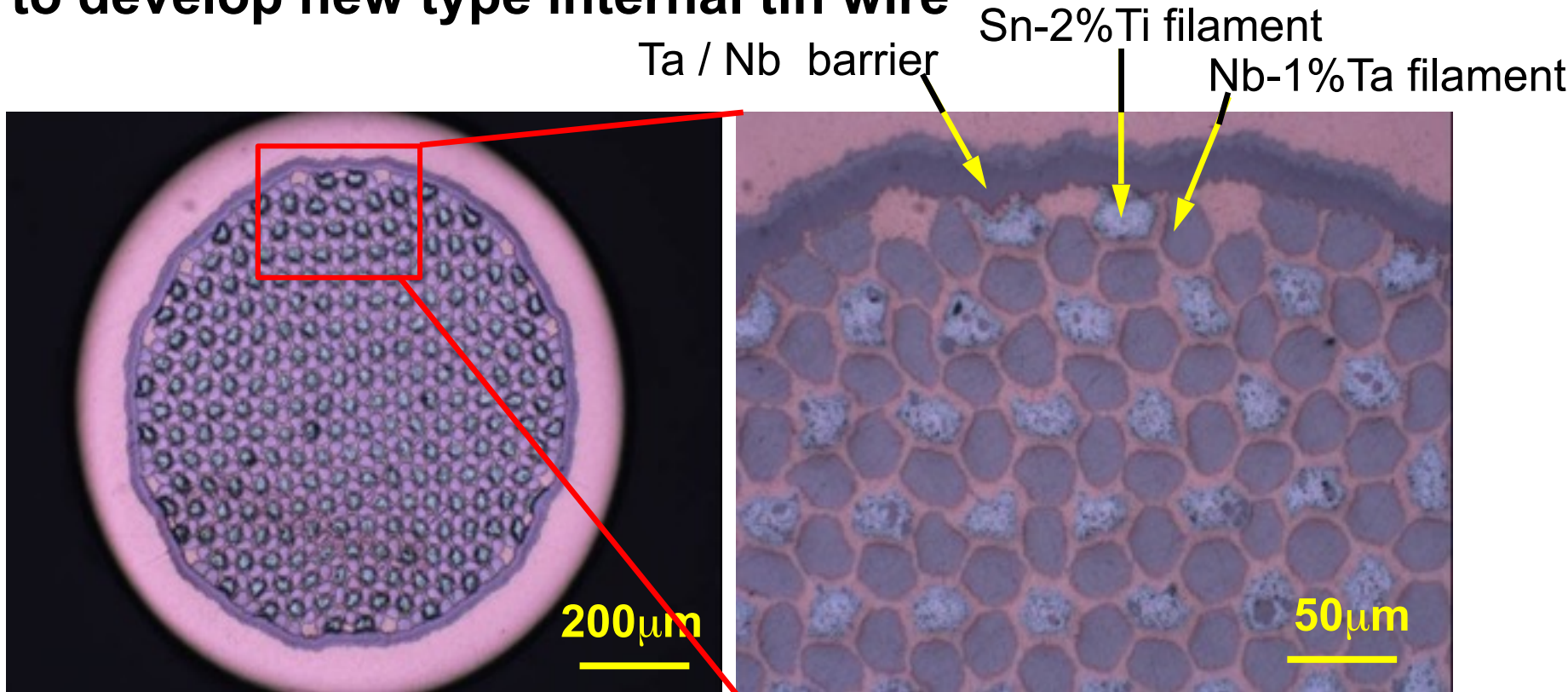


# Team Organization



Development of high performance and low cost  
Nb<sub>3</sub>Sn wires at SH Copper Products

For high field magnet application, since 2009, we started to develop new type internal tin wire



**Sn Filament size is almost equal to Nb filament size.**

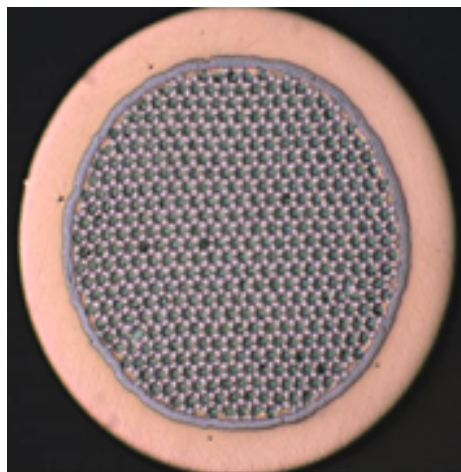
**Simple structure ⇒ Low Cost**

**Corresponding to more than Cu-35wt%Sn bronze**

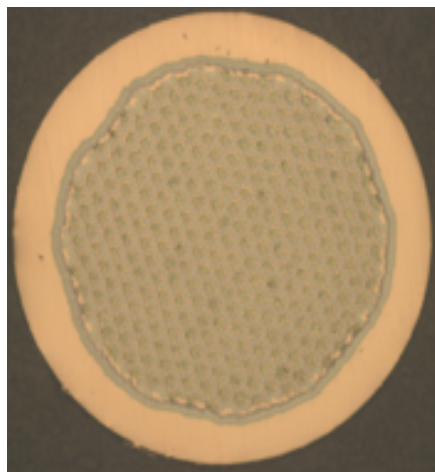


Four manufacturing size billets are successfully extruded and drawn.

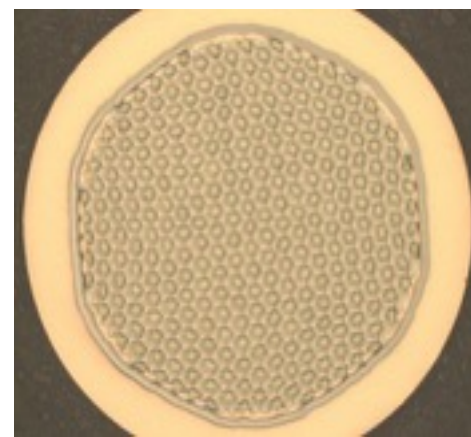
Billet ID No.	<b>HE5000</b> (2010)	<b>HE5143</b> (2011)	<b>HE5542</b> (2012)	<b>HE6585</b> (2014)
Number of Nb filament	840	564	564	277
Number of Sn filament	421	283	295	120
Total number	1261	847	859	397
Filament dia. @ $\phi$ 1mm	15 $\mu$ m	20 $\mu$ m	20 $\mu$ m	30 $\mu$ m
Cu ratio	0.51	0.51	0.51	0.51
Mole ratio (Nb/Sn)	<b>2.53</b>	<b>2.72</b>	<b>2.40</b>	<b>2.89</b>



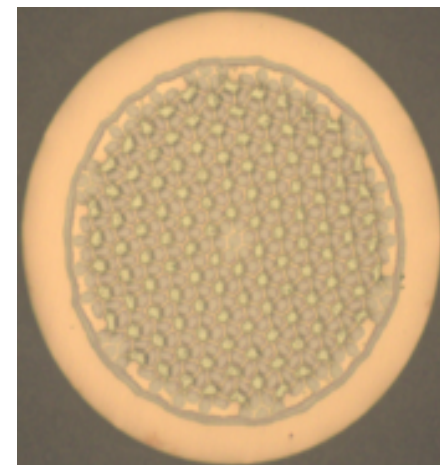
HE5000



HE5143



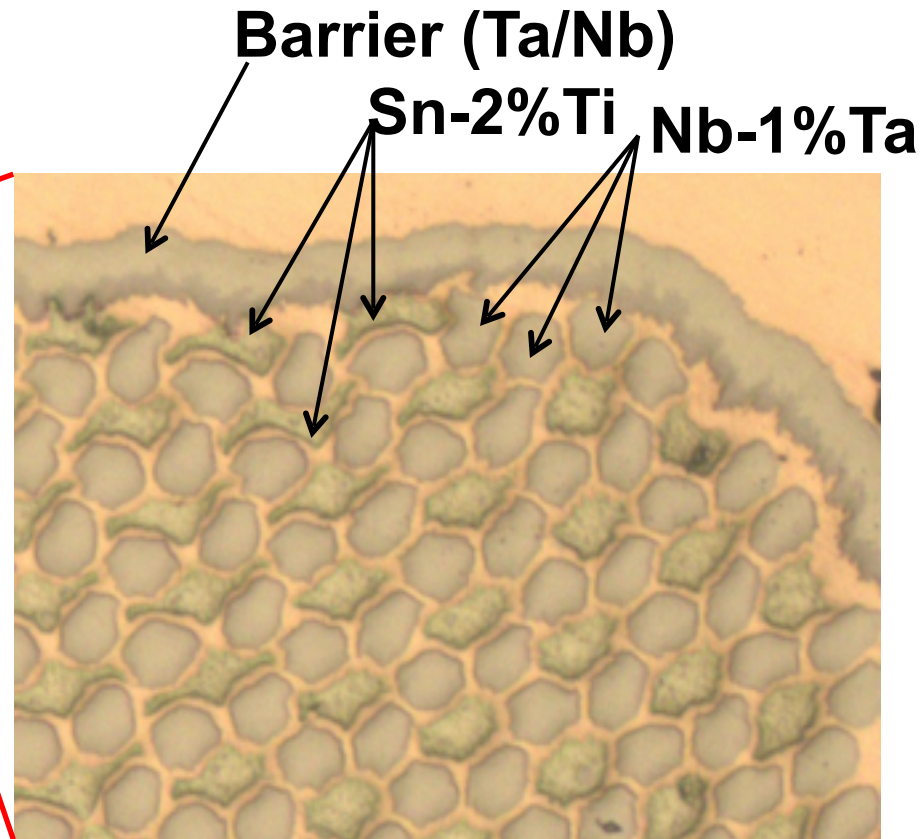
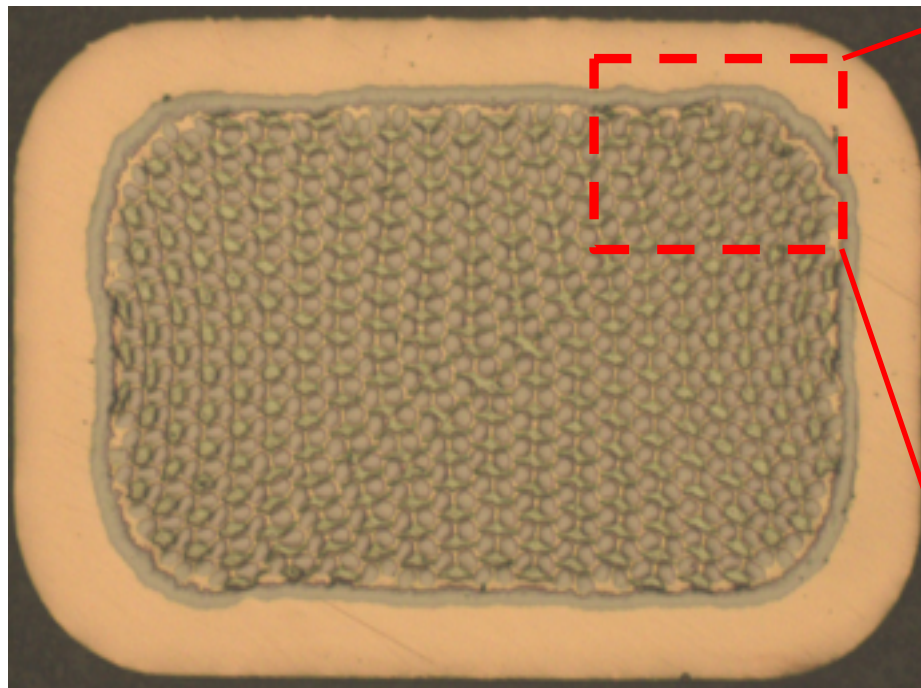
HE5542



HE6585

**Good barrier shape (no deformation)**  
**High RRR ;  $\geq 250$**

It is possible to work rectangular shape without  $I_c$  & RRR degradation.



ID No. HE5143, 1.14X1.72mm  $I_c=504A$  at 18T (Non-Cu  $J_c = 403A/mm^2$ ),  
RRR=254

Item	FCC final target	Status
Non Cu Jc at 16T (A/mm <sup>2</sup> )	>1500	685
$\mu_0\Delta M$ @ 1T, 4.2K (mT)	<150	233
D <sub>eff</sub>	<20	28~30
RRR*1	>150	>250
Unit Length (km)	>5	>20 ( $\phi$ 0.8mm)

**\*1** : After wire deformation, RRR is NOT degraded.

## (1) High $J_c$ ( Non Cu > 1500A/mm<sup>2</sup> @16T)

### (a) Optimization of ratio of Nb, Sn & Cu inside barrier

Mole ratio; Nb : Sn : Cu = 2.5~2.9 : 1 : 2.5~3.5 in the past

= 3.0~3.5 : 1 : 1.8~2.3 in the future

( Nb/Sn > 3.0 & Cu-45~58wt%Sn bronze)

### (b) Nb-**X** filament ( **X** ; 1~4wt% **Ti** or **Ta**)

### (c) New cross section (For high $J_c$ & low magnetization)

### (d) Geometrical filament diameter ; < 20 $\mu$ m

### (e) Optimization of heat treatment

## (2) Low magnetization

### (a) Prevent of completely proximity effect between each Nb<sub>3</sub>Sn filament.

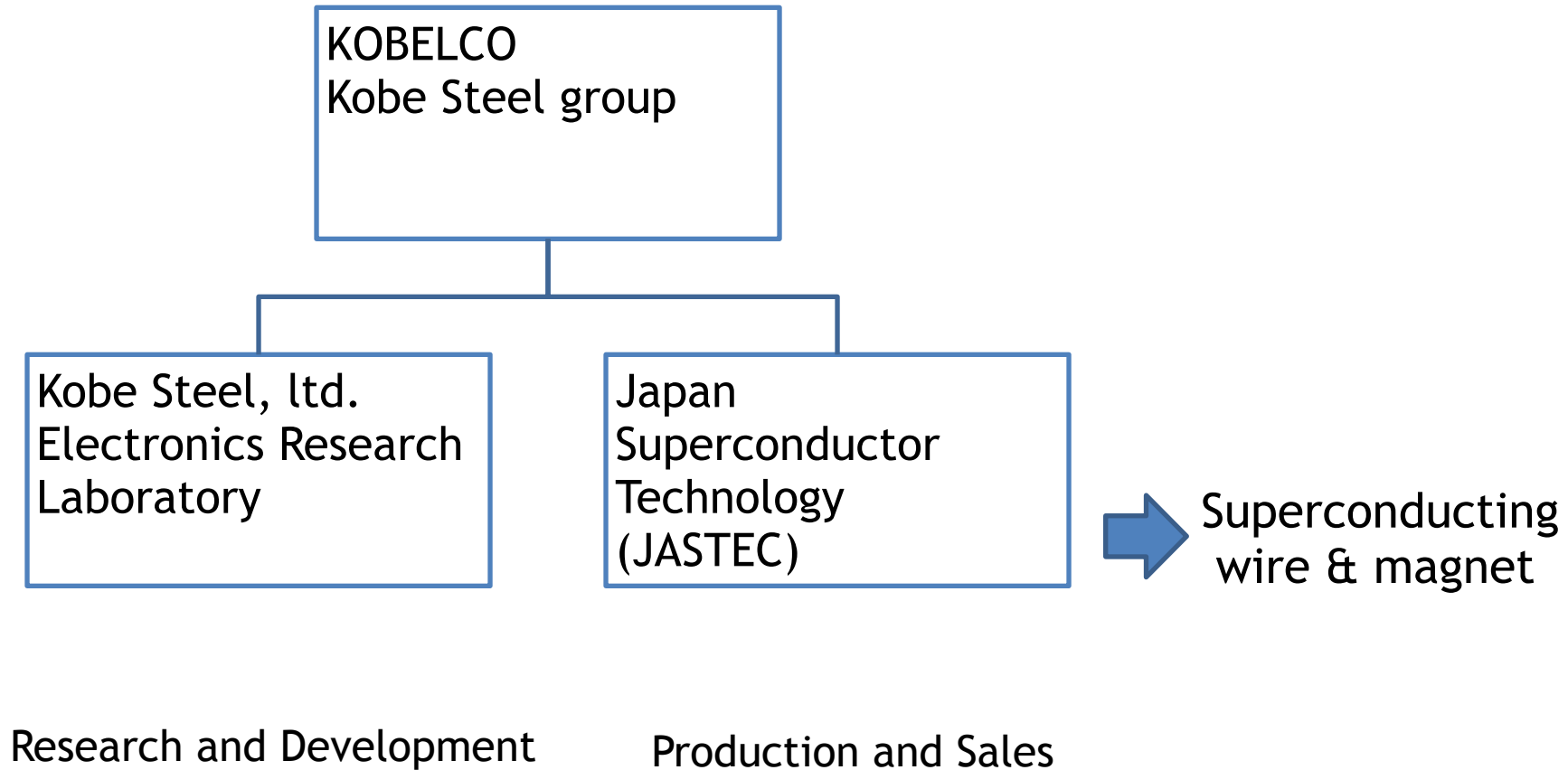
## (3) Stability

### (a) Prevent of completely proximity effect $J_c$

# Development Status and plan at Kobe Steel/JASTEC

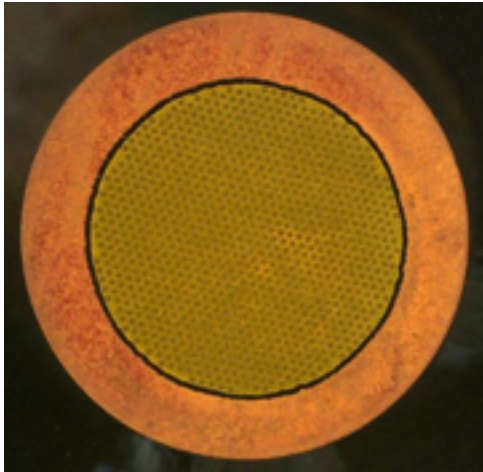
FCC Week 2016 High Jc Nb<sub>3</sub>Sn conductor development in Japan  
2016/4/11

# Kobe Steel and JASTEC



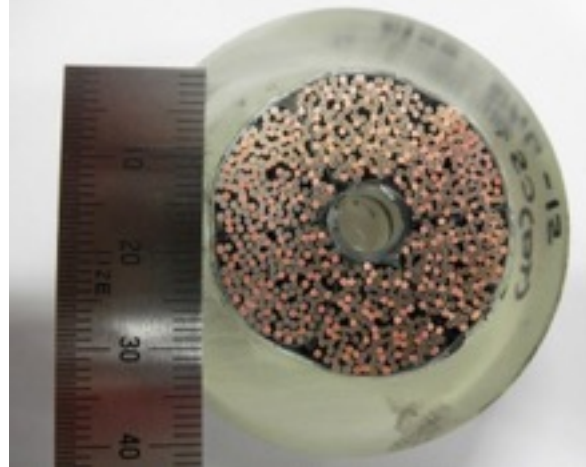


- JASTEC is a world primary wire manufacturers, especially for Nb<sub>3</sub>Sn.
  - For NMR magnet : ~10 tons every year
  - For ITER: 100 tons in total
- JASTEC supplied TF & CS (Nb<sub>3</sub>Sn) wires for ITER.
  - 40 tons for TF conductor (~1/10 of total)
  - 60 tons for CS conductor (1/3 of total)
- R&D division in KSL works for JASTEC.

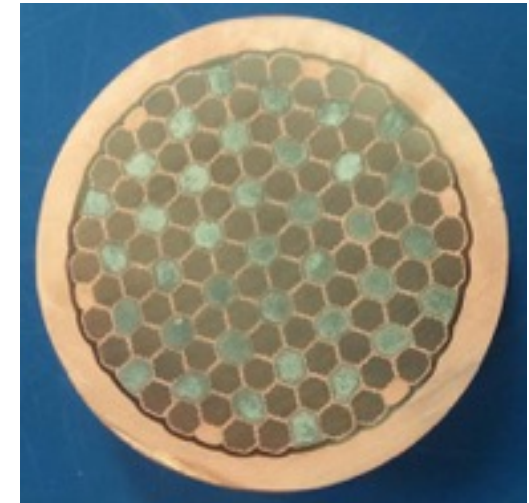


**Strand (TF)**

High-Jc Bronze wire/cable for ITER project



**Cable (CS)**



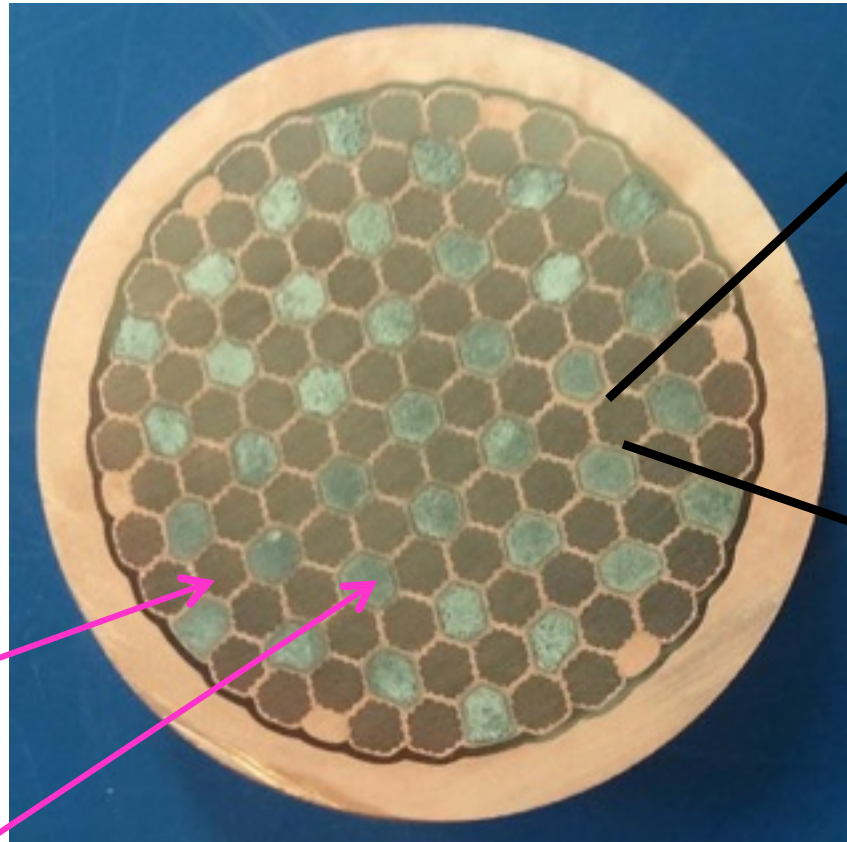
**Distributed Tin (DT) wire**  
with even higher Jc performance  
(under development)

# Distributed Tin Nb<sub>3</sub>Sn Wire (DT wire)

Development target :

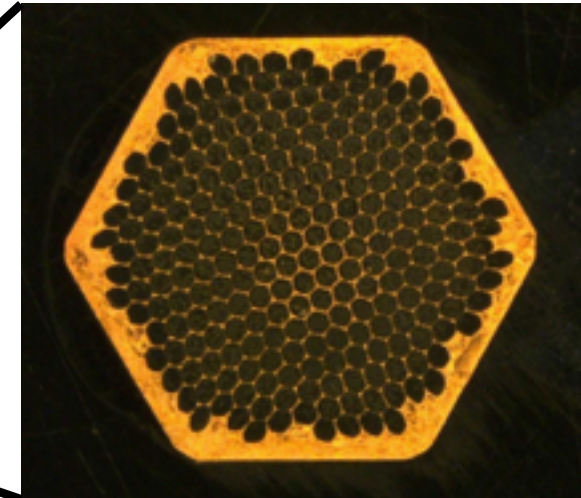
High I<sub>c</sub> wire at high magnetic field (>18T) for NMR magnet

FCC ⇒ High J<sub>c</sub> at 16T



Cu/Nb  
Multi filament

Sn in Cu tube



192 filaments in a  
module

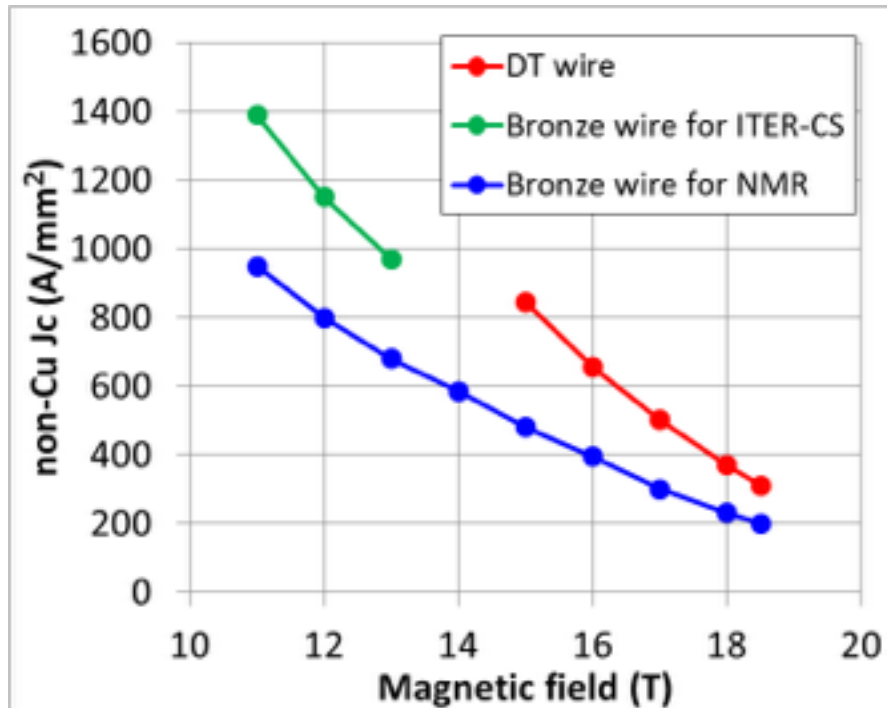
Wire diameter : φ1mm

Filament size : ~3μm

Copper-Non Copper ratio<sup>16</sup> 0.3

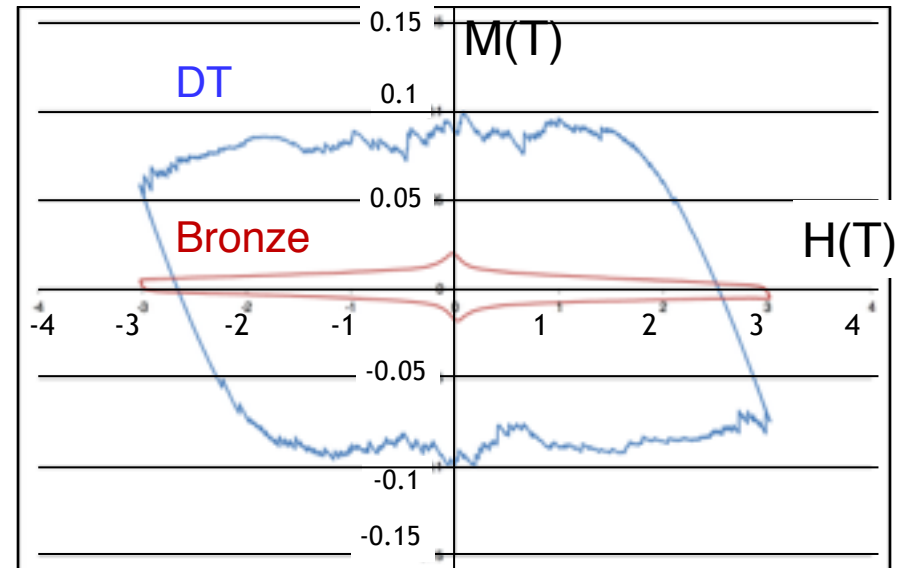


## Critical Current

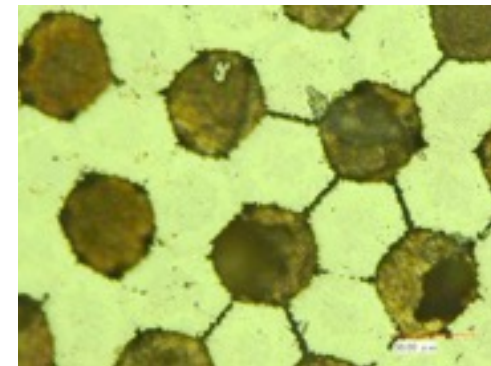


Non-Copper Jc : 650A/mm<sup>2</sup> at 16T

## Magnetization



DT-wire has larger value comparing to Bronze wire  $\Rightarrow$  some filaments are bridged.



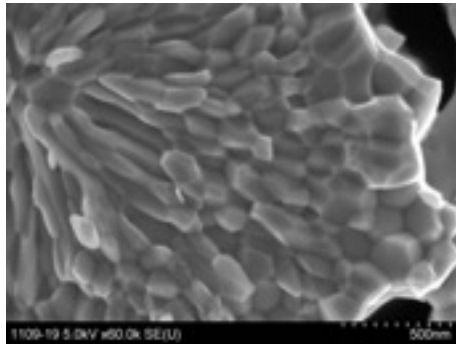
# Future develop plan

## Step 1 for Jc enhancement

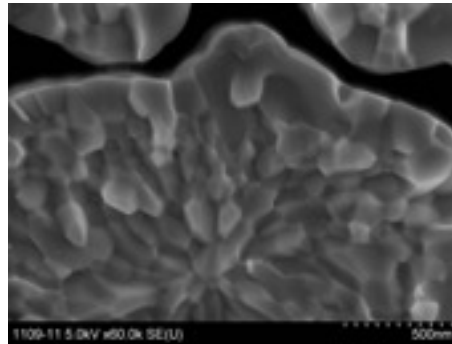
Intrinsic Jc (Nb<sub>3</sub>Sn layer Jc ) depends on the Nb<sub>3</sub>Sn grain size, chemical composition and the Jc enhancing impurity (Ti, Ta). Controlling those factors could be applied for the development.

Also, artificial pinning technique are being considered

Non-Copper Jc :  
540A/mm<sup>2</sup> at 16T

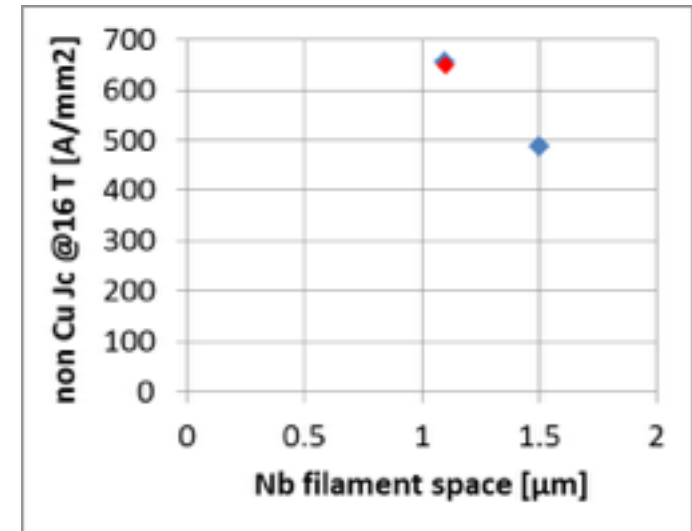


Non-Copper Jc :  
650A/mm<sup>2</sup> at 16T



Refinement of grain size via adjustment of heat treatment condition

Step 2 for decrease magnetization  
magnetization relate to the cross section design.



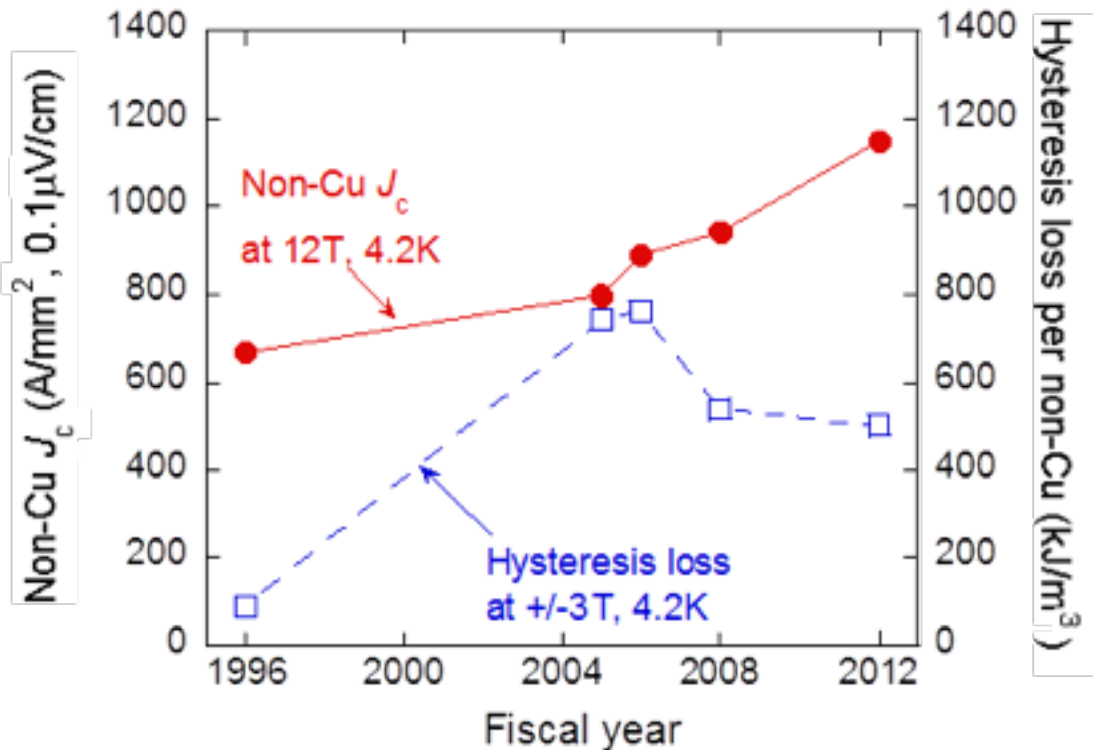
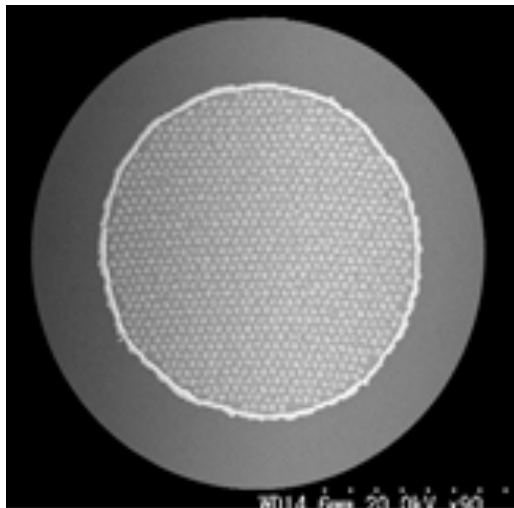
Trade-off relation between Jc and filament spacing

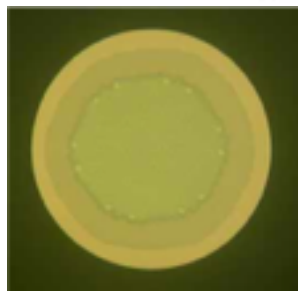
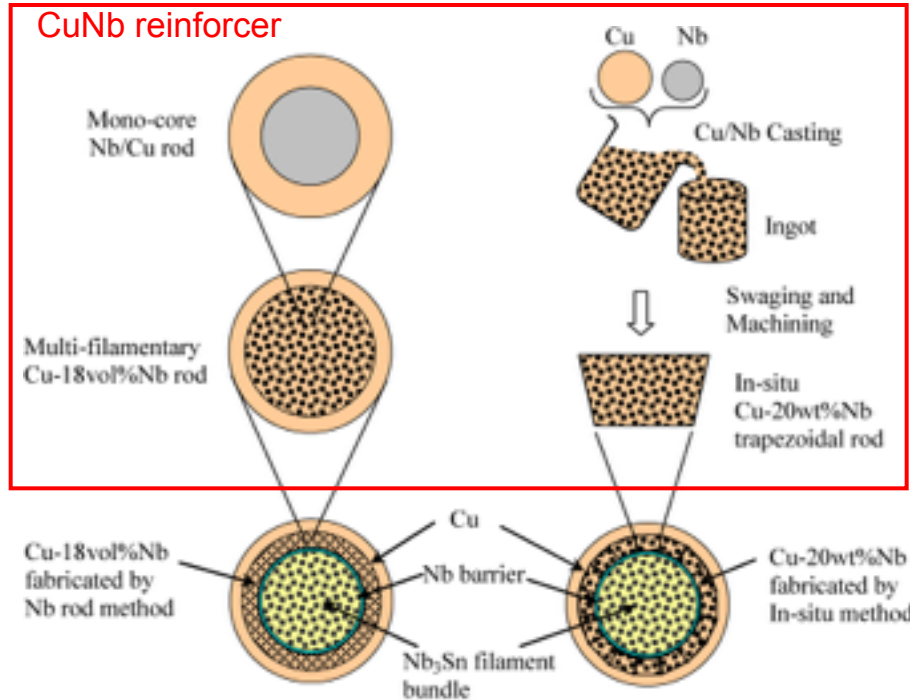
# Development at Furukawa

**March 31, 2016  
Furukawa Electric Co., Ltd.**

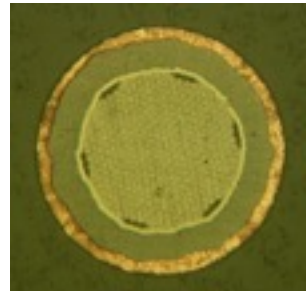
# Increasing $J_c$ of Bronze Method $Nb_3Sn$

Items	2008 design	2012 design
Strand final diameter (mm)	0.83±0.005	0.83±0.005
Cu/Non-Cu ratio	1.0 ±0.1	1.0 ±0.1
Diffusion Barrier	Ta	Ta
Bronze composition (wt%)	16Sn-0.3Ti	15.7Sn-0.3Ti
Filament dia. (μm)	3.3 (nominal)	2.3 (nominal)



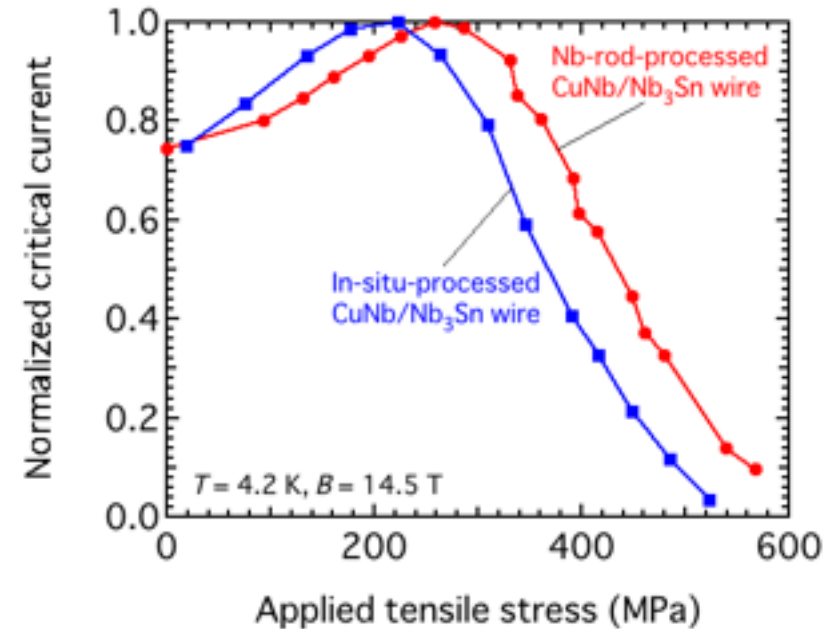


(a)Nb-rod method



(b)In-situ method

## CuNb reinforced $Nb_3Sn$ wires

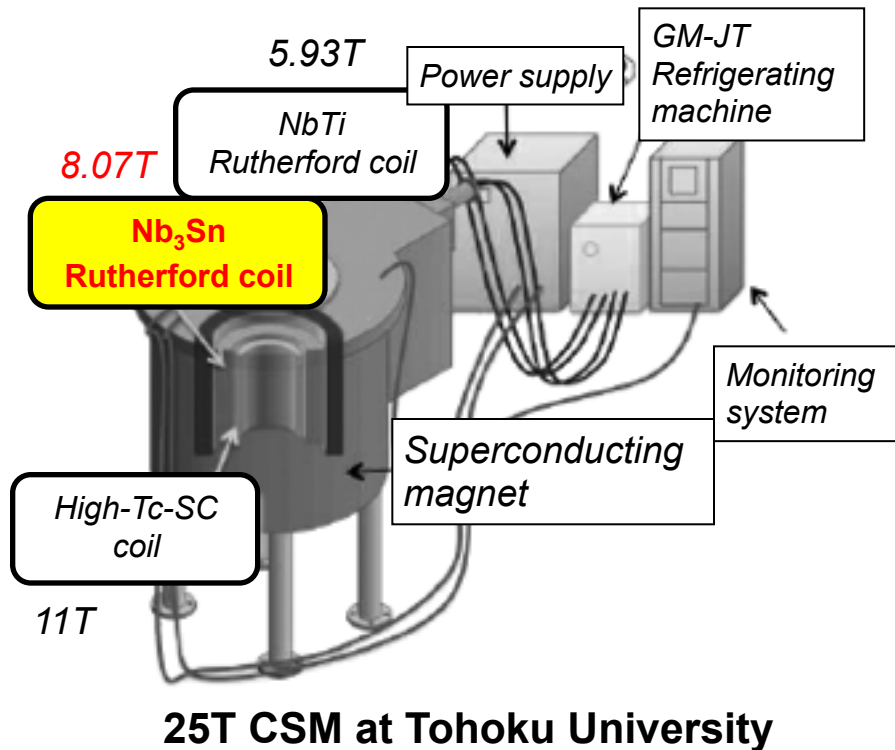


### Merit of Nb-rod method

- Better  $I_c$  properties vs tensile stress
- Larger RRR of wire (>100)
- **Excel in productivity**

[5] H. Oguro, S. Awaji, K. Watanabe, M. Sugimoto, and H. Tsubouchi, *Supercond. Sci. Technol.*, vol. 26, no.9, p.094002, Sep. 2013.

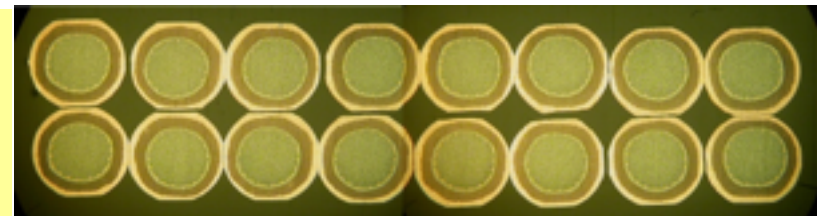
[6] K. Watanabe, S. Awaji, Y. Hou, H. Oguro, T. Kiyoshi, H. Kumakura, S. Hanai, H. Tsubouchi, M. Sugimoto, and I. Inoue, *IEEE Trans. Appl. Supercond.*, vol. 23, no. 3, p. 4300304, Jun. 2013.



(a) Strand	
Superconductor	Bronze- processed Nb <sub>3</sub> Sn
Reinforcement	Nb-rod-method Cu-20vol%Nb
Diameter	0.8mm
Cu/CuNb/non-Cu	20%/35%/45%
Filament diameter	3.3μm
Twist pitch (Direction)	24mm (S)
(b) Rutherford cable	
Number of strands	16
Heat treatment	670°C×96hr
Dimensions (After prebending)	6.5mm <sup>w</sup> ×1.55 mm <sup>t</sup>
Cabling pitch (Direction)	65 mm (Z)

## Requirements of Nb<sub>3</sub>Sn Rutherford cables for the 25T-CSM

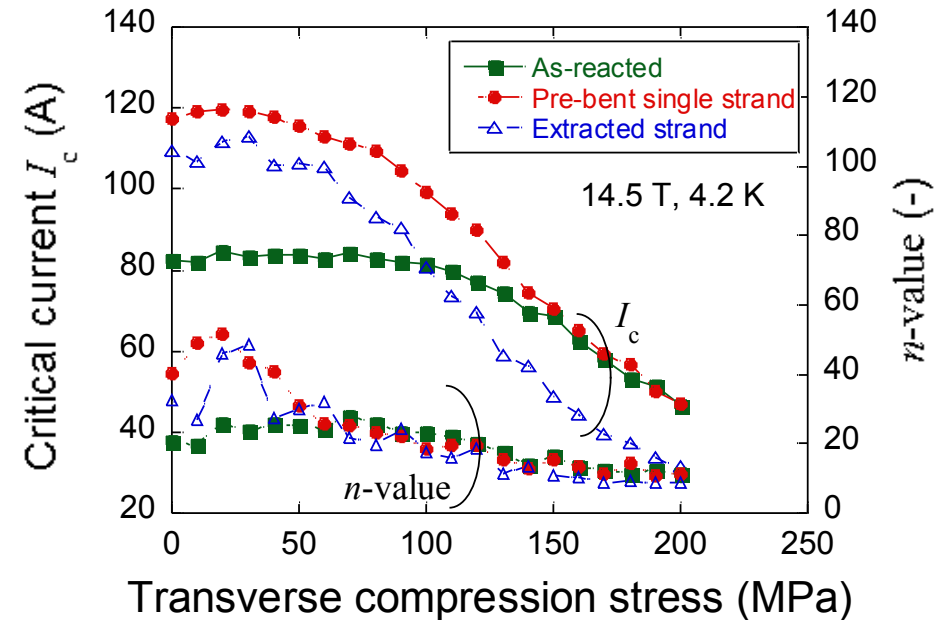
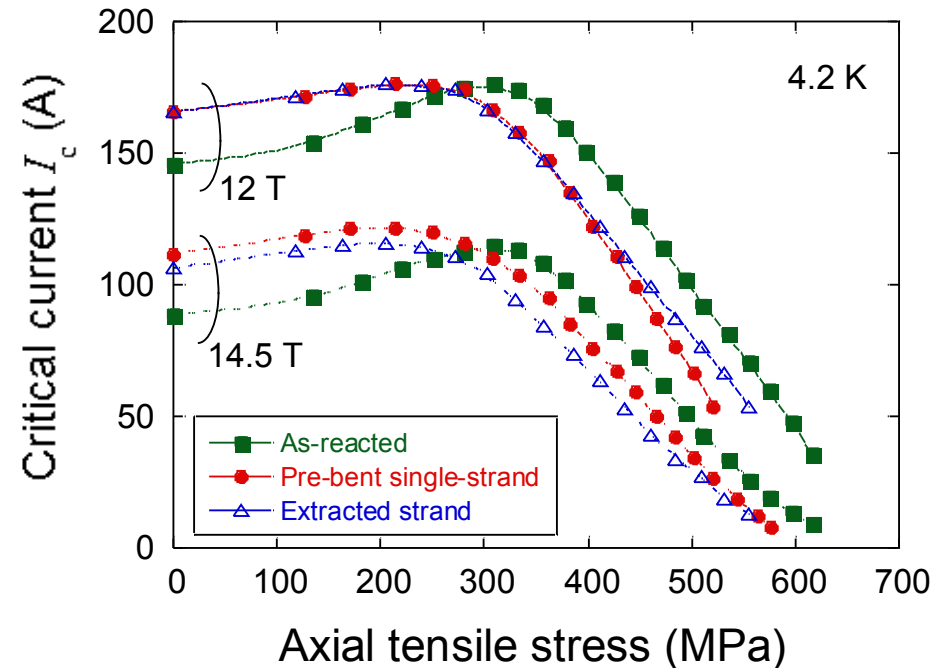
- React-and-wind process
- Applying Cu-Nb internal-reinforced Nb<sub>3</sub>Sn strand with Ta barrier
- Conductor critical current > 1,900 A @ 4.2 K, 12 T, 300 MPa
- Irreversible tensile stress > 350 MPa @ 4.2 K, 14.5 T
- *I<sub>c</sub>* degradation rate at transverse compressive stress 60 MPa <5%
- *I<sub>c</sub>* improvement by pre-bending treatments
- Excellent mass productivity for total length 7.8 km manufacturing



**Cu-Nb/Nb<sub>3</sub>Sn Rutherford cable**

[7] K. Watanabe et al.: IEEE Trans. Appl. Supercond., Vol. 23 (2013) 4300304, [8] S.Awaji et al.: Abstracts of CSJ Conference, Vol.87 (2013) p.154, [9] S.Awaji et al.: IEEE Trans. Appl. Supercond. Vol.24 (2014) 4302005, [10] M.Sugimoto et al., IEEE Trans. Appl. Super. – ASC 2014 Special Issue, accepted for publication.

[10] M.Sugimoto et al., IEEE Trans. Appl. Super. – ASC 2014 Special Issue, accepted for publication.



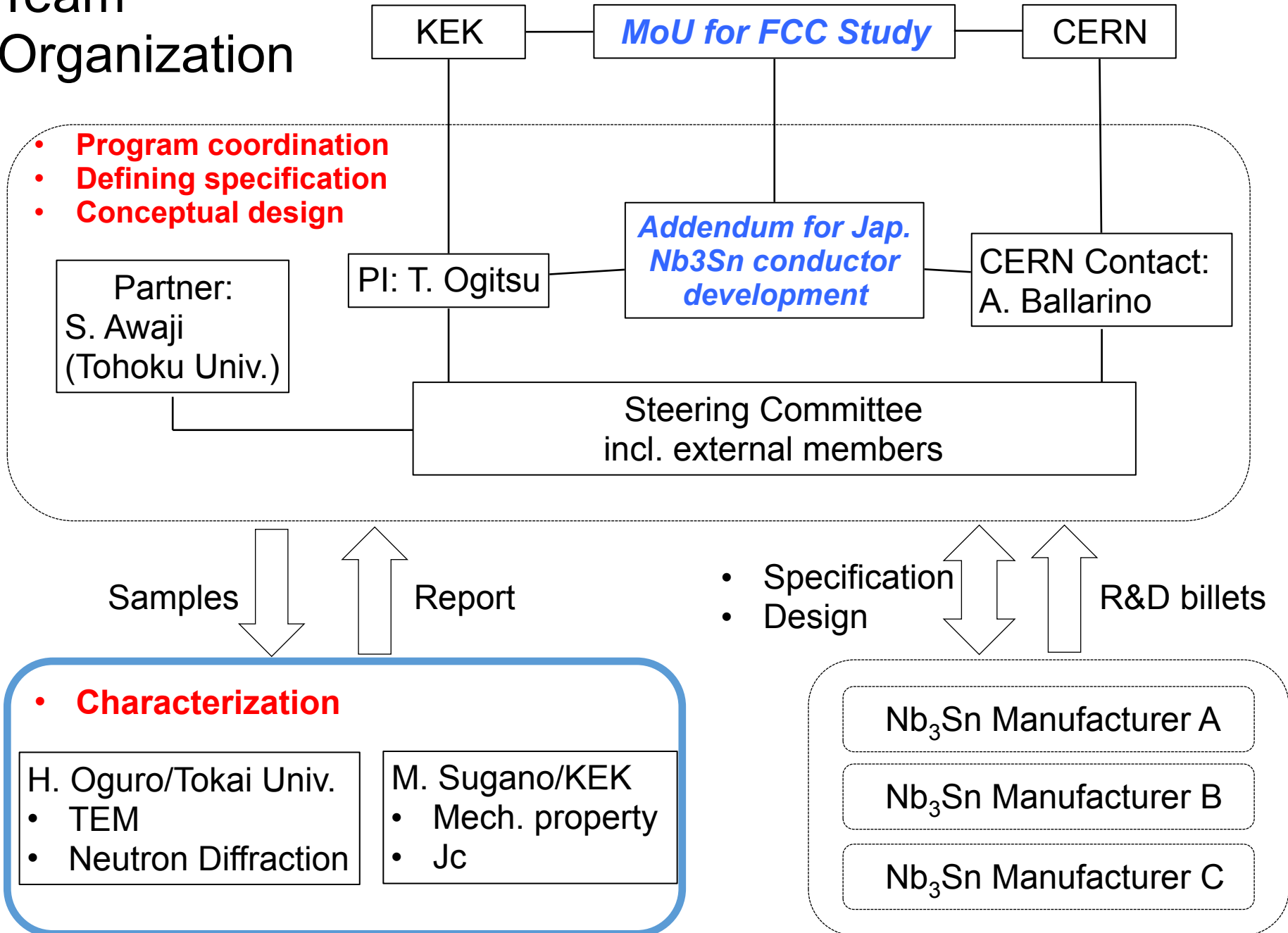
- $I_c$  enhancement range by pre-bending:  
tensile  $\leq 250$ MPa, transverse  $\leq 100$ MPa
- Optimum pre-bending to be suitable for stress condition in operating magnet
- Pre-bending is useful for superconductor used under stressed winding.



- ✓ Furukawa has launched research study to come up with breakthrough idea for FCC target;
- $J_c \geq 1,500 \text{ A/mm}^2$  at 4.2K, 16T
- Wire diameter  $\leq 1 \text{ mm}$
- Cu to non-Cu  $\geq 1$
- $\text{Nb}_3\text{Sn}$  filament diameter  $< 50 \mu\text{m}$
- RRR  $> 150$
  
- ✓ by modified Nb tube and/or modified internal tin (DT-like)
  
- ✓ under collaboration with CERN and KEK
  
- ✓ by Feb. 2020



# Team Organization



# Test items

## Electro-magnetic performance

- $I_c$  measurement at 4.2 K and  $B < 18$  T
- RRR measurement (after rolling and HT)
- B-H measurement (VSM) → Magnetic stability

## Microscopic observation

- Electron Backscatter Diffraction Analysis (EBSD) → Grain size
- Energy Dispersive Electron Spectroscopy (EDS) → Composition analysis
- SEM, TEM, ...

## Electro-mechanical performance

- $I_c$  vs axial-tensile strain, compressive stress

## Neutron diffraction at J-PARC

- Strain measurement at cryogenic temperature
- Observation of phase evolution during heat treatment

$I_c$  measurements: mainly by KEK at Tohoku University

More detailed analysis: by Tokai University (Dr. Oguro's group)

# Ic measurement

## Required specifications

- Magnetic field:  $> 16$  T
- Temperature: 4.2 K
- Current capacity

non-Cu  $J_c = 1500$  A/mm<sup>2</sup>

Wire diameter = 0.8 mm

Cu/non-Cu ratio = 1



$I_c \sim 380$  A



18 T solenoid magnet  
in Tohoku University

## Assignment of measurements

A series of  $I_c$  measurements will be performed in Japan, and conductors with high  $J_c$  will be sent to CERN.

$I_c$  measurements at  $B < 12$  T: Possible in all three conductor suppliers

$I_c$  measurements at  $B > 12$  T: by KEK with Tohoku and Tokai University + each supplier

## Standardization of measurement procedure

- Spiral sample for precise  $I_c$  determination
- Mandrel: ITER barrel
- Handling of samples



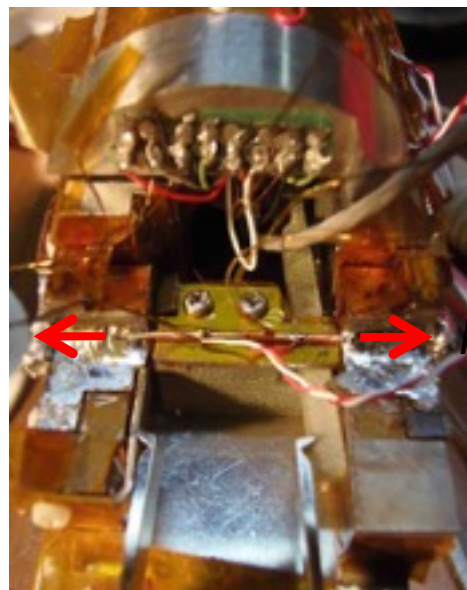
ITER barrel

Ghosh, IEEE TAS (2011)

# Electro-mechanical tests

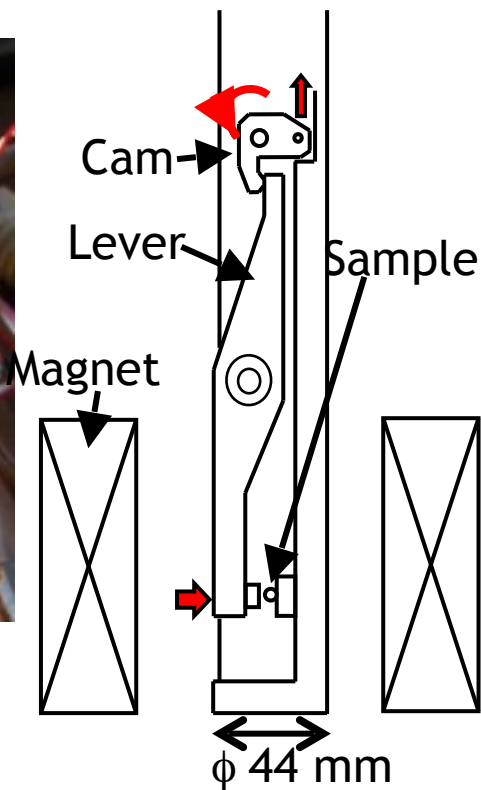
Various inserts for electro-mechanical tests for wires have been developed in Tohoku University.

## Axial tensile strain

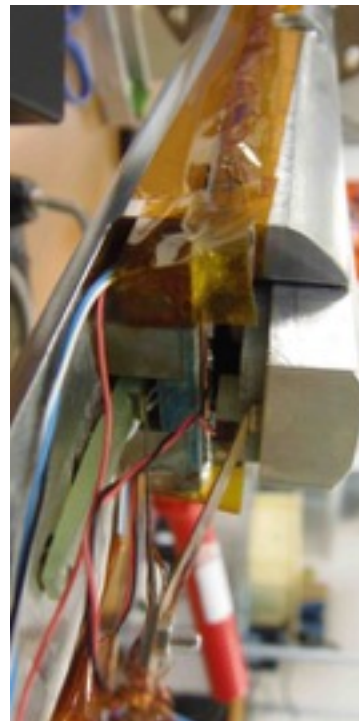


Field < 18 T  
Temperature : 4.2 K  
Load < 5 kN

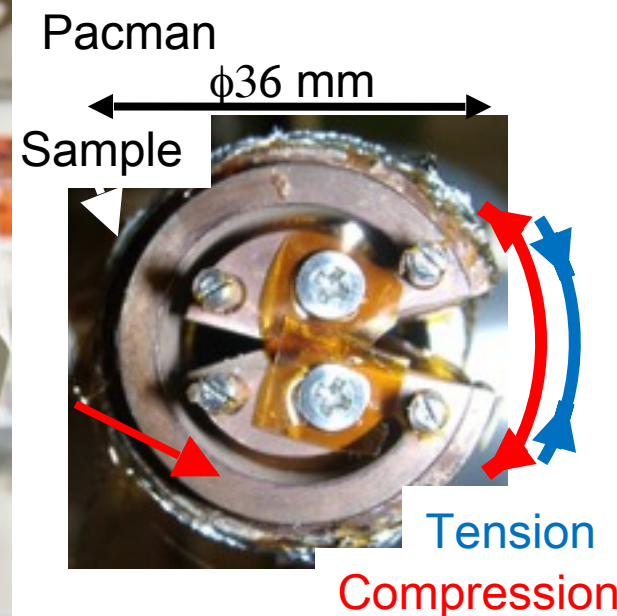
## Compressive stress



Field < 18 T  
Temperature : 4.2 K  
Load < 1 kN



## Axial tensile/compressive strain

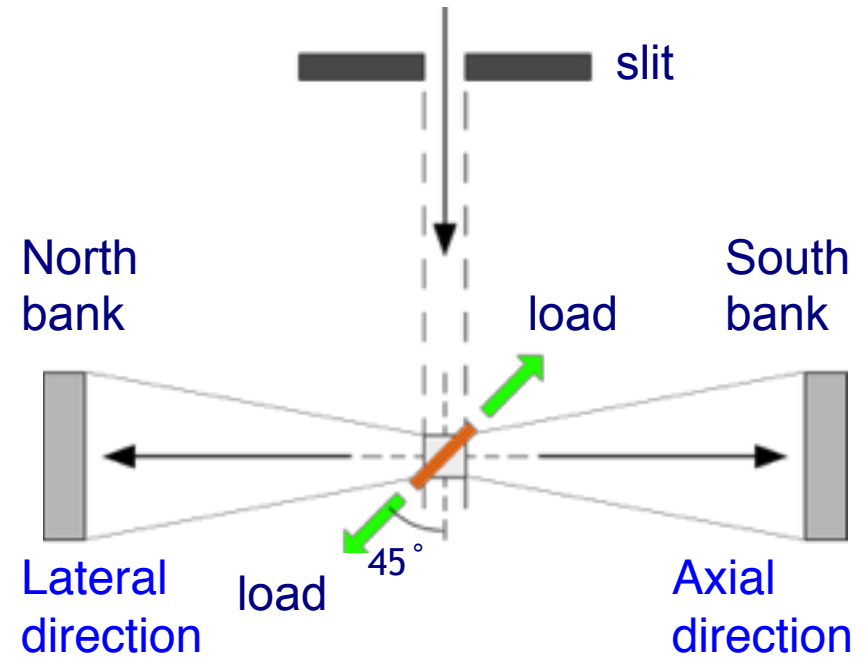
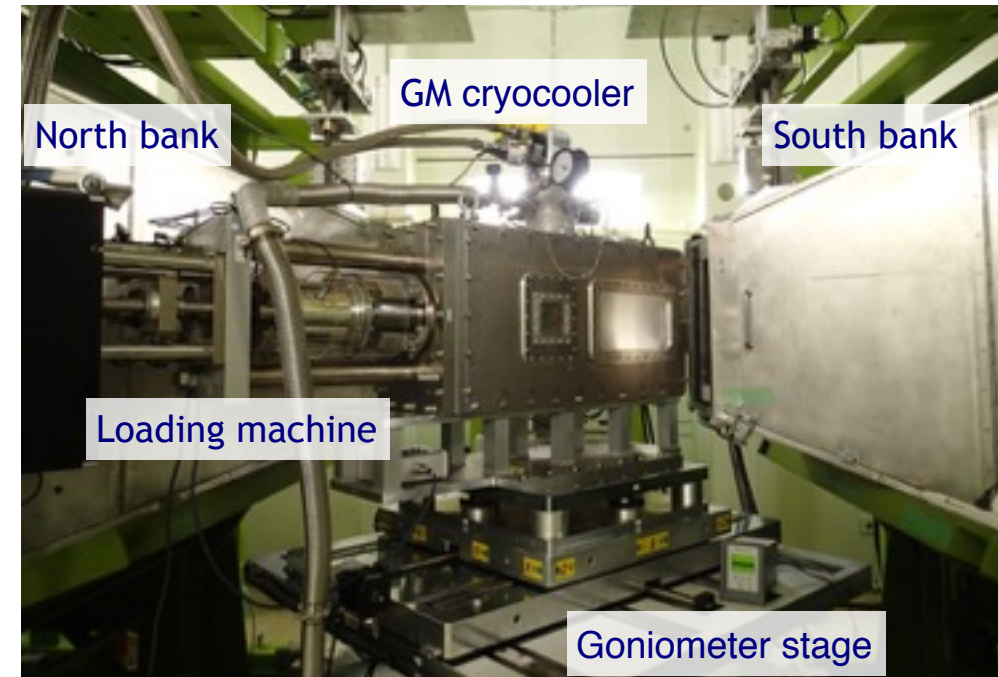


Field < 18 T  
Temperature : 4.2 K  
Strain -0.5 – 0.5%

# Neutron diffraction technique at J-PARC

Engineering Materials Diffractometer (TAKUMI), J-PARC

Cryogenic tensile testing system



Temperature : 10 - 300 K  
Load: < 50 kN

Residual strain of Nb<sub>3</sub>Sn in composite conductors at cold can be evaluated.

Jc-strain performance for conductors with different cross-sections can be discussed.

Neutron diffraction technique will be also useful for observing phase evolution during heat treatment of Nb<sub>3</sub>Sn wires.

# Summary

- Joint R&D program: CERN, KEK and Tohoku university
  - 4 year program
- 3 Manufacturer will corporate
  - SH-Copper: DT (single stack)
  - JASTEC/KobeSteel:DT (Nb sub bundle)
  - Furukawa: Nb Tube (or DT)
- Characterization
  - KEK, Tohoku Univ., and Tokai Univ.