

# Stable Mass-Production by Hot-Wall PLD Approach for Uniform REBCO Wires and Magnet Applications

Y. Iijima, M. Ohsugi, K. Kakimoto, S. Muto, W. Hirata, S. Fujita,  
N. Nakamura, S. Hanyu and M. Daibo

**Fujikura Ltd.**

Acknowledgement:

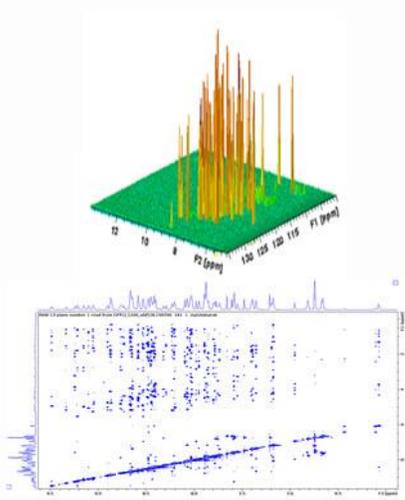
We appreciate Prof. T. Kiss at Kyushu Univ. and Prof. S. Awaji at Tohoku Univ. for collaboration to sample evaluation. A part of this work was also performed at the High Field Laboratory for Superconducting Materials, IMR, Tohoku University.

# Contents

- **REBCO wire mass production started:**
  - Market started from **high-field application** : NMR, Fusion
  - **Why IBAD/PLD process?** High in-field  $J_c$  / Strong in-field mechanical strength
  - Trade-off of **laser cost** and **PLD advantage**
  
- **Advantages of PLD process for mass-production**
  - **Reproducibility:** controllable near **best phase point**
  - **High in-field  $I_c$**  : small and dispersed pinning centers by **vapor deposition**
  - **High growth rate:** high transfer yield, high supersaturation and adequate adatom energy
  - **Uniformity:** temperature stability improved by **Hot-wall architecture**
  
- **Uniform REBCO wire lineup by Fujikura for magnet applications**
  - **Demand of magnet technology:** **Uniformity** and **Mechanical reliability**
  - **New area:** NI technology, assembled conductors, quench protection,,,
  
- **Summary**

# Business demand of REBCO urged scale-up investment

## BRUKER 1.0/1.2GHz NMR (2019~)



**First Commercial Practical device using REBCO wire**

**1.2 GHz NMR  
28.2 T magnet  
with 54 mm bore**

**1.0 GHz NMR with compact 23.5 T magnet**

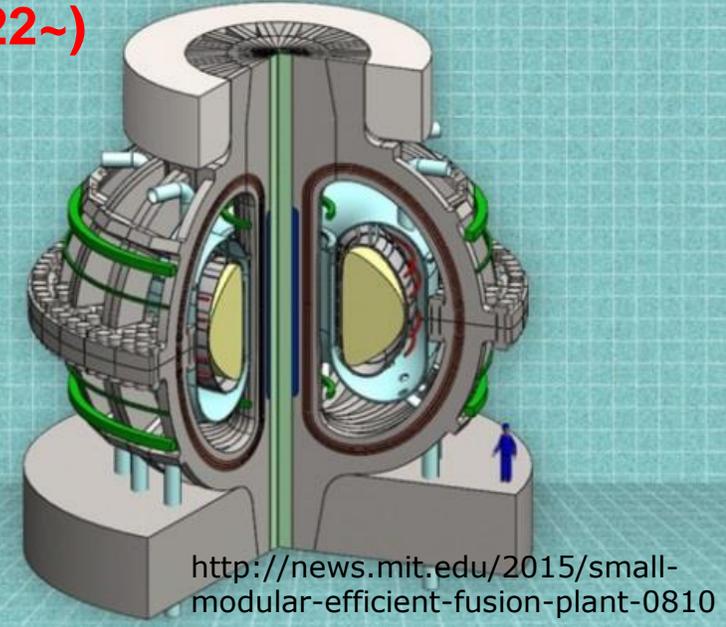
longitudinal Ic uniformity strongly required

## Compact fusion reactor (2022~)

**Compact design marketable by using private funding**

**Toroidal field ~9T(ARC/CFS) (ITER/DEMO ~6T)**

**Neutron radiation damage inevitable come from thinner shield**



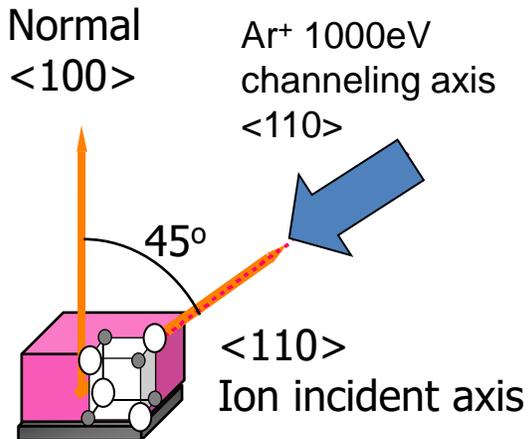
**REBCO wire demand up to 10000s km/reactor**

On going reactor program: SPARC (CFS), ST80 (Tokamak energy(TE))

Planned reactor program: ARC (CFS), ST-E1 (TE), STEP (UKAEA), and many others

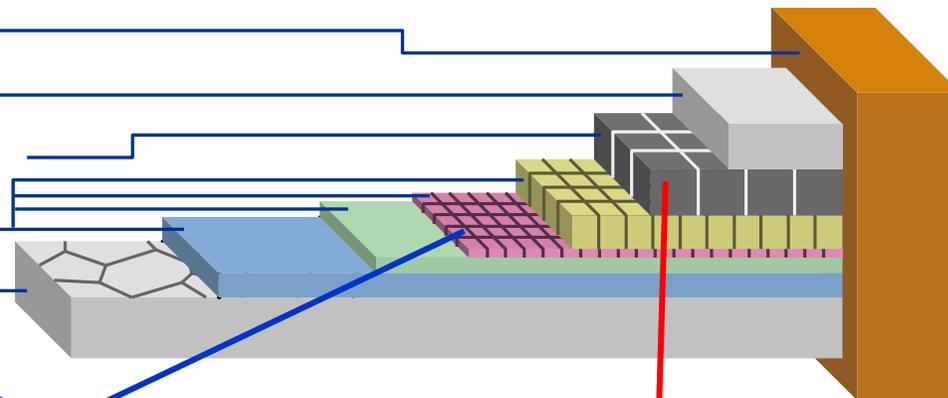
Big demand come from large scale/large current magnets with NI technology / assembled conductors  
High quality wires with desired delivery&cost

# Fujikura's 2G HTS wires processed by IBAD/PLD method

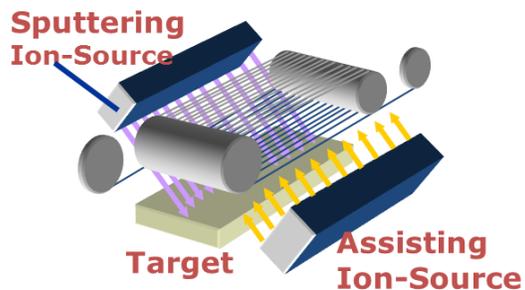


## < Schematic of 2G HTS wire >

- Stabilizer [electroplated copper] 20  $\mu\text{m}$
- Protection layer [Ag] 2  $\mu\text{m}$ ~
- HTS layer [GdBCO 2  $\mu\text{m}$ ] / [EuBCO+BHO 2.5  $\mu\text{m}$ ]
- Buffer layer [MgO, etc.] ~0.7  $\mu\text{m}$
- Substrate [Hastelloy®] 75 / 50  $\mu\text{m}$



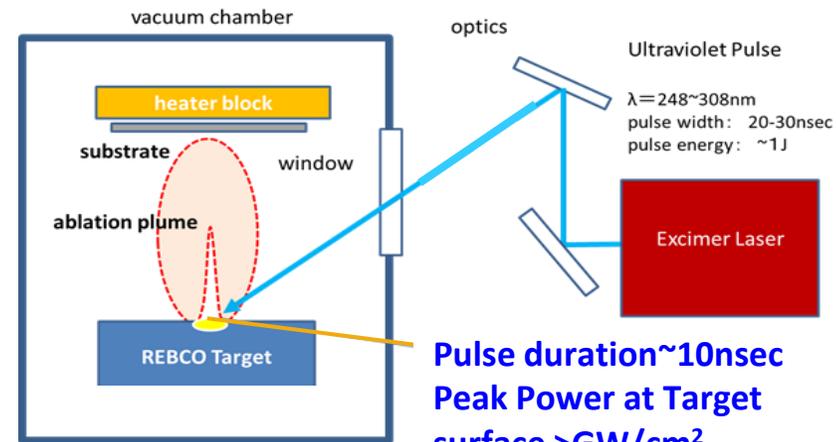
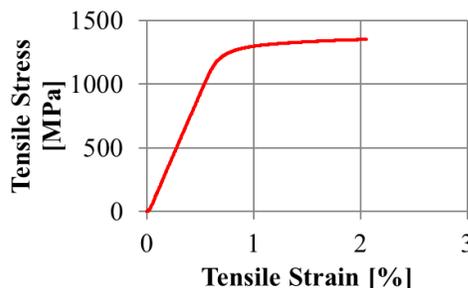
## Ion-Beam-Assisted Deposition (IBAD)



Reel-to-Reel IBAD system

Off-normal directional ion beam process allows direct use of thin non-textured Ni-Cr alloy tapes with strong enough mechanical strength

## Pulsed Laser Deposition (PLD)



# General cost evaluation of REBCO film production process

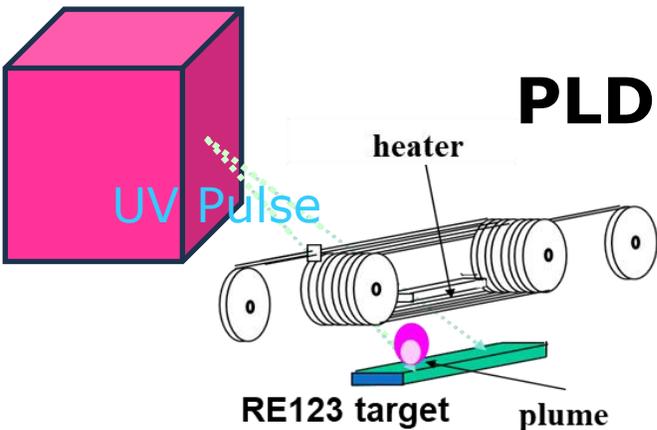
- PLD system has costly Excimer Laser, but **the equipment cost** shrinks by **High Throughput, Operation Lifetime, and High Production Yield.**

$$\text{Cost}_{\text{REBCO}} \sim \left( \text{Cost}_{\text{op.}} + \left( \frac{\text{Cost}_{\text{equipment}}}{\text{Throughput} \times T_{\text{op.}}} \right) \right) \times \frac{1}{\text{Production Yield}}$$

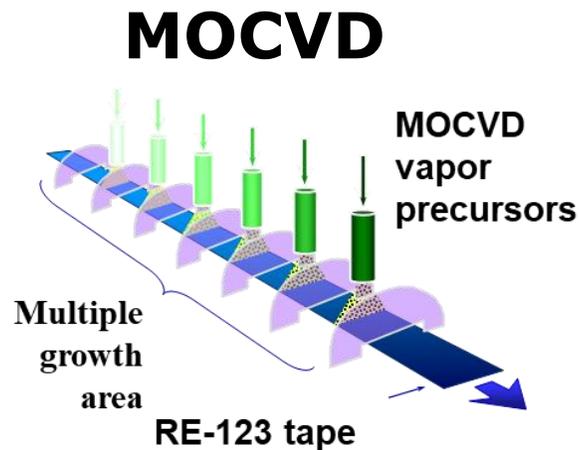
- ◆ Growth rate
- ◆ Laser power
- ◆ Laser lifetime

- ◆ Reproducibility
- ◆ In-field  $J_c/I_c$
- ◆ Uniformity

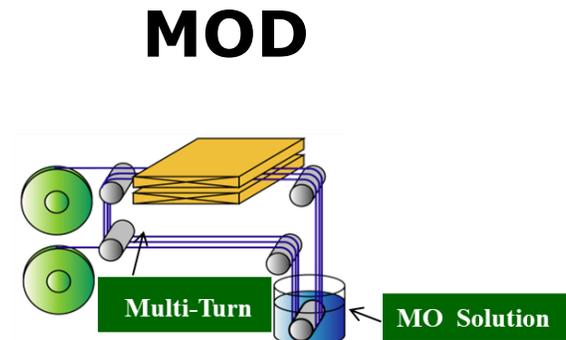
**Industrial Excimer Laser**



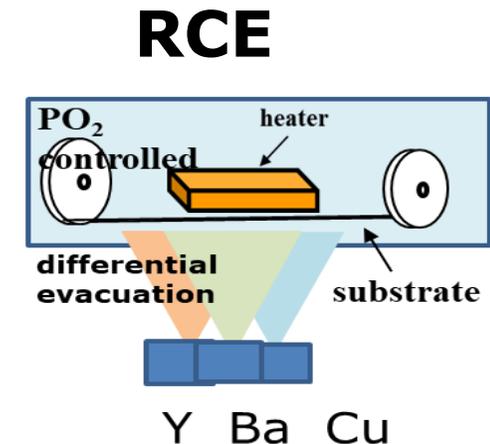
**PLD**



**MOCVD**

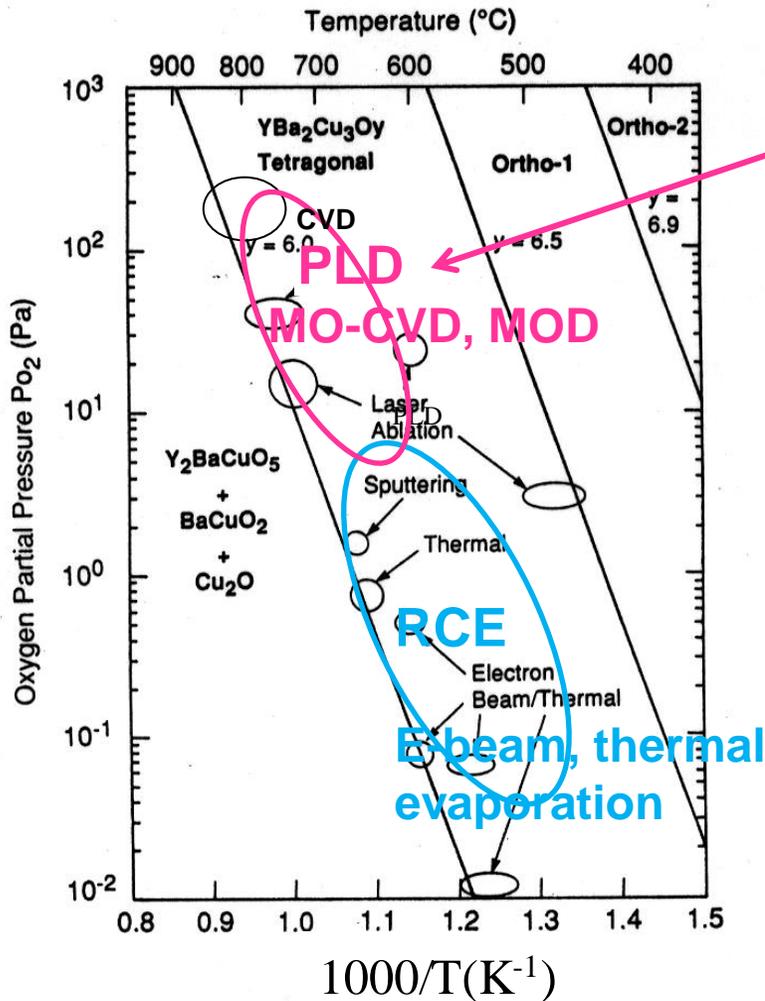


**MOD**



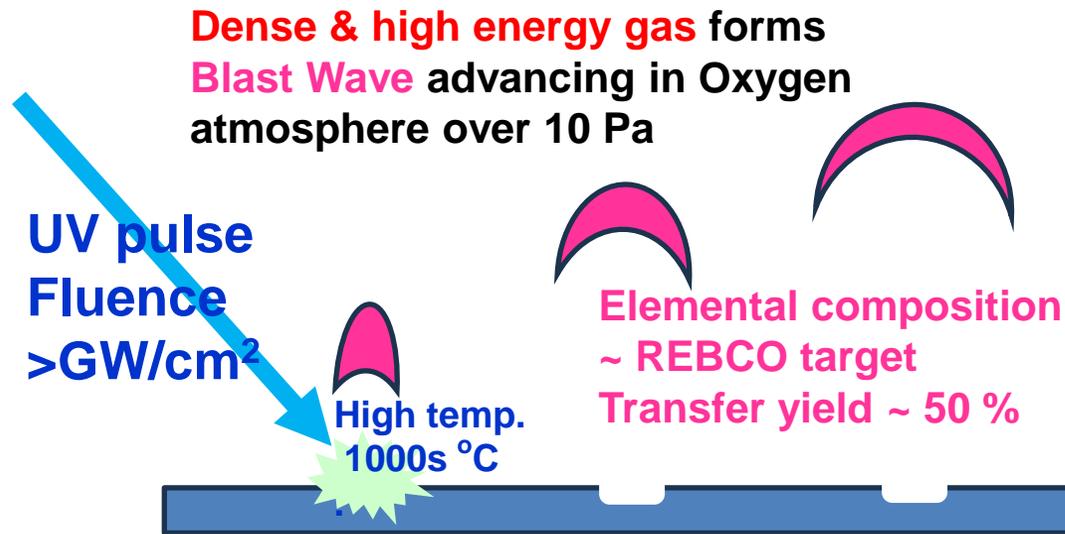
**RCE**

# Why stable? : Controllable process at best phase position

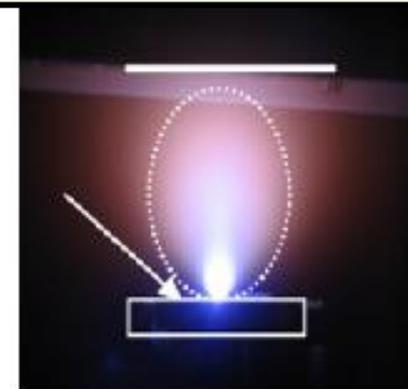


R. H. Hammond and R. Bormann;  
Physica C, 162 (1989)703

- ◆ Rapid, energetic and fine evaporation by UV pulse laser
- ◆ Large window of oxygen pressure
  - ◆ Favorable  $P_{O_2} > 10\text{Pa}$  with growth temp. 700-800 °C
  - ◆ Ablation Plume can reach far longer than mean free path ( $\lambda = 7\text{mm}$  @10Pa) which cannot be expected in conventional evaporation process



## Ablation Plume



R. Delmdahl et al. Appl. Phys. A 93(3):611-615 (2008)

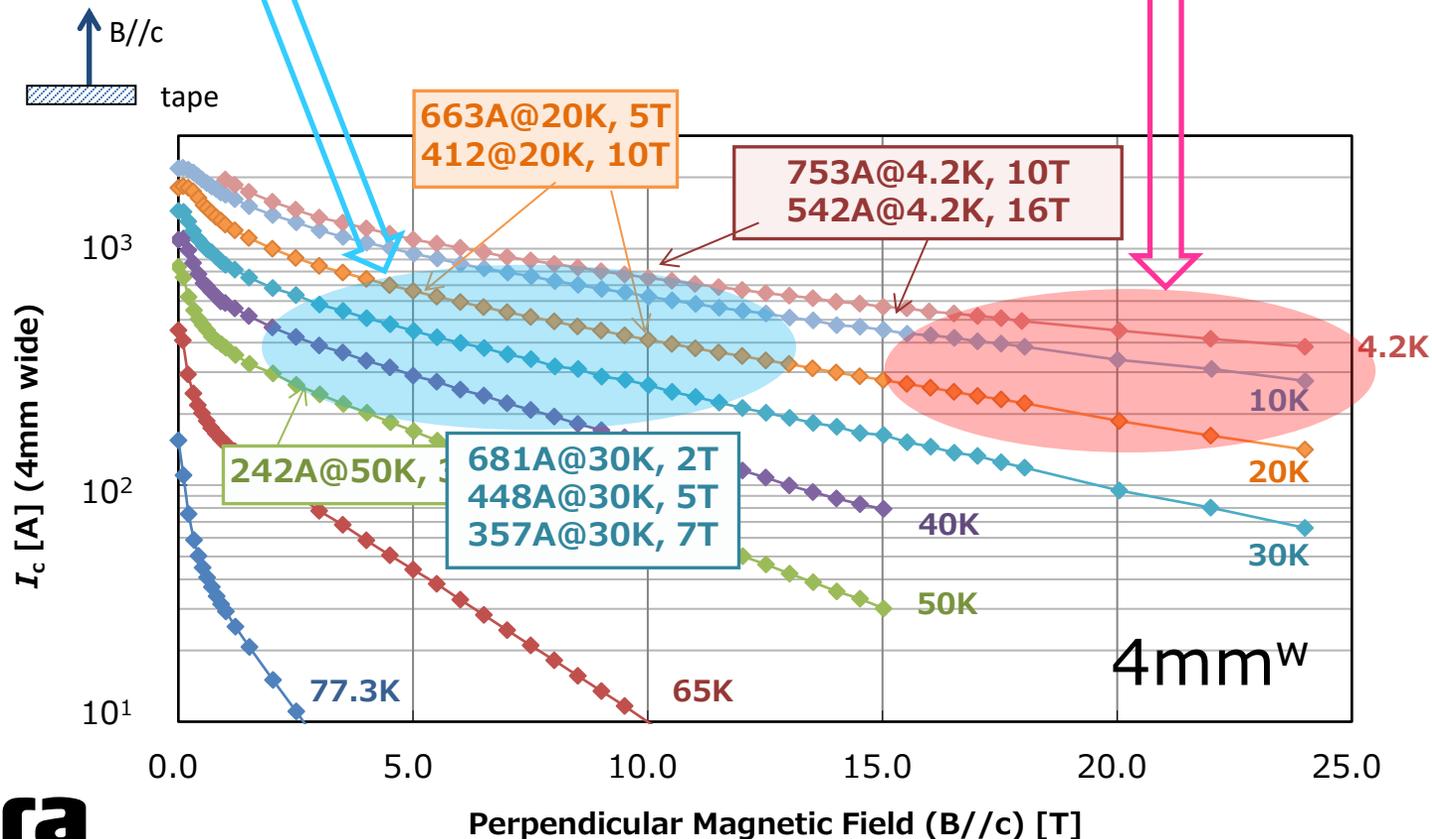
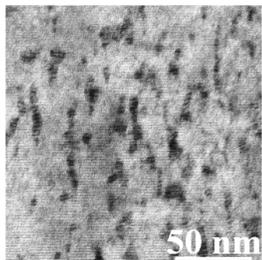
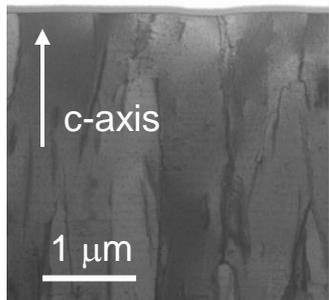
A.V. Bulgakov, N.M. Bulgakova, "Dynamics of laser-induced plume expansion into an ambient gas during film deposition", J. Phys. D: Appl. Phys. 28 (1995) 1710–1718.

- ◆ PLD is the only in-situ vapor deposition process controllable near best phase position with simple physical parameters
- ◆ No need of differential pumping or chemical decomposition

# Why high in-field $J_c$ : vapor phase deposition with supersaturated gas

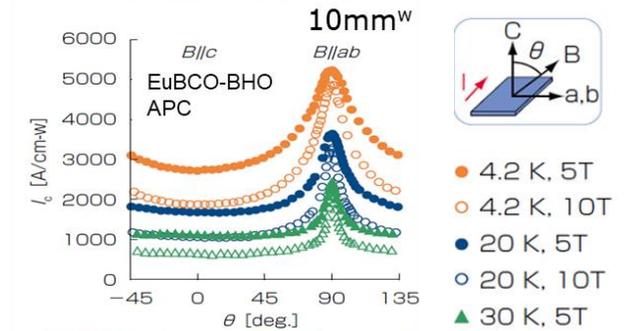
- ◆ PLD: Vapor growth process with
  - ◆ Favorable condition of cation composition & oxygen-temperature diagram
  - ◆ Dense & supersaturated gas with favorable particle energy for adatom mobility

Good c-axis aligned matrix growth with dense and small defects of secondary phase, nanorod, nanoparticles, point defects, oxygen vacancy...

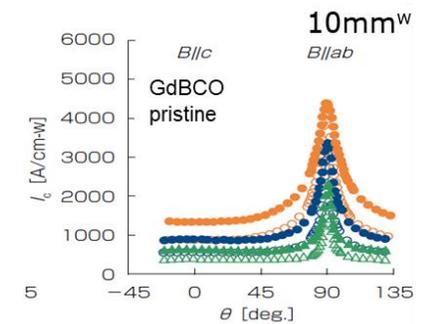


## $I_c$ - $B$ - $\theta$ in lower field $\sim 10$ T

### FESC (APC type)

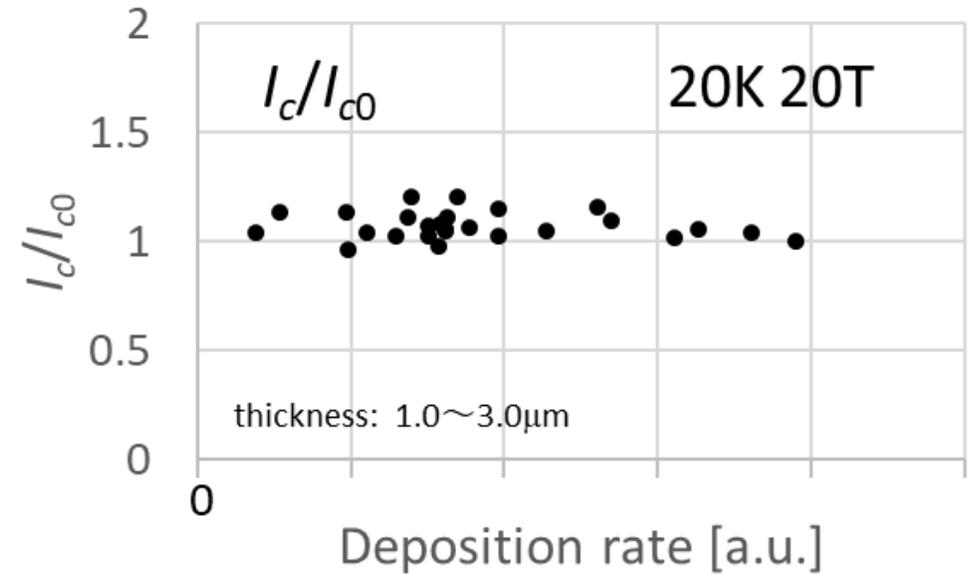
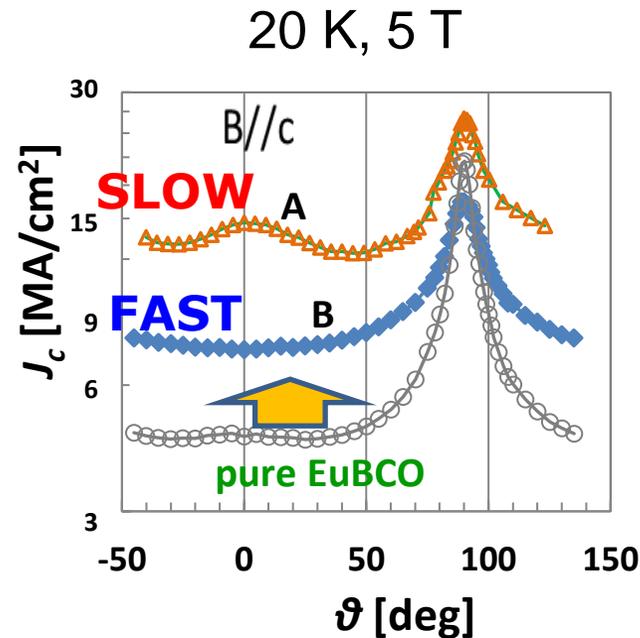
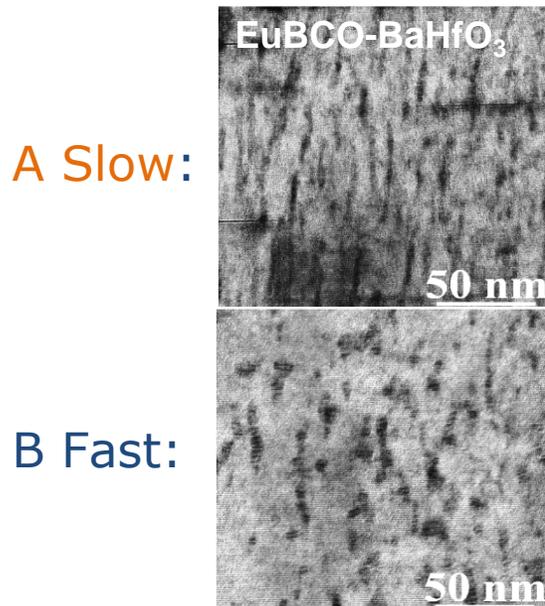


### FYSC (non-APC type)



# Why fast? Growth rate dependence for $I_c$ properties

- ◆ High-rate growth by high-rate evaporation with high transfer yield ~50%
- ◆ High-rate limit not observed for  $I_c$  in high field
  - ◆ High adatom mobility compensate defective growth by dense & supersaturated gas
- ◆  $I_c$  in lower field affected by growth rate
  - ◆ Nanorod shape depends on growth speed

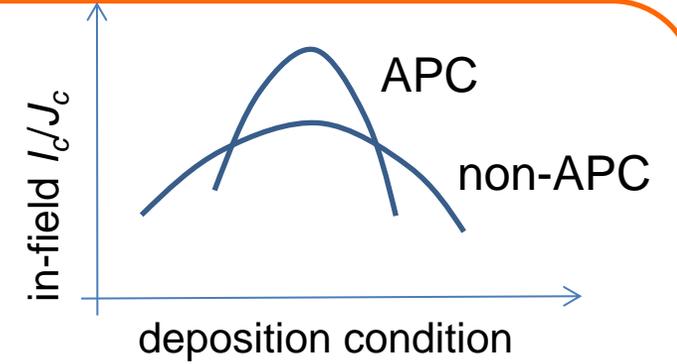
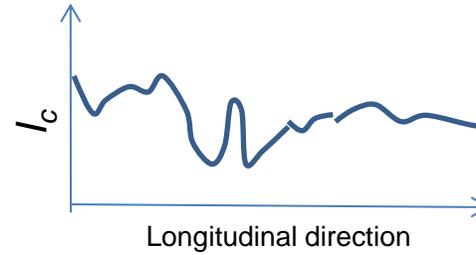


- ◆ High growth rate obtained just by high power laser
- ◆ which allows high throughput even with not too large deposition area
- ◆ Controllability maintains with simple deposition parameters

# Why uniform? Development of substrate heating stability

## Long-length & Longitudinal $I_c$ uniformity

Depends strongly on temperature stability during continuous deposition at **growth area**



Hot-wall PLD system has **furnace-like stable substrate heating**

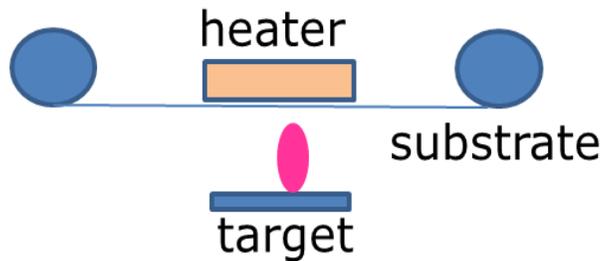
Window width  $Y < Gd < Eu$   
**APC < non-APC**

## Heater block system

### Single-lane PLD

Used 1990s-1999

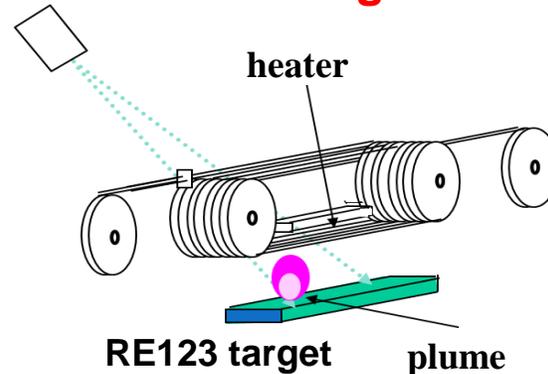
**Short piece ~ 10s m long**



### Multi-lane / beam scanned

Used 2000-2008

**~100s m long**

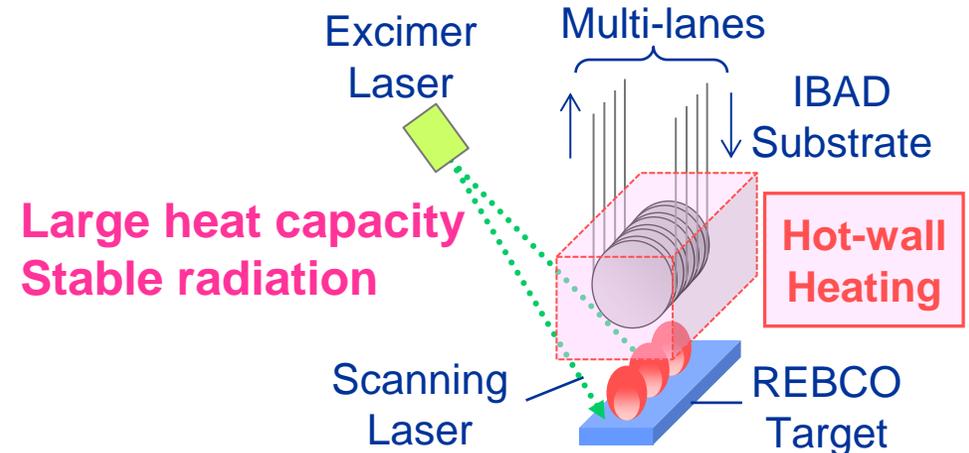


## Hot-wall PLD system

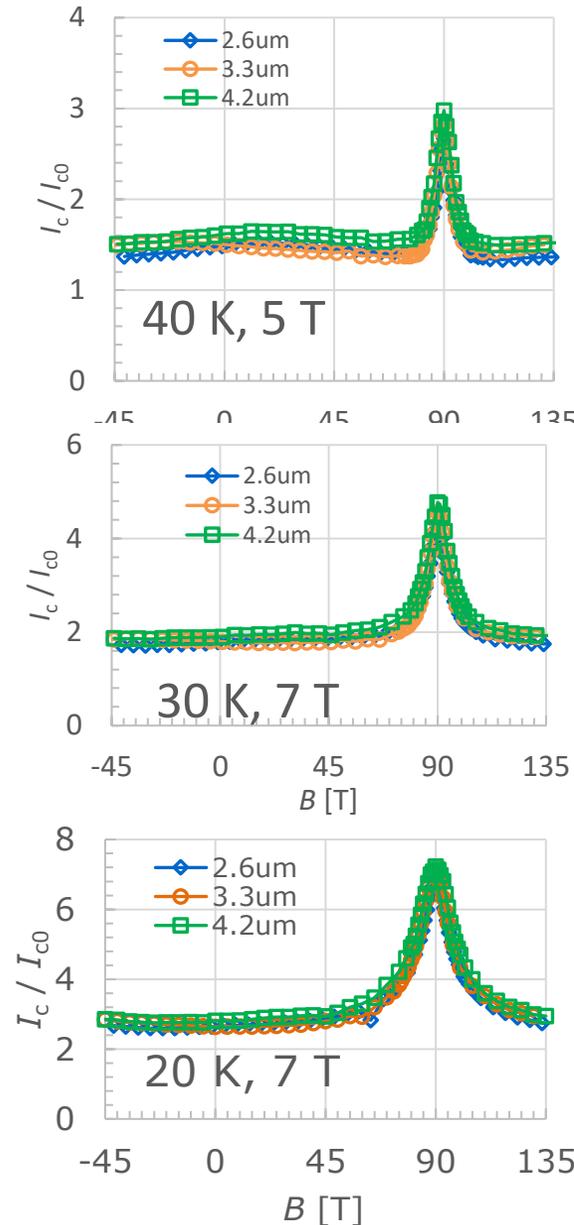
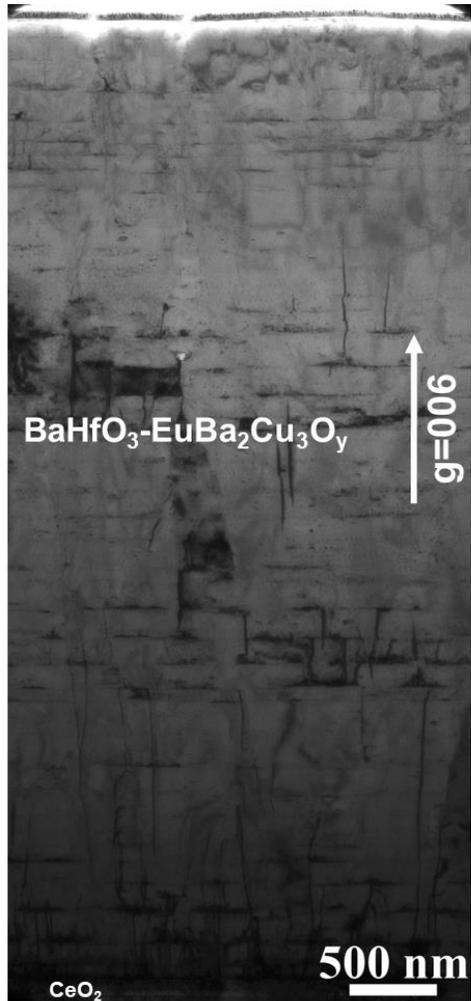
Initial set up 2003-2008

reformed 2016-2018

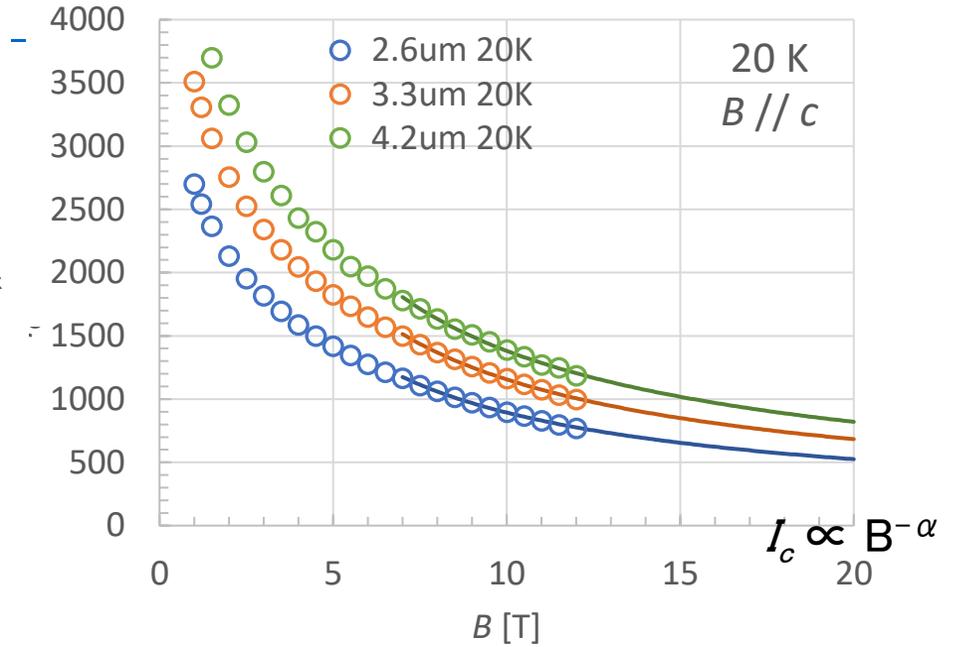
**>1 km long**



# Thickness dependence for in-field $I_c$ properties of BHO-EuBCO



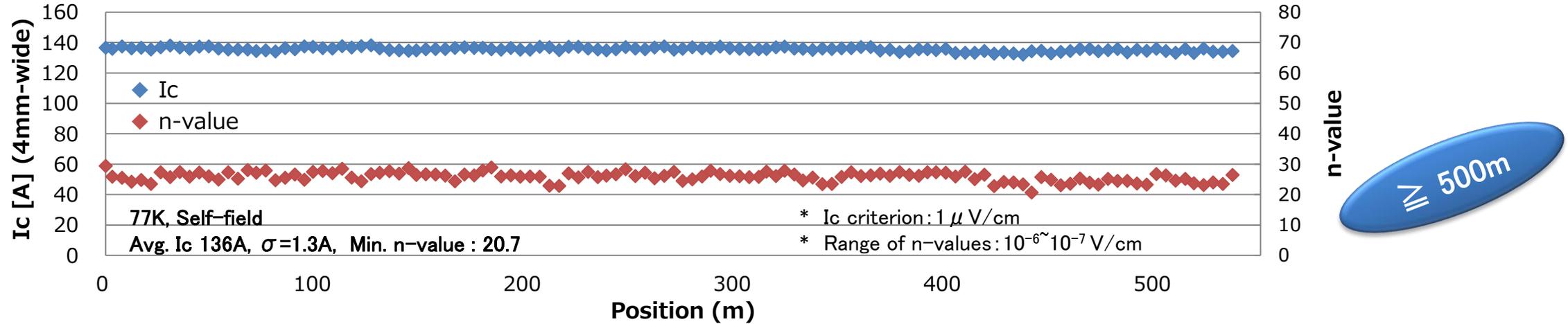
Almost the same  $I_c$ - $B$ - $\theta$  shape up to 4.2  $\mu\text{m}$  thick



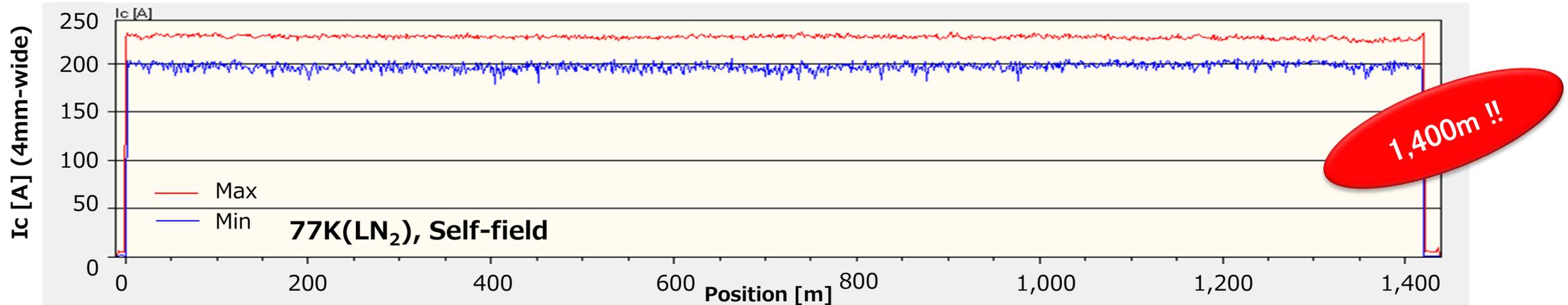
$J_e = 595 \text{ A/mm}^2$  ( $I_c = 357 \text{ A}$ , 4mm wide)  
@30K, 7T  
obtained with 2.5  $\mu\text{m}$  thick  
at high throughput

# Example data of longitudinal $I_c$ uniformity of 4mm-wide tape

- Measured by Current conduction measurement every 4.7 m (with APC / FESC-SCH04)

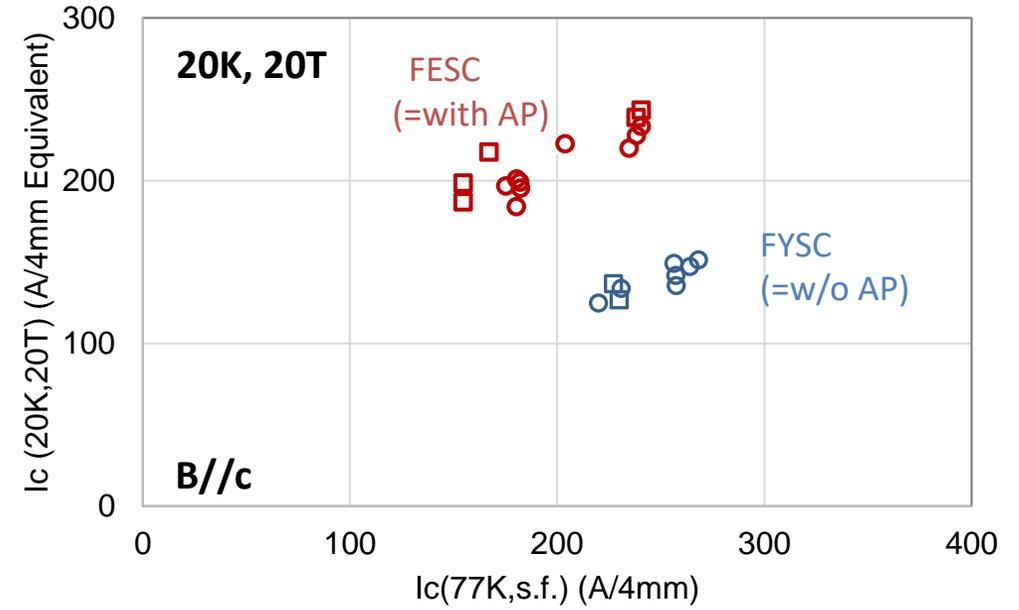
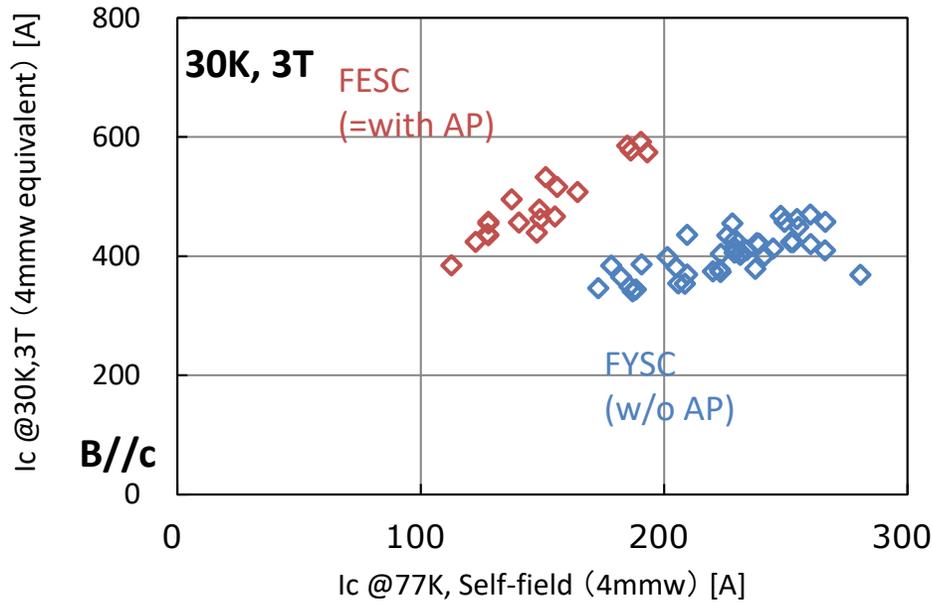


- Magnetic measurement @Tapestar™ (4mm-wide with APC / FESC-SCH04)

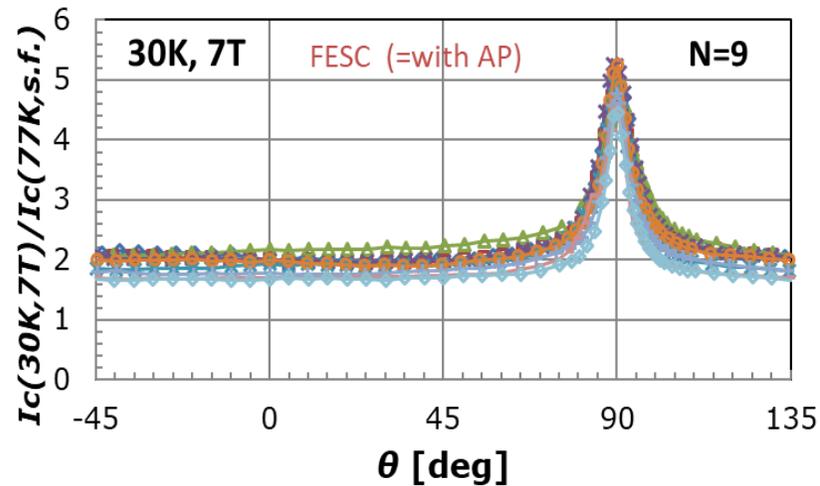


# Lot-to-lot in-field $I_c$ distribution of 4 mm<sup>w</sup> wire

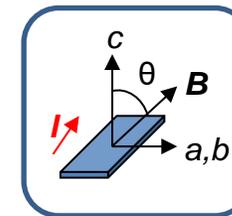
## ■ rot-to-rot variation of in-field $I_c / I_c$ (77 K, s.f.)



Good correlation to self field  $I_c$  and in-field  $I_c$  observed for both EuBCO+BHO and pristine GdBCO



◇○ measured at Fujikura, and exploited values with  $I_c$   
 □ in-field  $I_c$  measured at Tohoku university

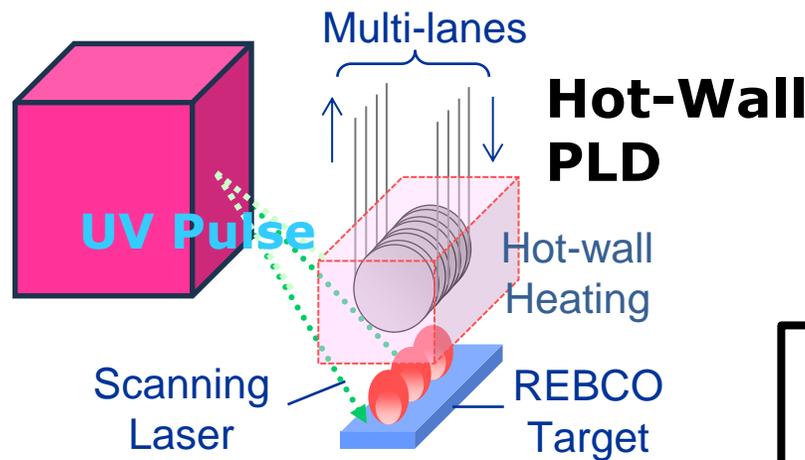


# General evaluation of PLD-REBCO process

- Excimer laser cost has effectively been compensated by good throughput and excellent production yield of PLD with wide process window.

$$\text{Cost}_{\text{REBCO}} \sim \left( \text{Cost}_{\text{op.}} + \left( \frac{\text{Cost}_{\text{equipment}}}{\text{Throughput} \times T_{\text{op.}}} \right) \right) \times \frac{1}{\text{Production Yield}}$$

## Industrial Excimer Laser



### ◆ High growth rate

- ◆ High materials yield
- ◆ High supersaturated gas
- ◆ High adatom mobility

### ◆ Laser power/lifetime

- ◆ Still improving by strong demand of FPD industry

PLD should still contribute for tough final goal of C.C. with low cost, & high quality by a reliable & accountable mass production approach

### ◆ Reproducibility

- ◆ Large oxygen window without differential pumping or chemical decomposition
- ◆ Cation Stoichiometric stability with fine evaporation by UV

### ◆ Jc/Ic in high field

- ◆ Point defects & secondary phase APC introduced just by simple vapor process

### ◆ Uniformity

- ◆ Growth temperature control succeeded by Hot Wall

# Typical Specifications of 2G HTS Tape at Fujikura

Products	Width [mm]	Thickness [mm]	Substrate [ $\mu\text{m}$ ]	Stabilizer [ $\mu\text{m}$ ] <sup>*5</sup>	APC Option	Critical Current [A]	
						77K, S.F.	20K, 5T (Ref.) <sup>*4</sup>
<b>FYSC-SCH04</b>	4	0.13	75	20	Non-AP <sup>*2</sup>	$\geq 165$	368
FYSC-SCH12	12	0.13	75	20	Non-AP <sup>*2</sup>	$\geq 550$	1,104
FYSC-S12 <sup>*1</sup>	12	0.08	75	—	Non-AP <sup>*2</sup>	$\geq 550$	—
FESC-SCH02	2	0.11	50	20	AP <sup>*3</sup>	$\geq 30$	257
FESC-SCH03	3	0.11	50	20	AP <sup>*3</sup>	$\geq 63$	497
<b>FESC-SCH04</b>	4	0.11	50	20	AP <sup>*3</sup>	$\geq 85$	663
FESC-SCH04(05)	4	0.07	50	5	AP <sup>*3</sup>	$\geq 85$	663
FESC-SCH12	12	0.11	50	20	AP <sup>*3</sup>	$\geq 250$	1,990
FESC-S12 <sup>*1</sup>	12	0.06	50	—	AP <sup>*3</sup>	$\geq 250$	—

\*1 Non-copper stabilizer specification is available in only 12mm-wide for current lead or low thermal conducting applications.

\*2 Non-AP specification is mainly for conductors or other general use at relatively higher temperature.

\*3 Artificial pinning specification is mainly for use in magnet applications at low temperature and high magnetic field.

\*4  $I_c@20\text{K}, 5\text{T}$  is a reference value and no guarantee of the actual performance.

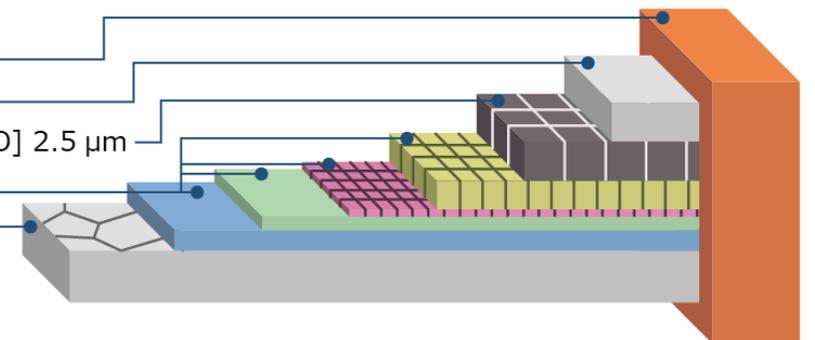
\*5 If requested, an option **customizing copper plating thickness is also available**. (e.g., 5 $\mu\text{m}$ , 10 $\mu\text{m}$  or 40 $\mu\text{m}$ )

- FYSC(w/o APC: **GdBaCuO**) is mainly for power cables or other general use at relatively higher temperature.
- FESC(w/ APC: **EuBaCuO+BaHfO**) is recommendable for use in magnet applications at lower temperature and higher field.



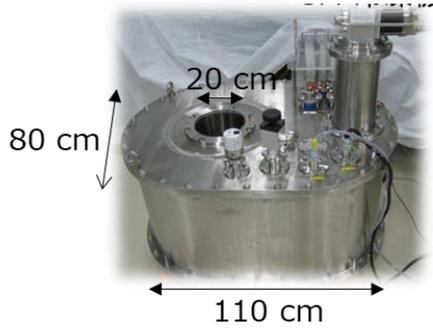
## <Schematic of RE-based HTS tape>

- Stabilizer [Cu plating] 20 $\mu\text{m}$
- Protection layer [Ag] 2 $\mu\text{m}$
- Superconducting Layer [GdBCO] 2 $\mu\text{m}$  / [EuBCO+BHO] 2.5 $\mu\text{m}$
- Buffer layer [MgO, etc.] 0.7 $\mu\text{m}$
- Substrate [Hastelloy®] 75 / 50 $\mu\text{m}$



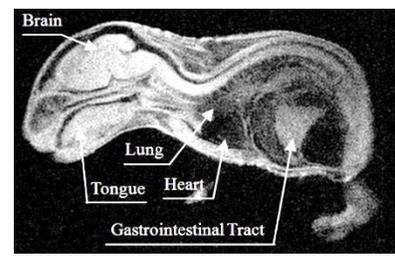
# Demand from magnet application for IBAD/PLD REBCO wires

■ 5 T cryocooled magnet by Fujikura Ltd. (2012)

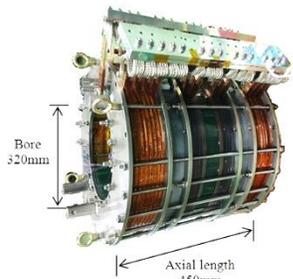


>400kJ with 20 cm RT bore

■ World 1<sup>st</sup> 3T MRI by Mitsubishi Electric (2015, 2016-2018)



1/3 demo of drive mode 3 T class MRI (AMED/NEDO)

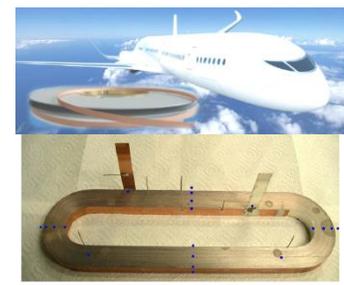


Airbus, KIT, Siemens  
Presented at EUCAS 2019

■ World 1<sup>st</sup> 1.2GHz NMR by BRUKER (2019)

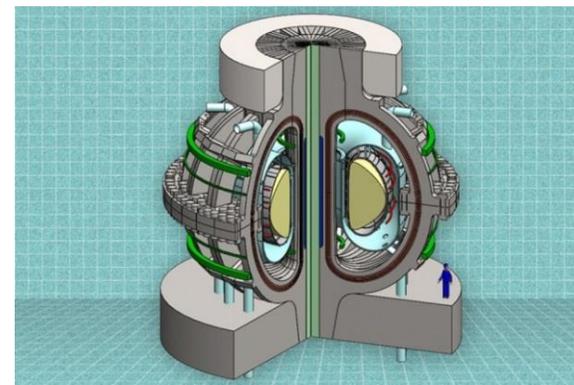


■ TELOS Project (2016-2019)



- ◆ Drastic improvement toward high-field inner magnet for NMR in 2010s
  - ◆ Ic uniformity and Mechanical reliability of IBAD/PLD REBCO wires
  - ◆ Coiling technology of insulated wires avoiding delamination
- ◆ NEW era started for large scale magnet as fusion etc. requires
  - ◆ NI technology / assembled conductors used for large current operation
  - ◆ Quench protection for both NI and insulated magnet

■ Compact Tokamak by CFS (2022-)

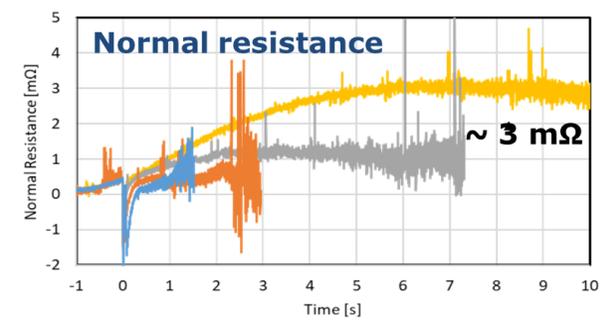
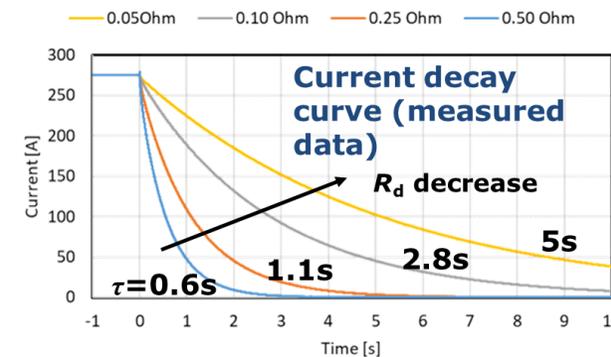
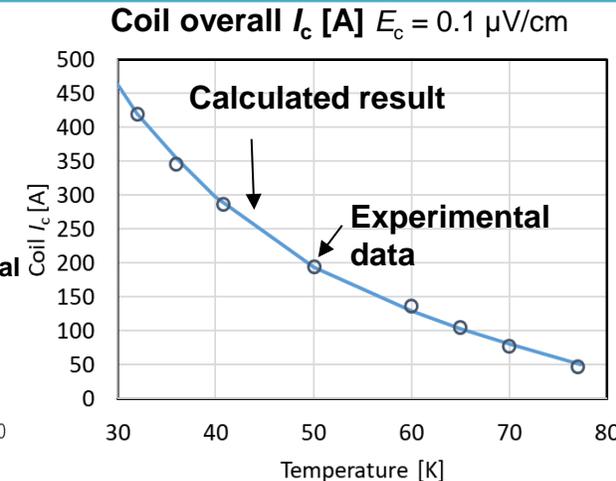
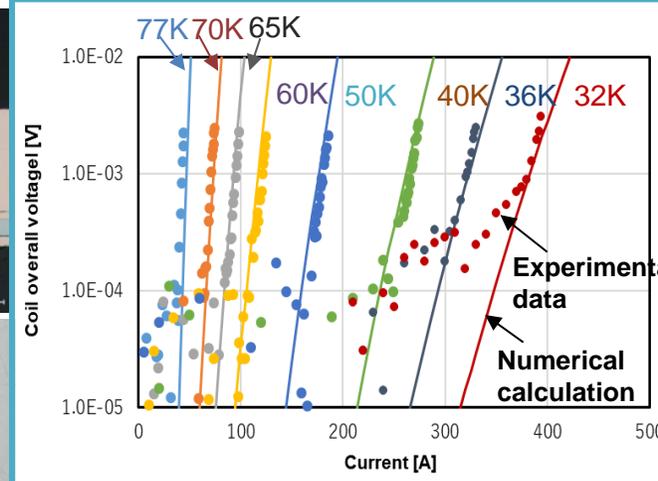
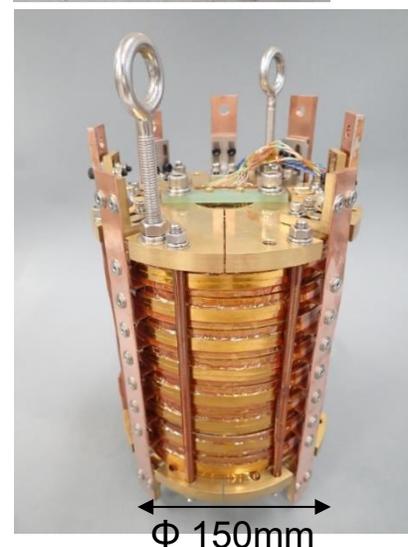
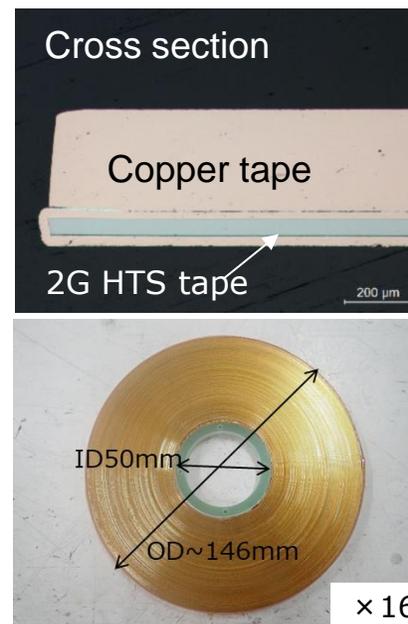


Ic uniformity anyway important

# 10T small test coil to check $I_c$ uniformity & quench protection

Parameters	REBCO tape
Substrate thick.	50 $\mu\text{m}$
Copper thick.	20 $\mu\text{m}$ $\times$ 2 (plates) + 300 $\mu\text{m}$
Type of HTS tape	FESC-SCH04
Insulation	Fluorine coating polyimide tape/Polyimide tape
Width/Thickness	4.1 mm / 0.47 mm

Parameters	10 T test coil	
Inner diameter	50 mm	
Outer diameter	146 mm	
Coil height	166 mm	
Impregnation	Epoxy resin	
No. of pan cake	32 (2 $\times$ 16)	
Number of turns	2976 (93 $\times$ 32)	
Tape length	0.9 km	
$I_{op}$	300 A	500 A
$B_0$	5.8 T	9.7 T
Stored energy	13 kJ	35 kJ
Load factor at 20 K	44%	73%



- **Good agreement** between experimental and calculated results for coil  $I_c$
- **No degradation** observed after quench test.

# Summary

- **IBAD/PLD: most reliable process to answer current business demand**
  - **strong mechanical strength, in-field high-Jc** and production reliability.
- **The advantage of Hot-wall PLD process covers laser cost well under mass production so far**
  - **Reproducibility:** controllable near best phase point with simple parameters
  - **High in-field Ic :** small and dispersed pinning centers by vapor deposition
  - **High growth rate:** high transfer yield and adequate adatom energy
  - **Uniformity:** temperature stability improved by Hot-wall architecture
- **Uniformity is still essential for magnet applications**
  - **Demand** of magnet technology: **Uniformity** and **Mechanical reliability**
  - **New area:** **NI technology, assembled conductors, quench protection.....**  
,,,also require **uniformity**

**PLD should contribute for tough final goal of C.C.  
with **low cost & high quality** as a **reliable approach****

**Scale-up investment is on-going at Fujikura**

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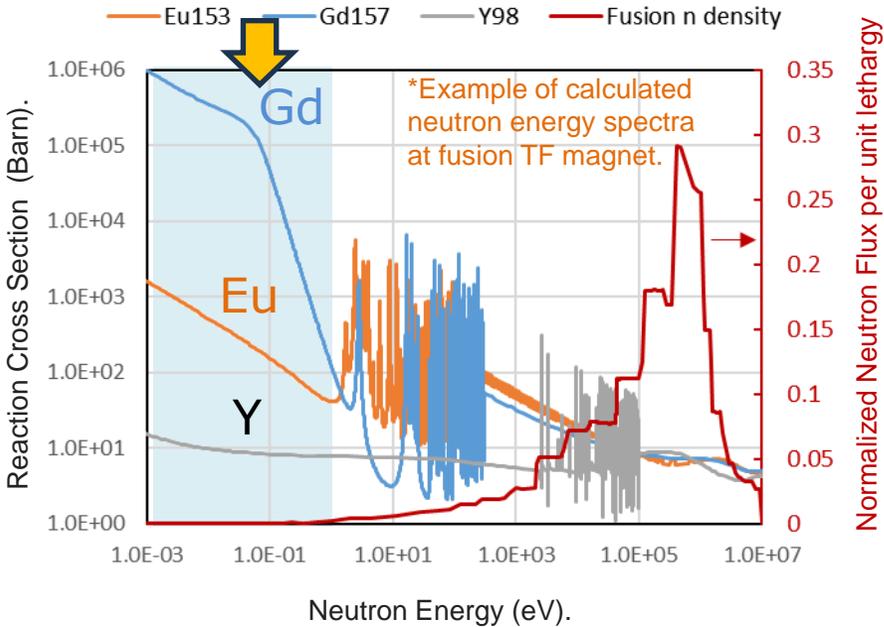
**END**

**Thank you for attention**

# RE elemental dependence of neutron radiation damage

Large cross section of neutron capture for Gd/Eu isotopes ~ 1 eV anticipated to damage lattice structures by gamma decay with large recoil energy

RE=Gd, Eu: irradiation strength should be carefully evaluated for fusion environment

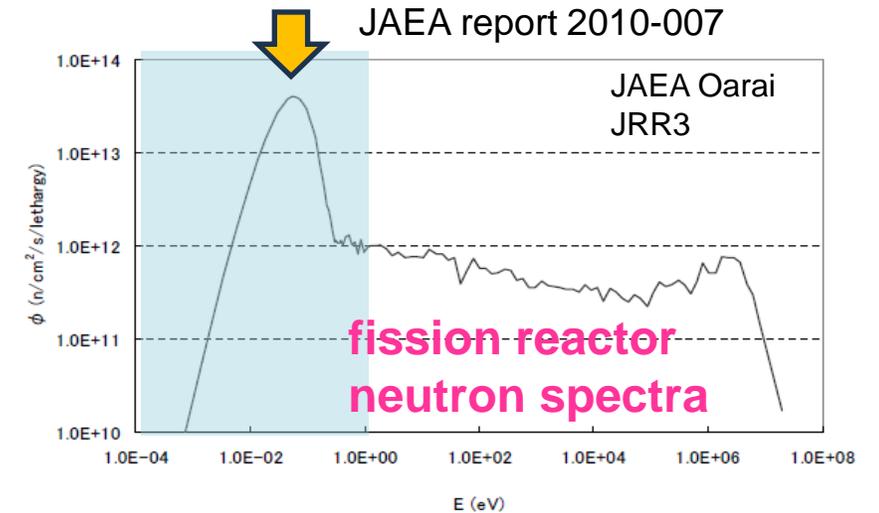


Fission reactor environment often used for irradiation tests has big peak in neutron spectra below 1 eV come from cooling water

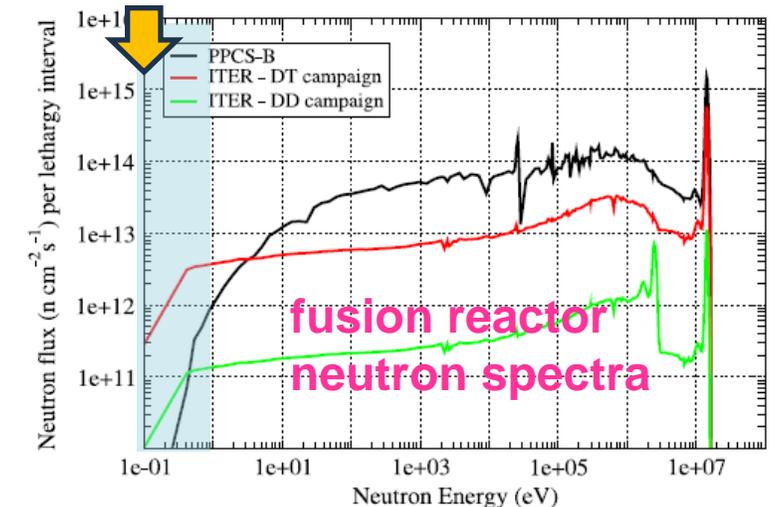
where fusion ones estimated by calculation have quite low flux there

K. Shibata, et al., "JENDL-4.0: A New Library for Nuclear Science and Engineering," *J. Nucl. Sci. Technol.* **48**(1), 1-30 (2011).

\* Weber, H.: *Int. J. Mod. Phys. E* 20.06 (2011): 1325-1378.



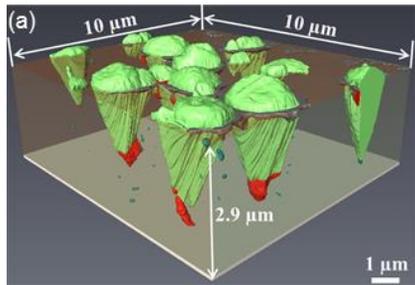
M.R.Gilbert and J.Ch.Sublet: *Nuclear Fusion* 2011 **51** 043005



# RE elemental dependent growth stability for BMO-REBCO

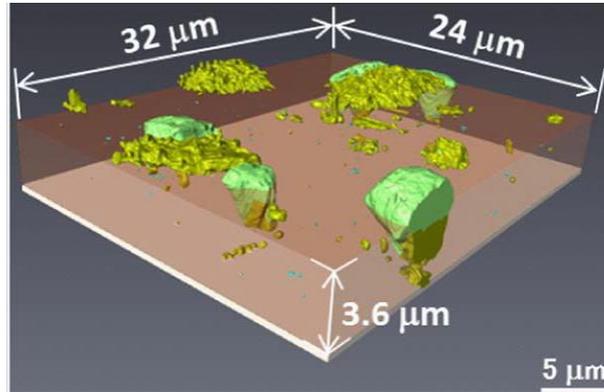
FIB-SEM 3D observation on misoriented grains (mainly a-axis aligned normal) for thick BMO-REBCO films

BaHfO-GdBaCuO



[https://www.jfcc.or.jp/re\\_sult/16r33.html](https://www.jfcc.or.jp/re_sult/16r33.html)

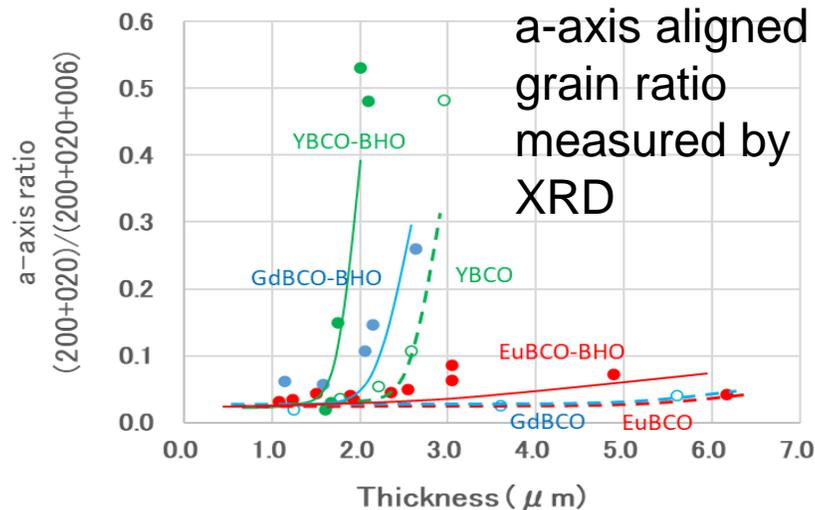
BaHfO-EuBaCuO



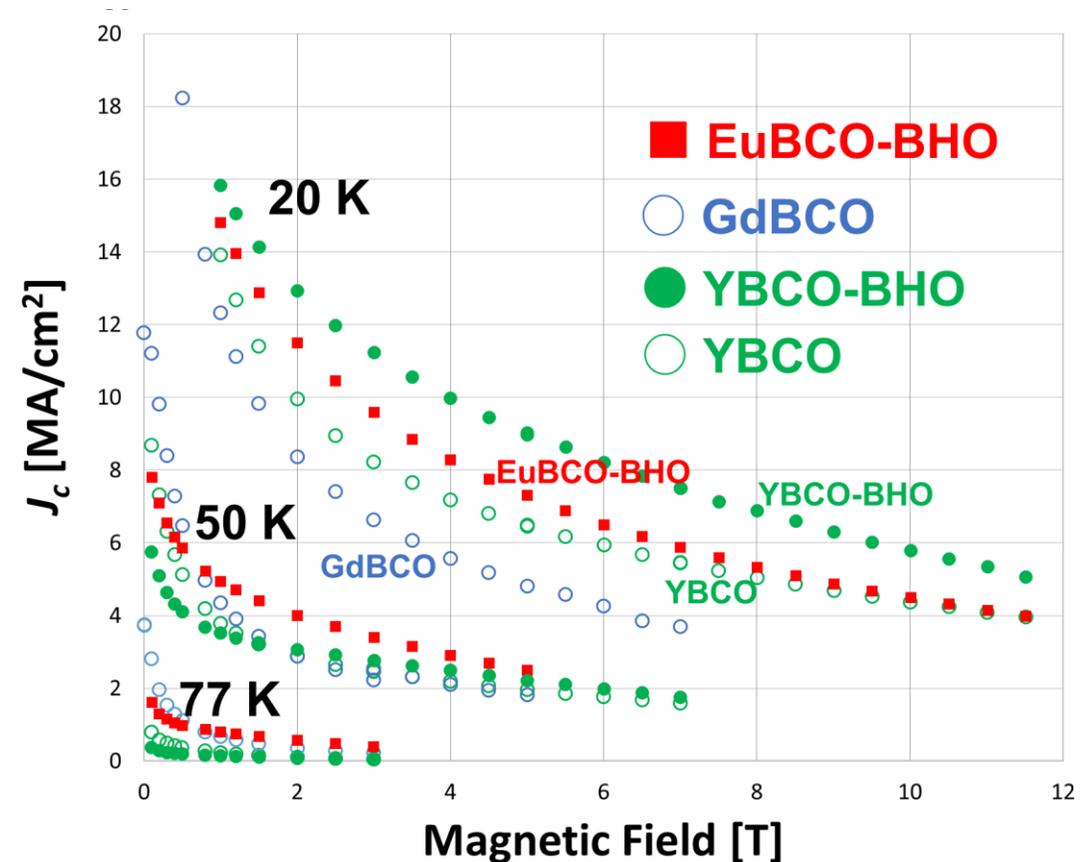
D. Yokoe et al., Supercond.Sci.Technol. **33** (2020) 024002  
T. Yoshida et al., Physica C **504** (2014) 42

Oriented growth stability

EuBCO  
>GdBCO  
>YBCO



Typical  $J_c$ -B characteristics for BHO-EuBCO and pristine GdBCO, YBCO films



# Comparison of transporting $I_c$ and numerically estimated $I_c$ for wounded coil of BMO-doped EuBCO wire

S.Muto et.al., Abstracts of CSSJ Conference, vol. 98 (2019) p.124.

## $I_c(\theta, B)$ fitting for 2 peak

$$I_c(\theta, B) = a_1 f_1(\omega_1(B), \theta) + a_2 f_2(\omega_2(B), \theta)$$

$$f_1(\omega_1(B), \theta) = \frac{1}{\omega_1^2 \cos^2 \theta + \sin^2 \theta} \quad \leftarrow B//c$$

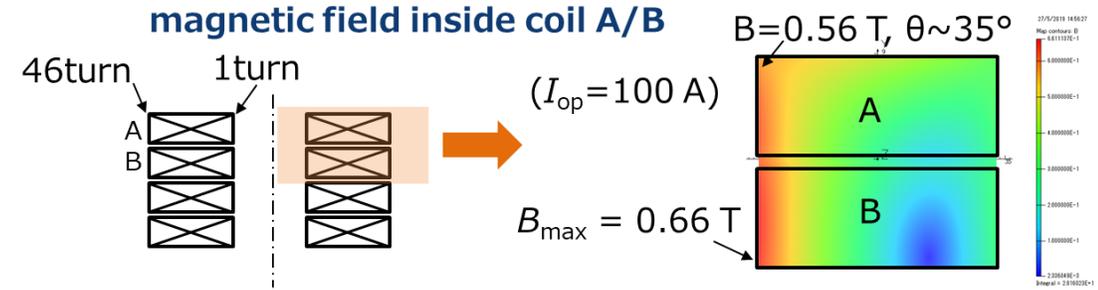
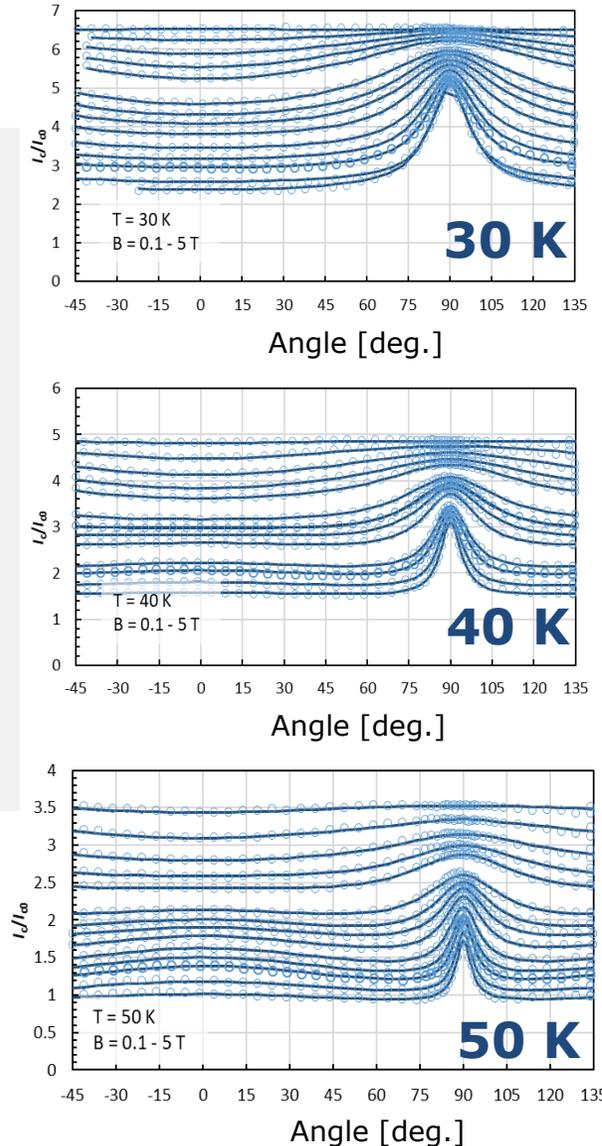
$$f_2(\omega_2(B), \theta) = \frac{1}{\sqrt{\omega_2^2 \sin^2 \theta + \cos^2 \theta}} \quad \leftarrow B//ab$$

$$I_c(0^\circ, B) = \frac{a_1}{\omega_1^2(B)} + a_2$$

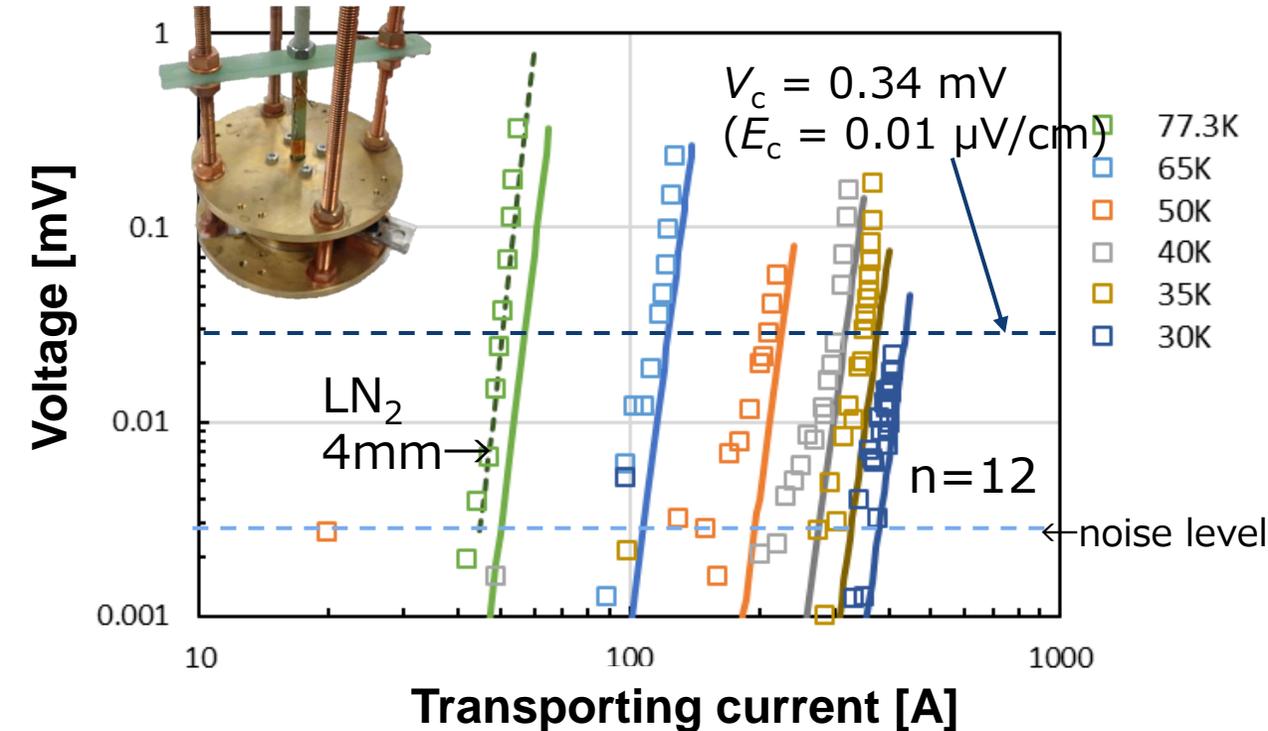
$$I_c(90^\circ, B) = a_1 + \frac{a_2}{\omega_2(B)}$$

fitting parameter:  $\omega_1(B), \omega_2(B)$

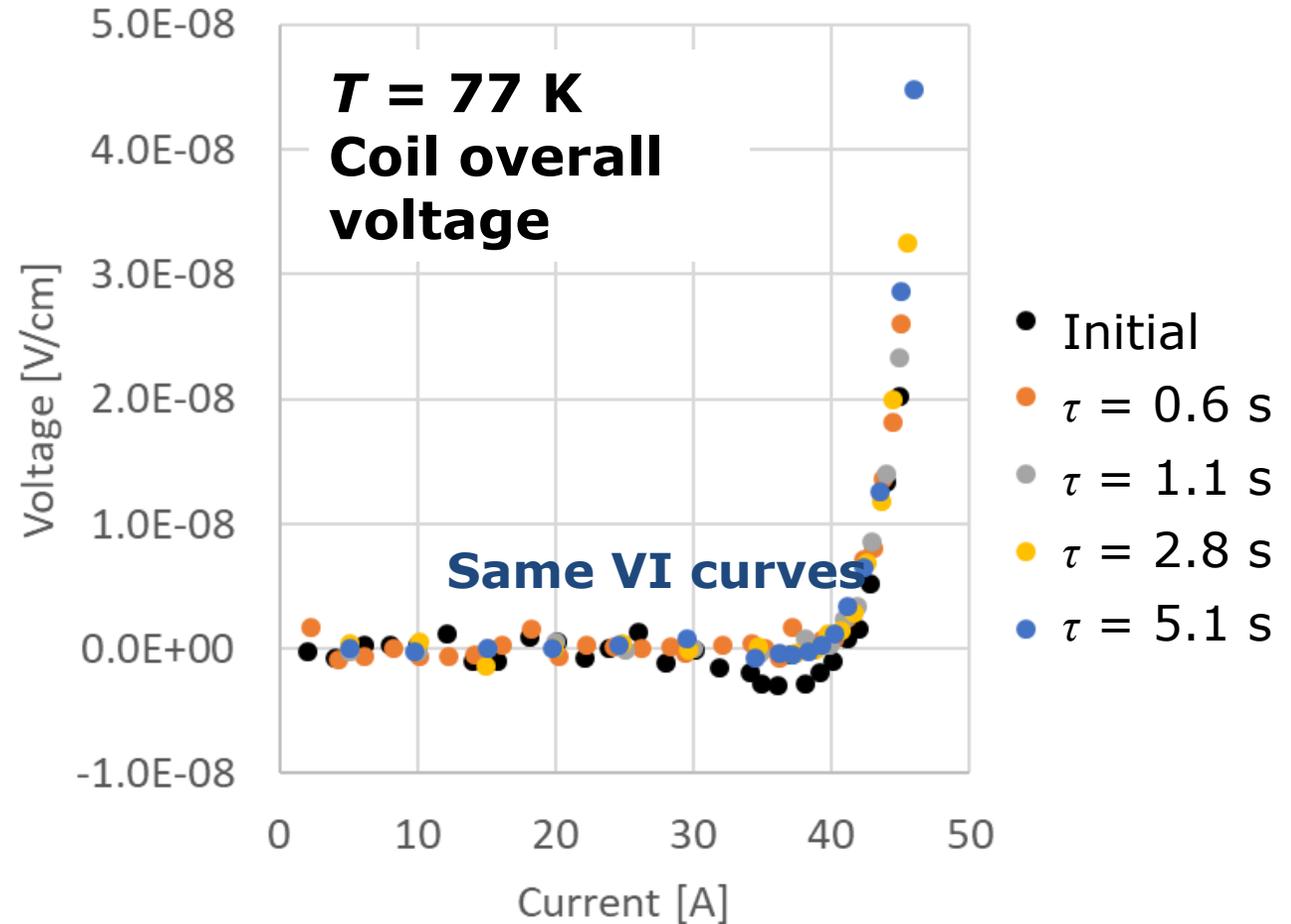
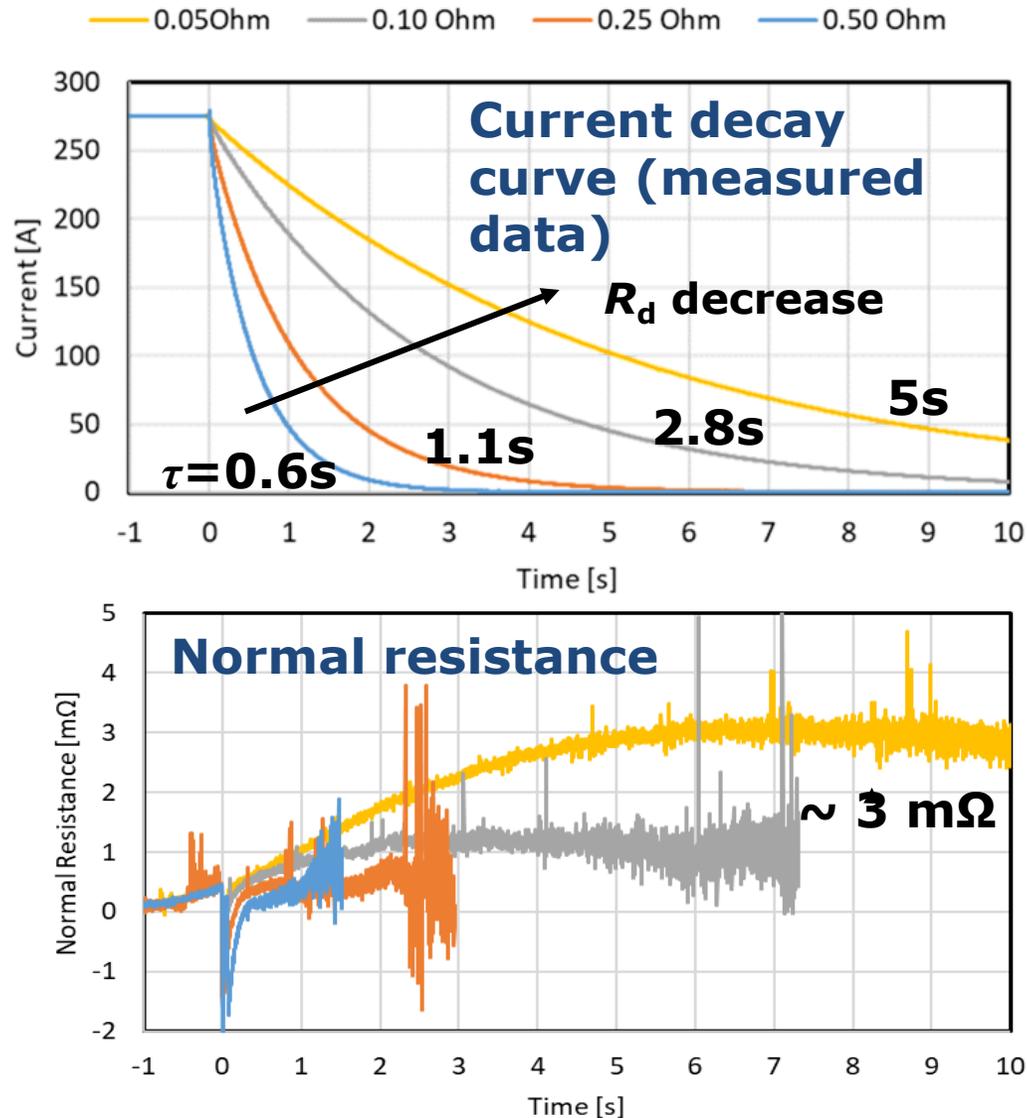
arranged from the equation derived by  
D.K.Hillton et. al., SuST 28 (2015) 074002



## 4-layer single pancake coils



# No degradation observed after quench test

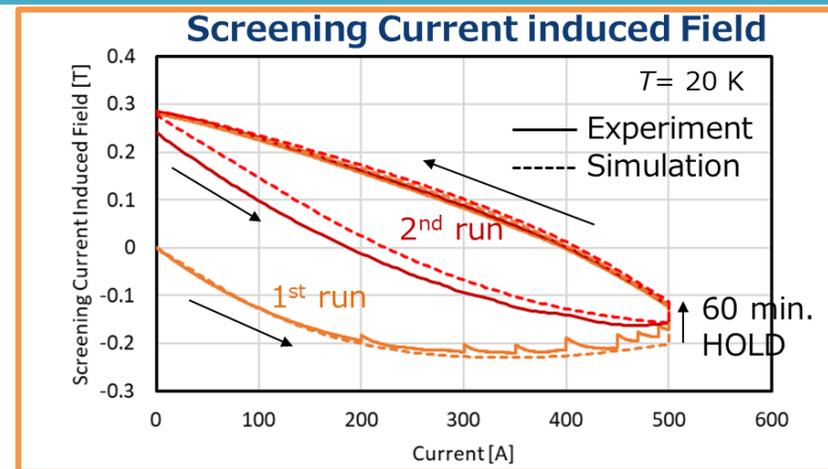
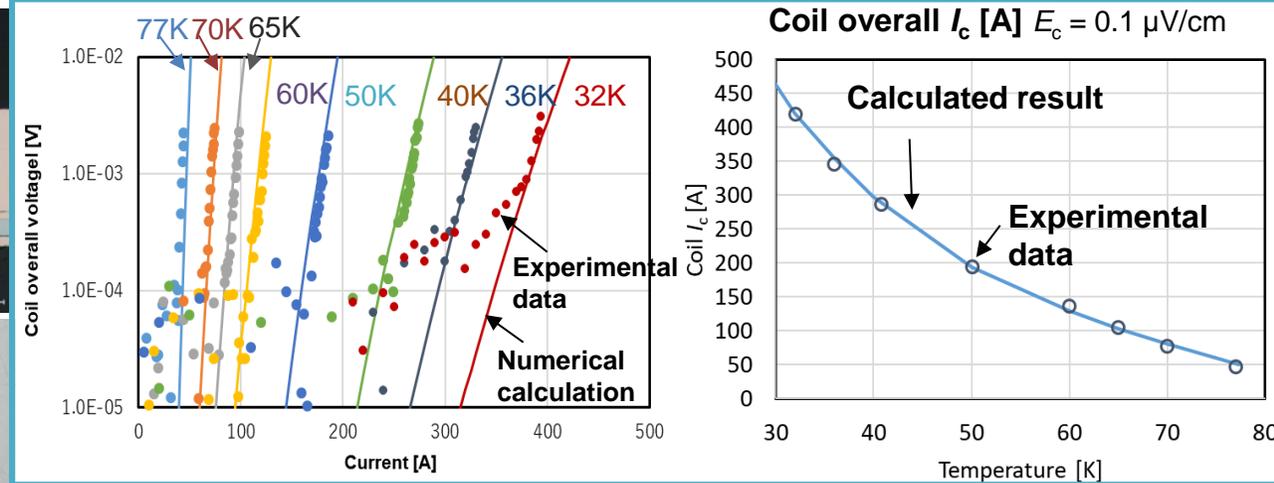
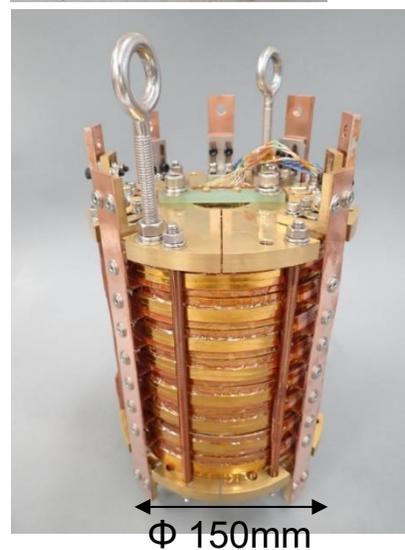
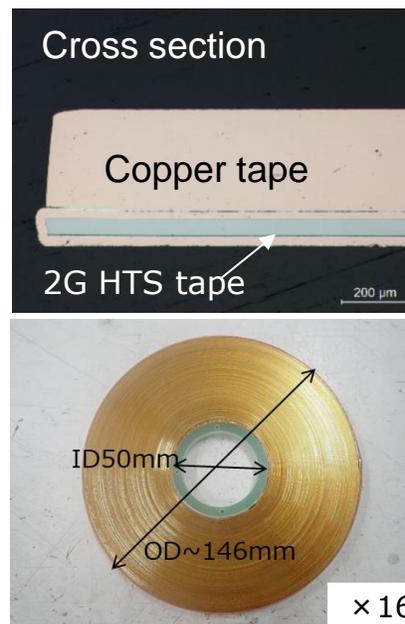


S. Muto et al. presented in MT-28, 2PoM03-2

# 10T small test coil to check $I_c$ uniformity & quench protection

Parameters	REBCO tape
Substrate thick.	50 $\mu\text{m}$
Copper thick.	20 $\mu\text{m}$ $\times$ 2 (plates) + 300 $\mu\text{m}$
Type of HTS tape	FESC-SCH04
Insulation	Fluorine coating polyimide tape/Polyimide tape
Width/Thickness	4.1 mm / 0.47 mm

Parameters	10 T test coil	
Inner diameter	50 mm	
Outer diameter	146 mm	
Coil height	166 mm	
Impregnation	Epoxy resin	
No. of pan cake	32 (2 $\times$ 16)	
Number of turns	2976 (93 $\times$ 32)	
Tape length	0.9 km	
$I_{op}$	300 A	500 A
$B_0$	5.8 T	9.7 T
Stored energy	13 kJ	35 kJ
Load factor at 20 K	44%	73%

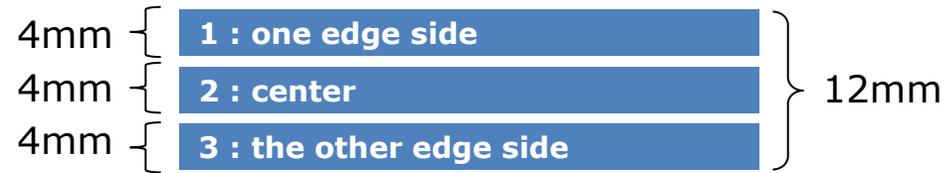


● **Good agreement between experimental and calculated results for coil  $I_c$  and screening current induced field.**

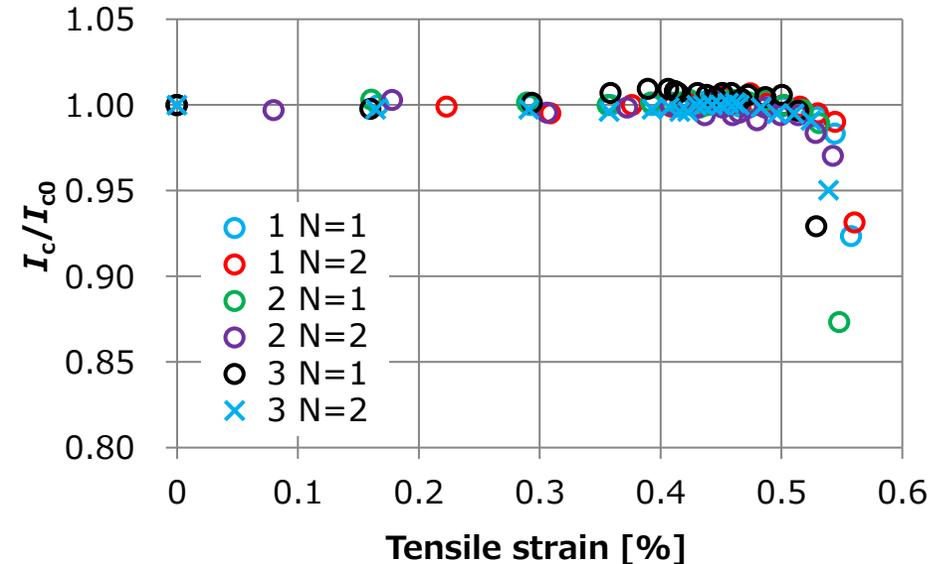
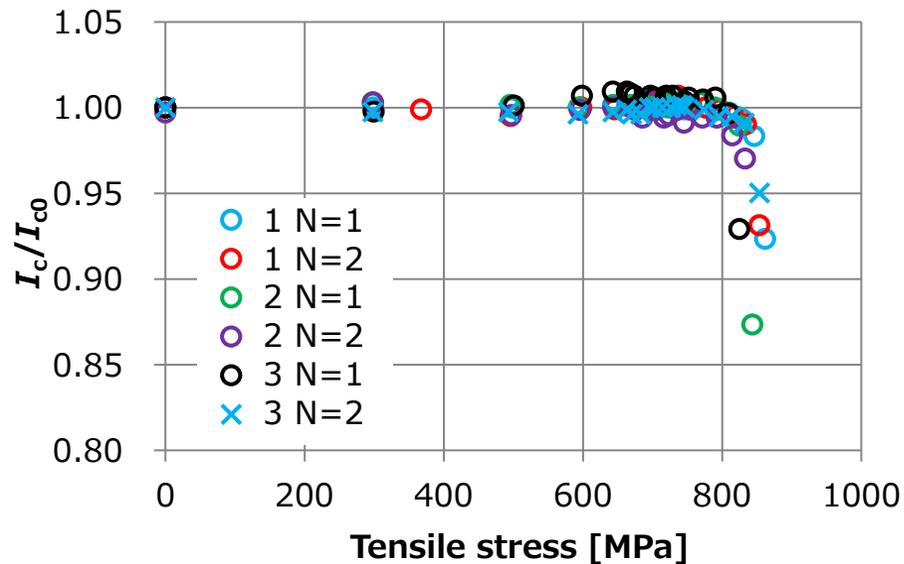
# Tensile Properties Evaluation of Divided 4 mm-wide

- Tensile properties of 3 parts of 4 mm-wide tapes divided from 12 mm-wide tape in LN<sub>2</sub>

Samples (FYSC-SCH04)		Reversible $I_c$	
		Strain [%]	Stress [MPa]
1	N=1	0.523	820
	N=2	0.513	817
2	N=1	0.521	813
	N=2	0.497	768
3	N=1	0.514	810
	N=2	0.496	795



\* Reversible  $I_c$  is defined at  $I_c/I_{c0} > 0.99$ . ( $I_{c0}$  : Initial)

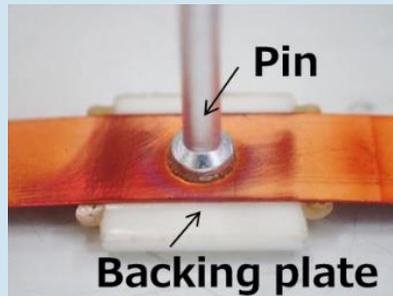
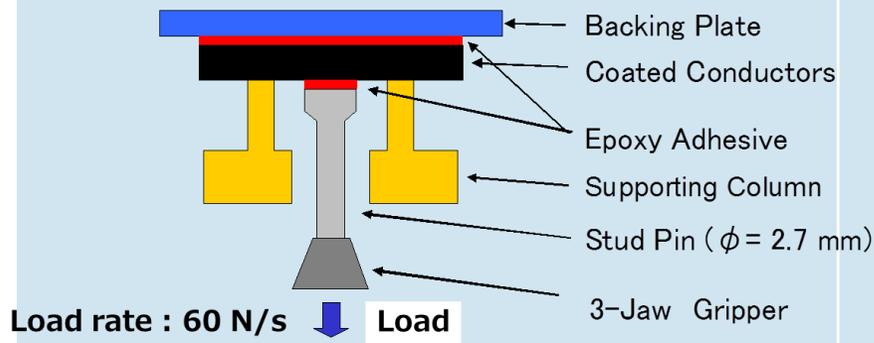


- Each divided 4 mm-wide HTS tapes by **laser slitting** have shown equivalent tensile properties.
- Laser slitting has been introduced to the production process at Fujikura for **over 10 years**.



# Evaluation method for delamination strength of REBCO tapes

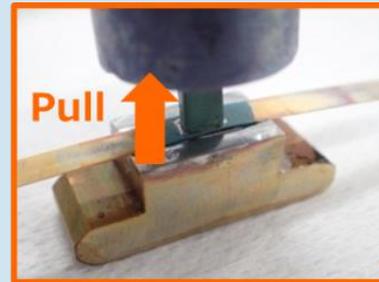
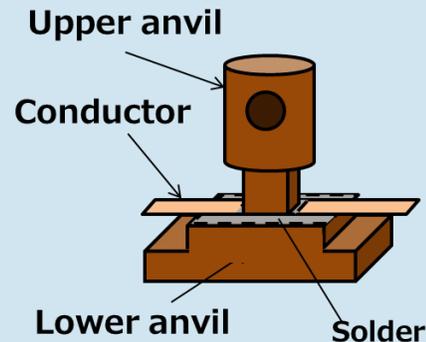
## Stud-pull Test



Test area: **Small** ( $5.7 \text{ mm}^2$ \*)  
\*in case of  $\phi 2.7$  mm Pin  
Delamination strength: **High**  
(~ several 10 MPa)

T. Oyama, *et al.*, Journal of The Surface Finishing Society of Japan, **58** (2007) P. 292

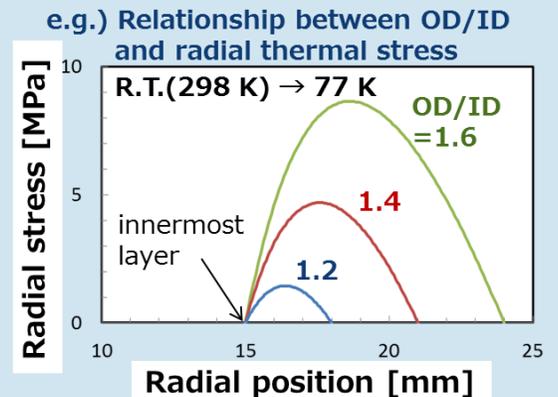
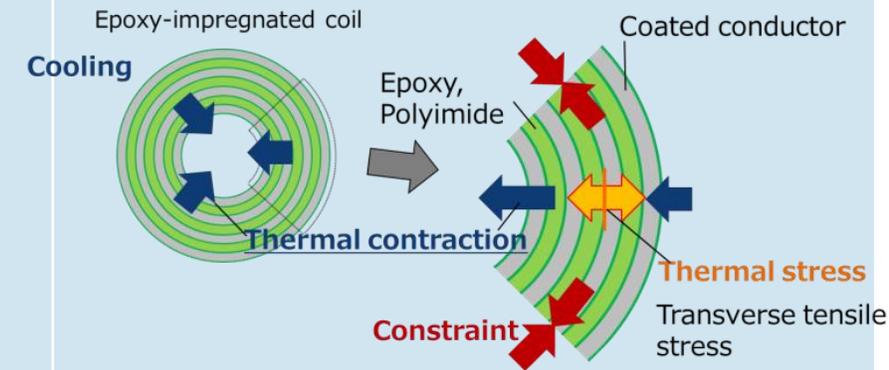
## Anvil Test



Test area: **Medium** ( $32 \text{ mm}^2$ \*)  
\*in case of  $4 \times 8$  mm Anvil  
Delamination strength: **Medium**

H. Shin, *et al.*, SuST, **27** (2014) 025001

## Impregnated Coil Test



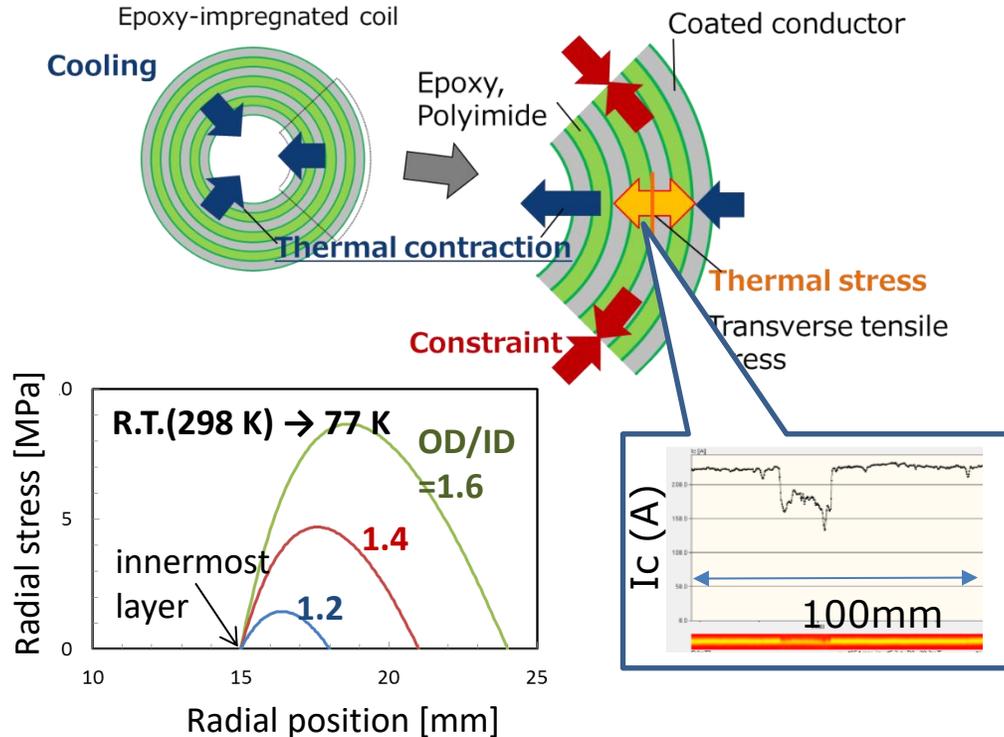
OD/ID: The ratio of outer diameter to inner diameter

Test area: **Large** ( $16000 \text{ mm}^2$ \*)  
\*in case of 4 mm wide  $\times$  4 m tape  
Delamination strength: **Low**  
(several MPa)

H. Miyazaki *et al.*, IEEE TAS, **25** (2015) 6602305

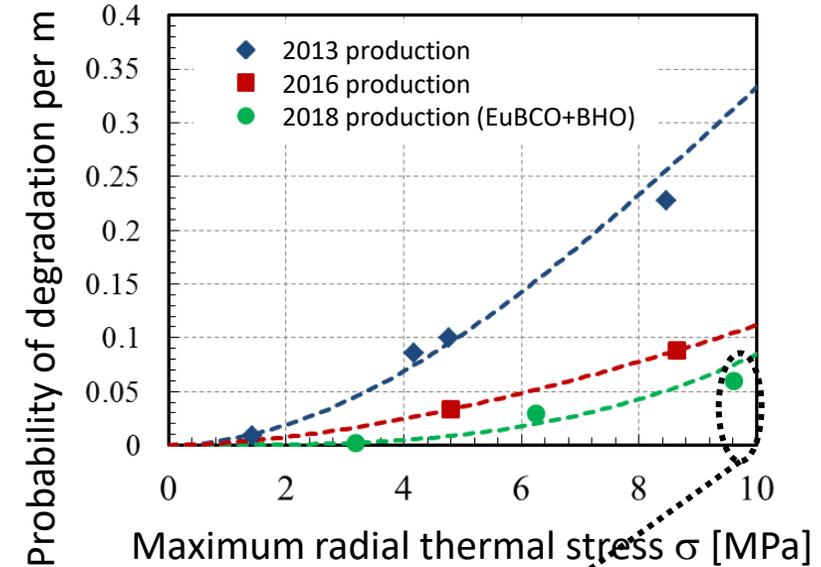
# Delamination strength evaluated by epoxy impregnated coils

## <Delamination stress test by thermal stress inside impregnated coil>



H. Miyazaki *et al.*, IEEE TAS, **25** (2015) 6602305

Average delamination stress of 2G HTS tapes have improved by Weibull analysis



## <Heat cycle test (R.T. ~ 77K)>

