

Reliable approach of mass production of REBCO coated conductor by using IBAD/hot-wall PLD for practical magnet applications

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Fujikura Ltd.

Acknowledgement:

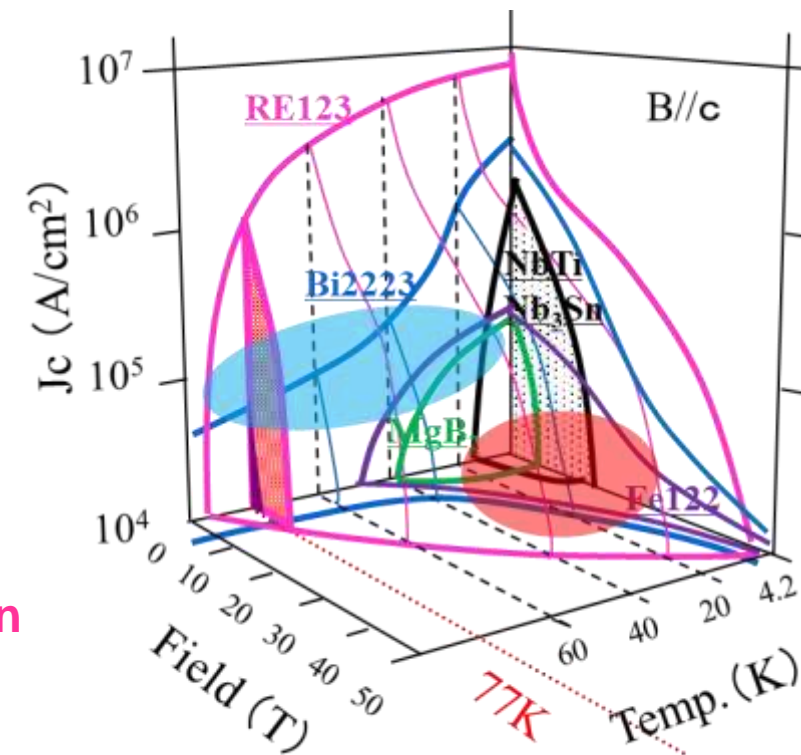
We appreciate Prof. T. Kiss at Kyushu Univ. and Prof. S. Awaji at Tohoku Univ. for collaboration to sample evaluation. A part of this work was also performed at the High Field Laboratory for Superconducting Materials, IMR, Tohoku University.

Contents

- **REBCO wire mass production started for magnet application**
 - Market started from **high-field application** : NMR, Fusion → **ONLY HTS reach over 20 T applications**
 - Demand of magnet technology: **Uniformity and Mechanical reliability**
 - Why peculiar vapor process of **IBAD/PLD** ?

- **Advantages of IBAD/PLD process for mass-production**
 - **Wide process window** come from:
 - IBAD substrates:
 - **Sharp texture, flat surface, strong mechanical strength**
 - PLD REBCO films:
 - **Reproducibility**: controllable near best phase point
 - **High in-field J_c** : small and dispersed pinning centers
 - **High growth rate**: high transfer yield, high supersaturation
 - **Uniformity**: Hot-wall architecture
 - **RE elemental dependance of process window**

- **REBCO wire lineup and neutron irradiation issues**
 - **RE elemental dependence of neutron collision cross section**
 - **Thermal neutron flux at SC magnets in Fusion reactor**



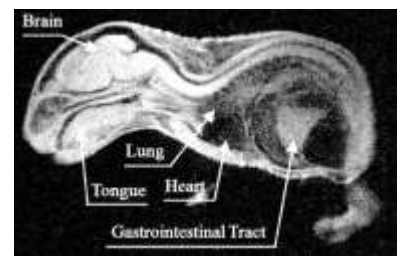
Demand from magnet application for IBAD/PLD REBCO wires

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

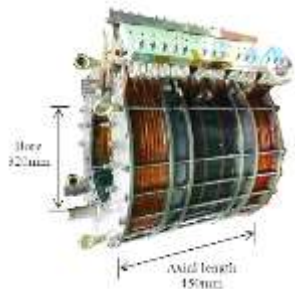


>400kJ with 20 cm RT bore

■ World 1st 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

■ TELOS Project (2016-2019)



■ World 1st 1.2GHz NMR by BRUKER (2019)

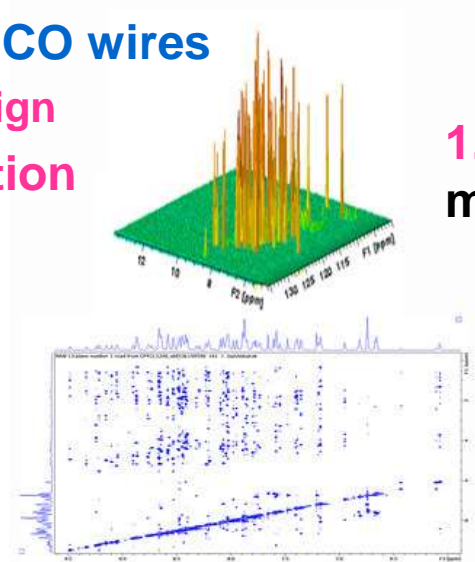


1.2 GHz NMR 28.2 T magnet with 54 mm bore

- ◆ Drastic improvement toward high-field DC magnet in 2010s
- ◆ **Ic uniformity and Mechanical reliability** of IBAD/PLD REBCO wires
 - ◆ Contribute to **accountability** required for **accurate field design**
 - ◆ **Coiling technology** of insulated wires avoiding **delamination**

First Commercial Practical REBCO device: high-end NMR

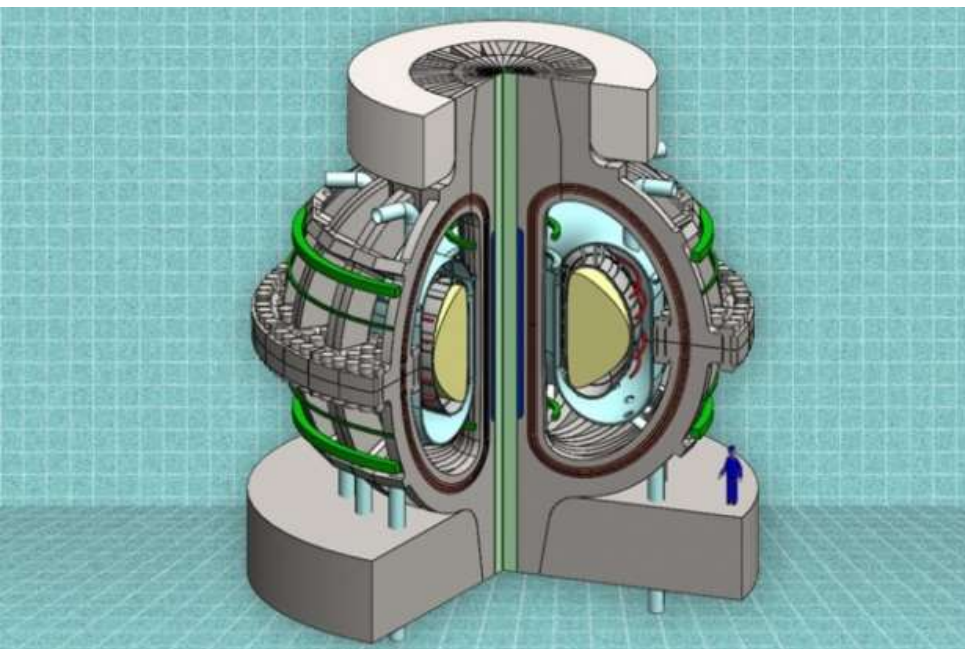
the most severe application strongly requires hoop stress durability & longitudinal Ic uniformity



1.0 GHz NMR with compact 23.5 T magnet

<https://ir.bruker.com/press-releases/press-release-details/2019/Bruker-Announces-Worlds-First-12-GHz-High-Resolution-Protein-NMR-Data/default.aspx>

Business scale demand of REBCO wires for fusion



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

Big REBCO wire demand up to 10000s km/reactor

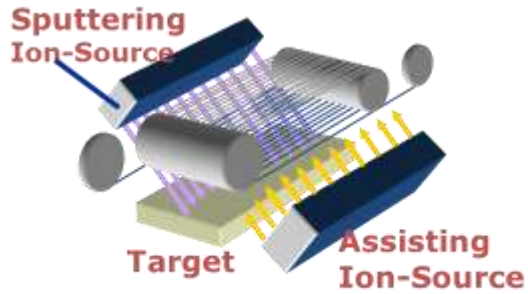
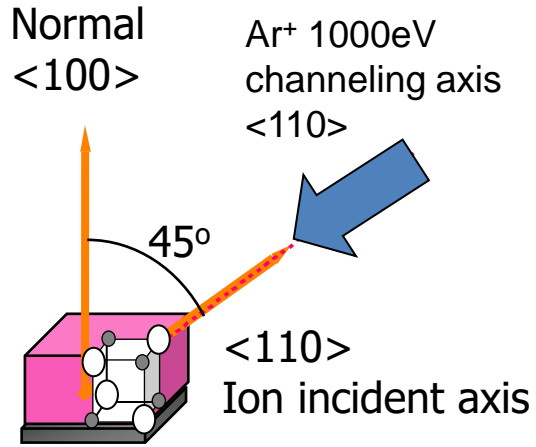
On going reactor program: SPARC (CFS), ST80 (Tokamak energy(TE))

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

- ◆ NEW era started for large scale magnet as fusion etc. requires
 - ◆ Tons of high-quality wires with desired delivery & cost
 - ◆ NI technology / assembled conductors used for large current operation
 - ◆ Quench protection for both NI and insulated magnet

Ic uniformity anyway important

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

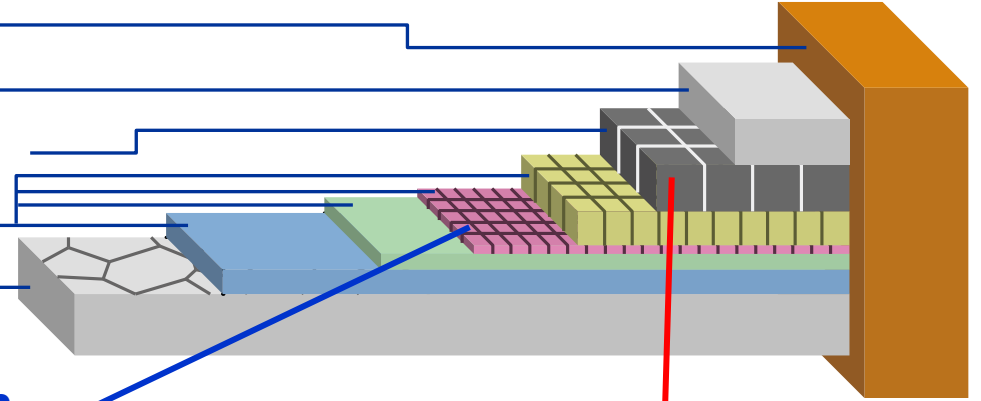


Reel-to-Reel IBAD system

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

<Schematic of 2G HTS wire>

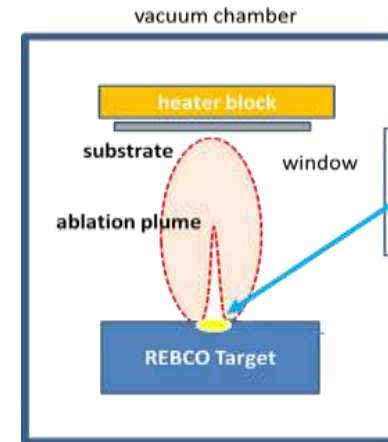
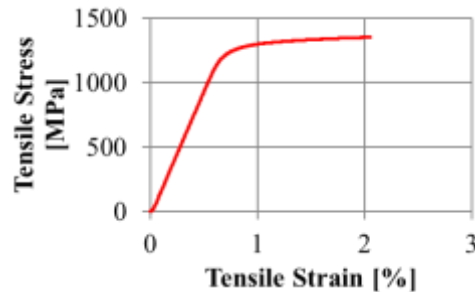
- Stabilizer [electroplated copper] 20 μm
- Protection layer [Ag] 2 μm ~
- HTS layer [GdBCO 2 μm] / [EuBCO+BHO 2.5 μm]
- Buffer layer [MgO, etc.] ~0.7 μm
- Substrate [Hastelloy®] 75 / 50 μm



Ion-Beam-Assisted Deposition (IBAD)



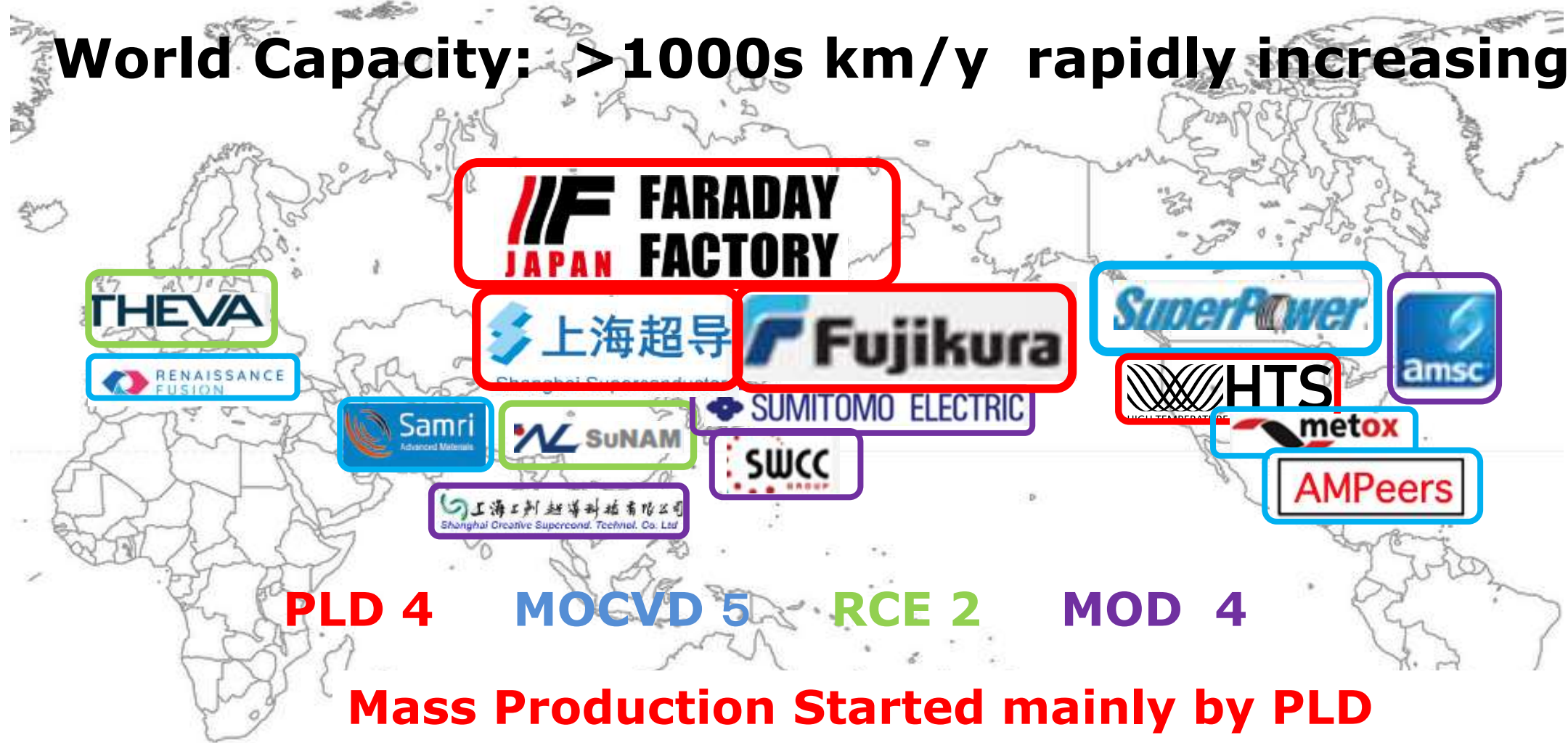
Pulsed Laser Deposition (PLD)



High quality REBCO films with desired growth condition

Manufacturer of REBCO C. C. 2024

World Capacity: >1000s km/y rapidly increasing

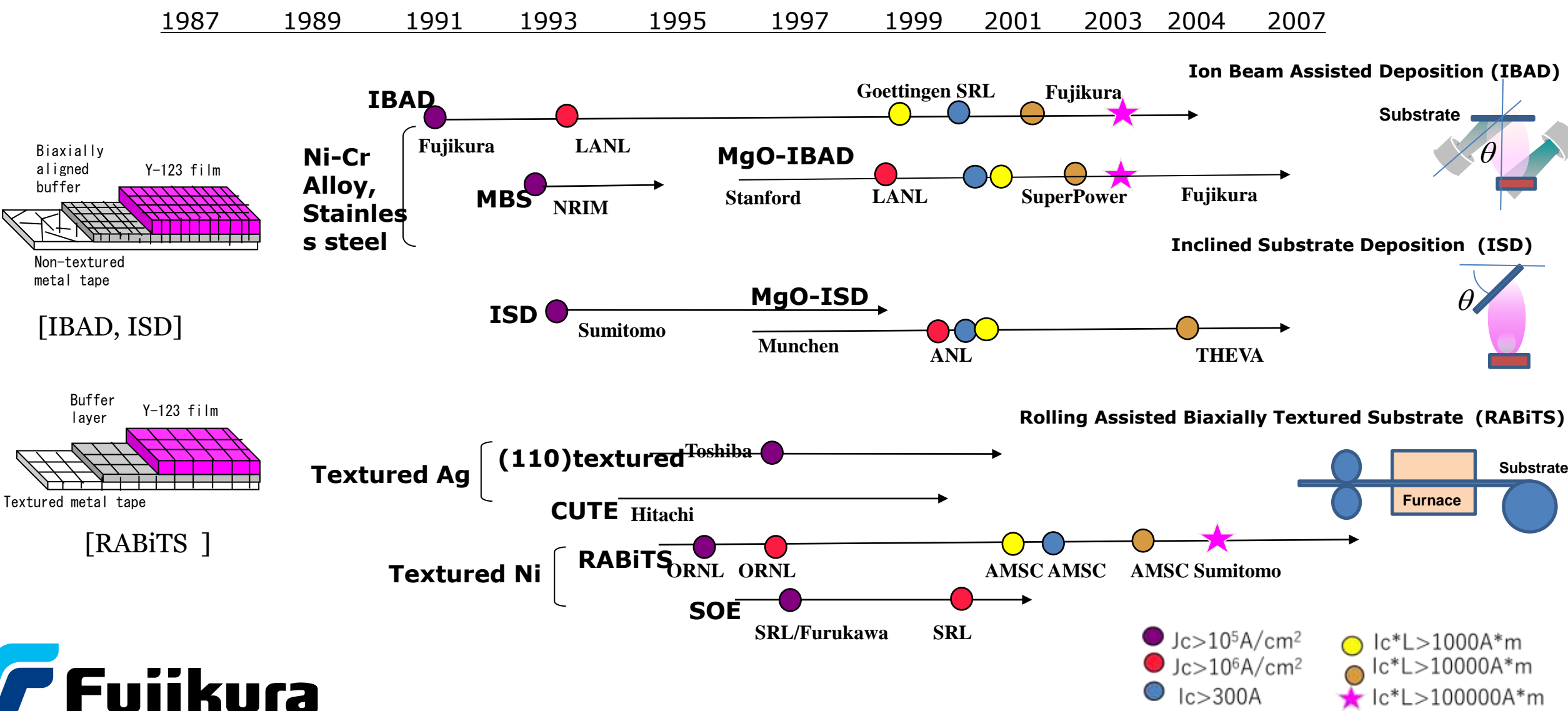


PLD 4 MOCVD 5 RCE 2 MOD 4

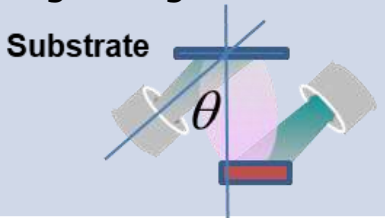
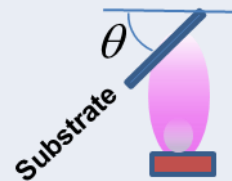

**Mass Production Started mainly by PLD
IBAD textured substrates used by almost all
manufacturers**

Development of biaxially textured substrate for C.C.

□ Many Process proposed ... **MgO-IBAD/ISD, RABiTS survived**



Advantage of IBAD process

Process	Substrate	In plane texture formation	Orientation sharpness	Surface flatness	Tensile strength
IBAD (<u>I</u> on <u>B</u> eam <u>A</u> ssisted <u>D</u> eposition)	Non-textured alloy (Ni-Cr, Fe-Cr etc.)	Ion beam modification during film growth 	⊙ Improved by collimated ion channeling	⊙	⊙ Improved by High-Jc & film smoothness
ISD (<u>I</u> nclined <u>S</u> ubstrate <u>D</u> eposition)	Non-textured alloy (Ni-Cr, Fe-Cr etc.)	Anisotropic growth with inclined evaporation flux 	○	△ Deteriorated by shadowed grain growth	○
RABiTS (<u>R</u> olling <u>A</u> ssisted <u>B</u> iaxially <u>T</u> extured <u>S</u> ubstrates)	Textured metal (Ni, Ni-W)	Rolled, recrystallized texture of metals 	○	○ Deteriorated by grooving of metal grains	△ Deteriorated by lower Young modulus

General cost evaluation of REBCO film production process

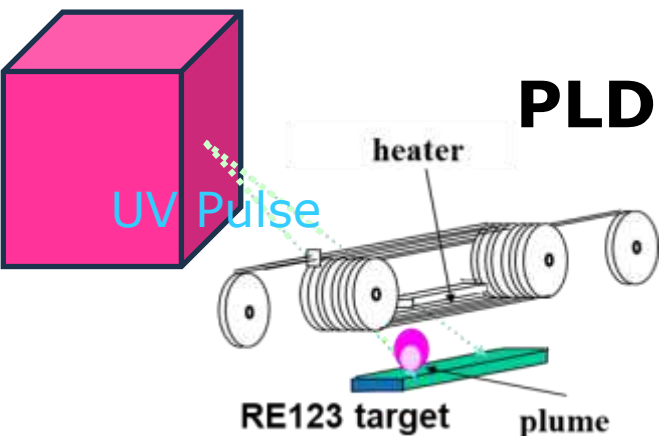
- PLD system has costly Excimer Laser, but **the equipment cost** shrinks by **High Throughput, Operation Lifetime, and High Production Yield.**

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

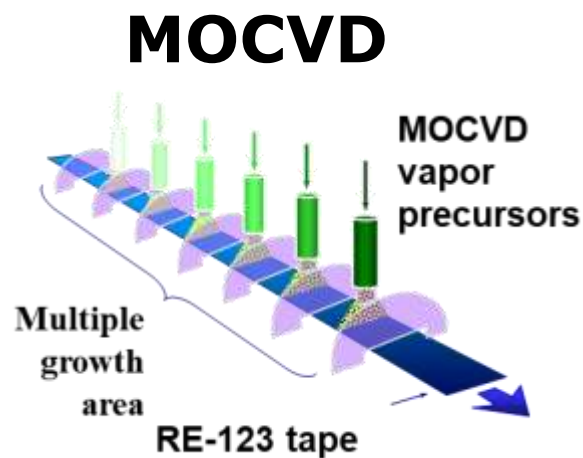
- ◆ Growth rate
- ◆ Laser power
- ◆ Laser lifetime

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

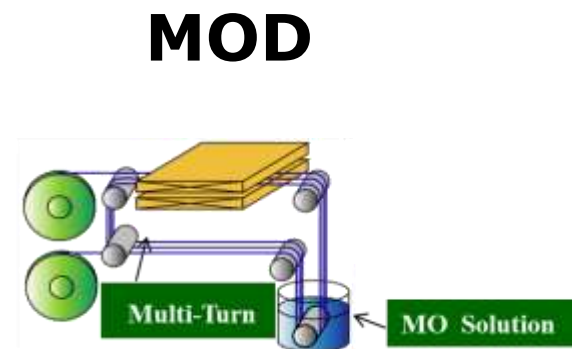
Industrial Excimer Laser



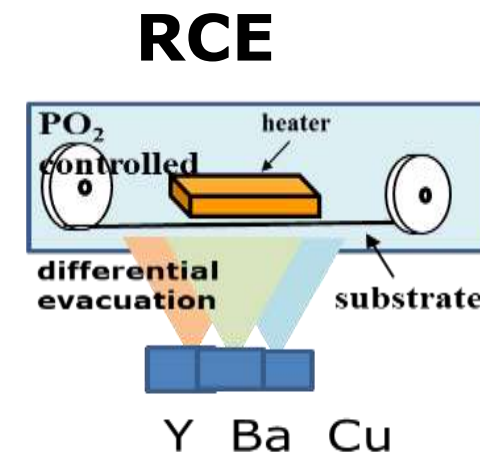
PLD



MOCVD

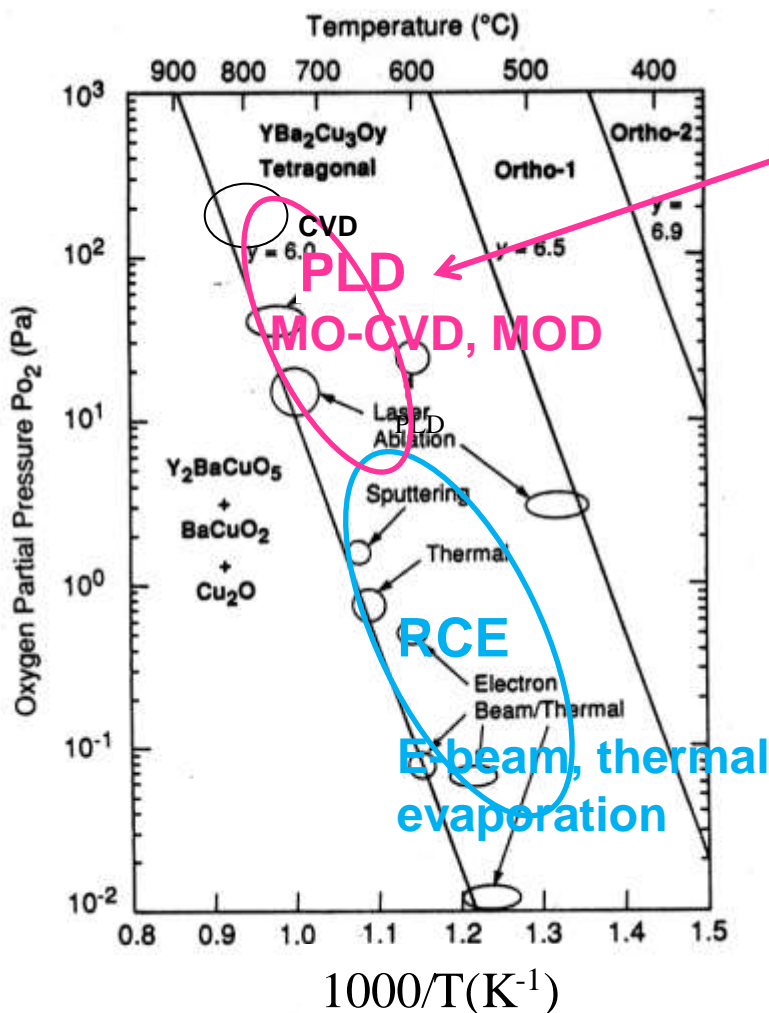


MOD



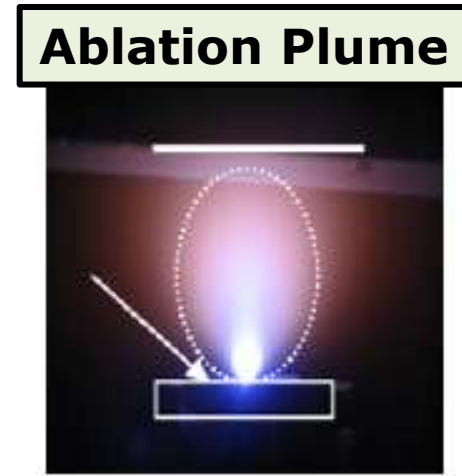
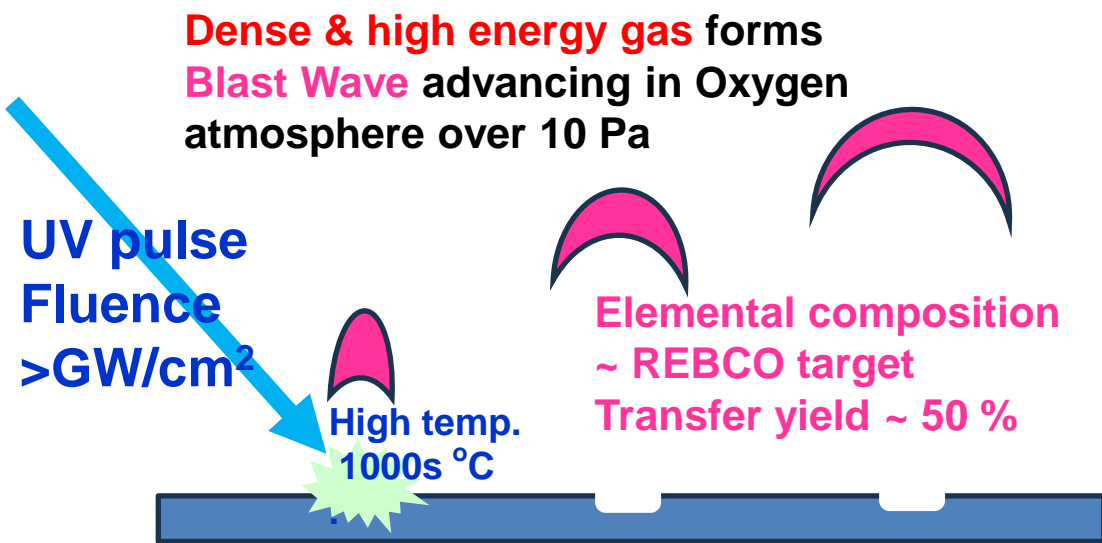
RCE

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**



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

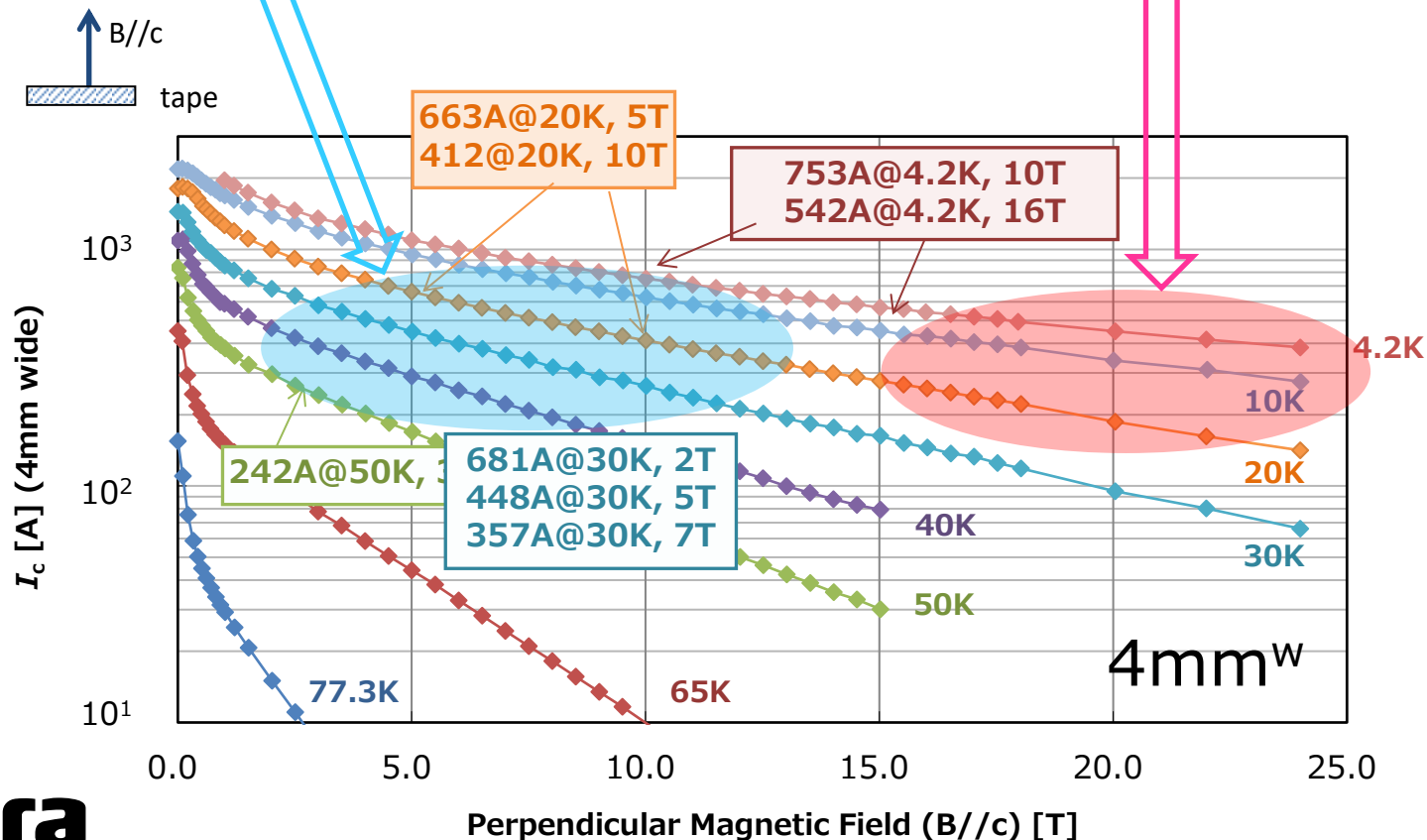
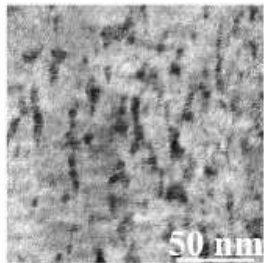
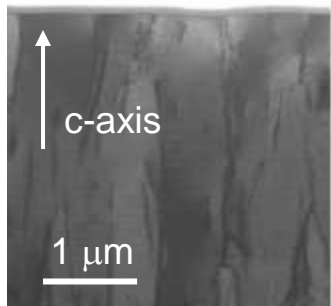
A.V. Bulgakov, N.M. Bulgakova, "Dynamics of laser-induced plume expansion into an ambient gas during film deposition", J. Phys. D: Appl. Phys. 28 (1995) 1710-1718.

- ◆ **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

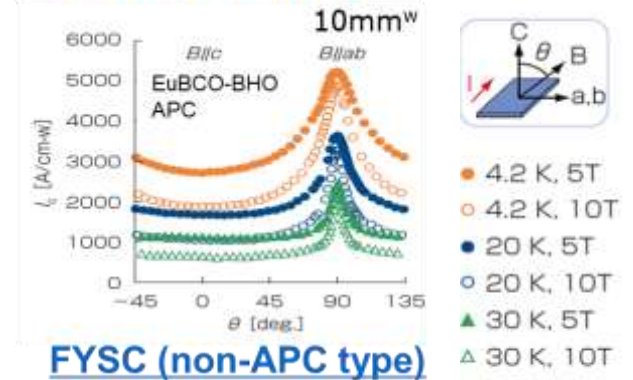
- ◆ **PLD: Vapor growth process with**
 - ◆ Favorable condition of cation composition & oxygen-temperature diagram
 - ◆ Dense & supersaturated gas with favorable particle energy for adatom mobility

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

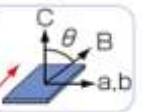
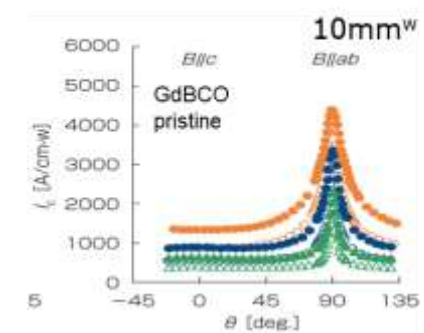


I_c -B- θ in lower field ~ 10T

FESC (APC type)

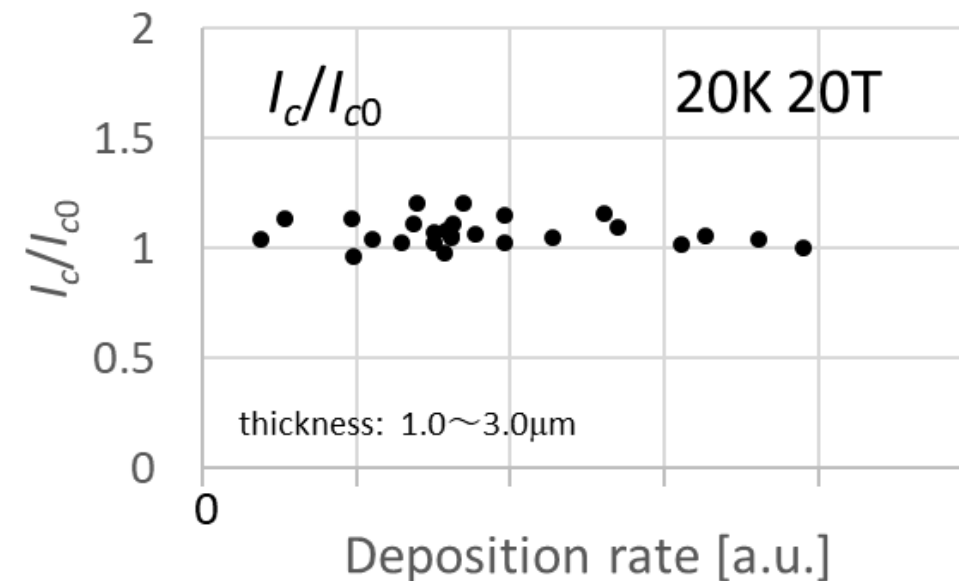
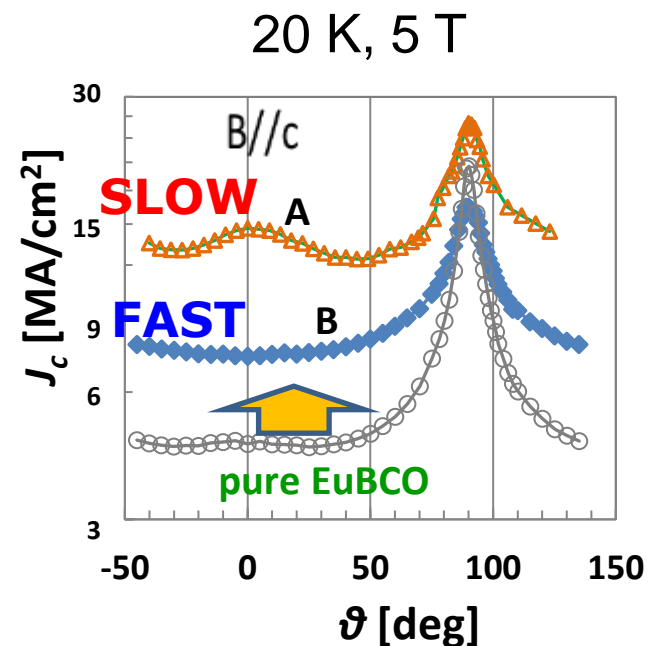
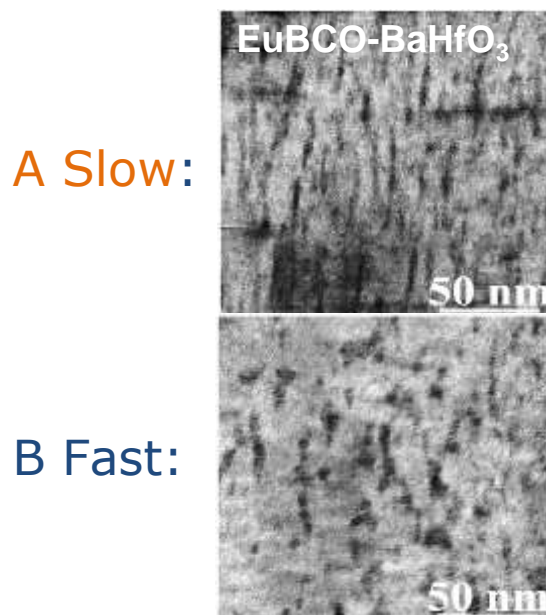


FYSC (non-APC type)



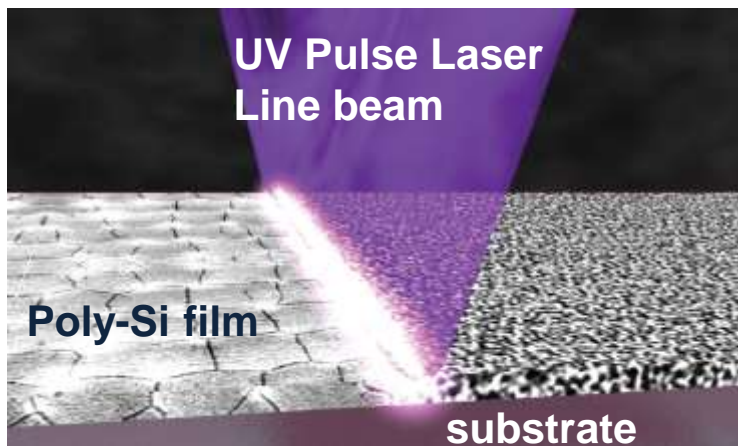
Why fast? Growth rate dependence for I_c properties

- ◆ 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

Development of high-powered excimer laser for FPD industry



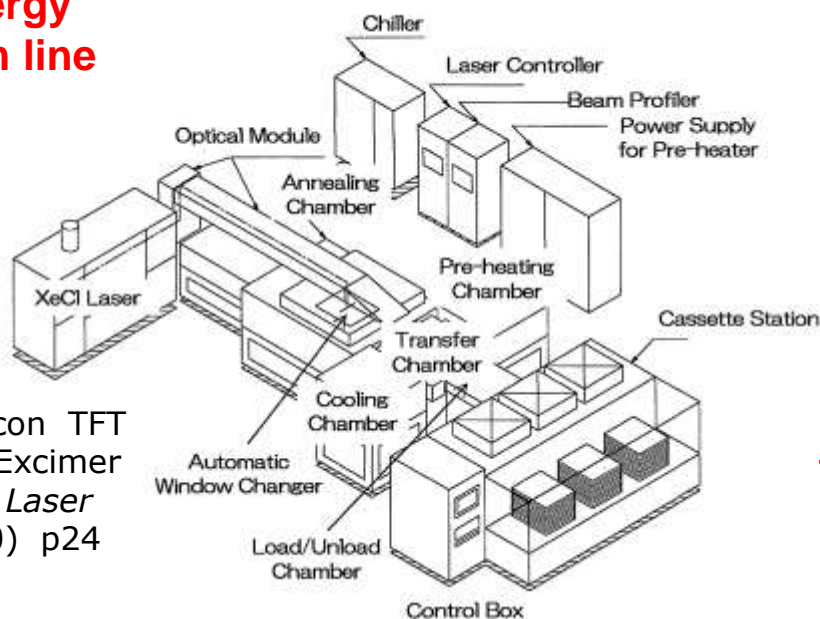
Industrial excimer laser with **large pulse energy (2000 ~)** widely Introduced for poly-Si TFT annealing of FPD industry

Amorphous Si film

➔ quite suitable to High rate PLD processing

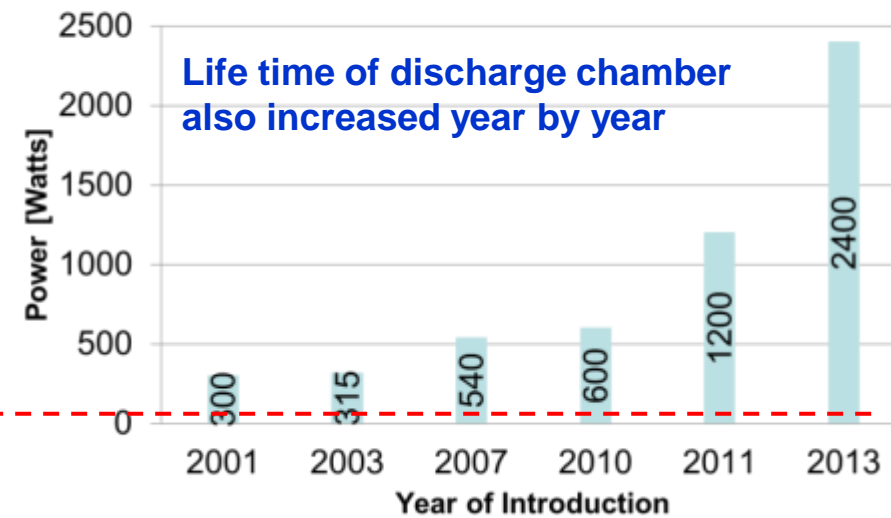
<https://www.coherent.com/ja/microelectronics-micromachining/displays>

Large pulse energy required to form line shaped beam



Laboratory use Excimer laser : 100-200w

Development of Maximum Laser Power of High Pulse Energy XeCl Excimer Laser



Life time of discharge chamber also increased year by year

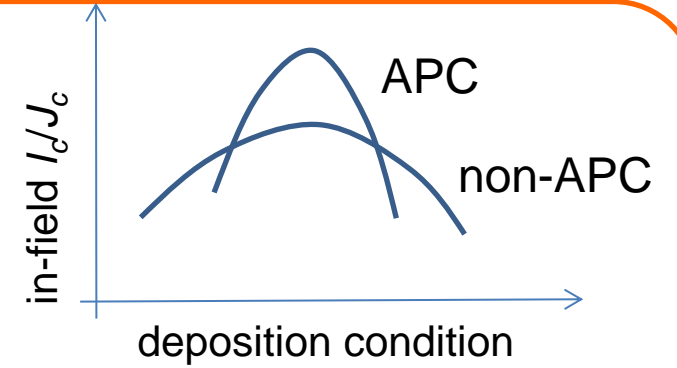
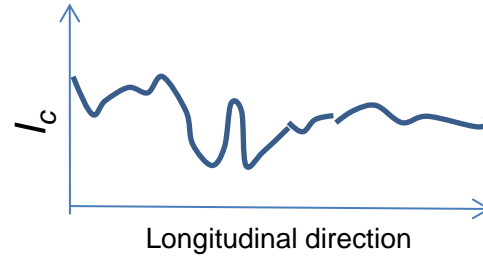
courtesy of Coherent Inc.

Shida et al: "Poly-Silicon TFT Annealing with XeCl Excimer Laser," *The Review of Laser engineering* **28**, (2000) p24 (in Japanese)

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**

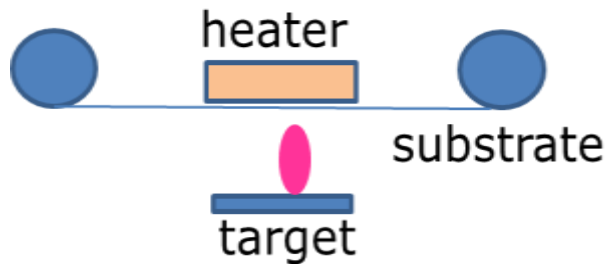
Window width $Y < Gd < Eu$
APC < non-APC

Heater block system

Single-lane PLD

Used 1990s-1999

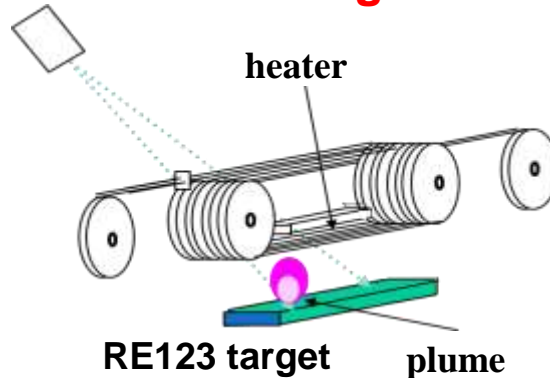
Short piece ~ 10s m long



Multi-lane / beam scanned

Used 2000-2008

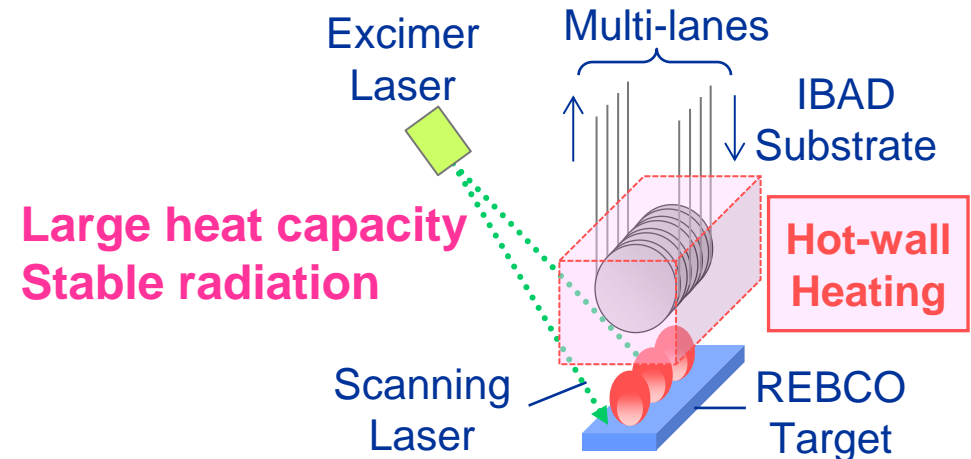
~100s m long



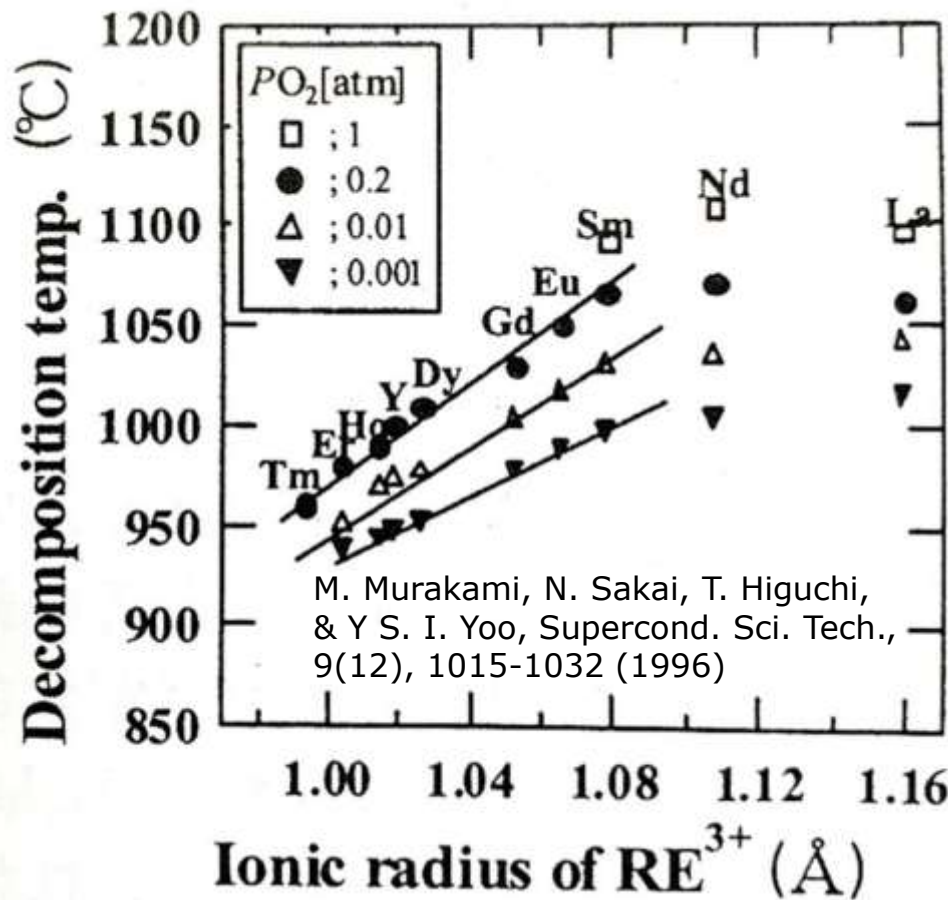
Hot-wall PLD system

Initial set up 2003-2008
 reformed 2016-2018

>1 km long

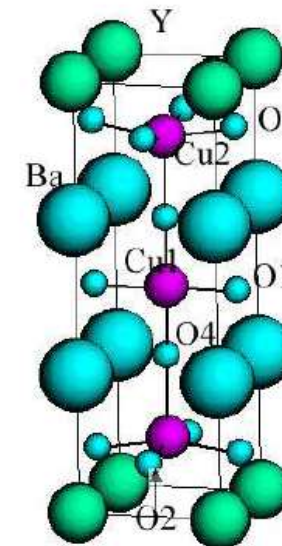


RE elemental dependent growth/lattice stability for REBCO



Dependence of oxygen partial pressure on ion radius and peritectic decomposition temperature (T_p) of various RE123

- ◆ The bigger the RE^{3+} radius approaching to Ba^{2+} radius...
- ◆ (1) The higher decomposition temperature with increased thermodynamic stability by the less internal strains etc.
- ◆ (2) The less substitution energy between RE^{3+} / Ba^{2+} degrade superconductivity
- ◆ Process window width determined by the trade-off of (1) and (2)
- ◆ around Gd, Eu were favorable



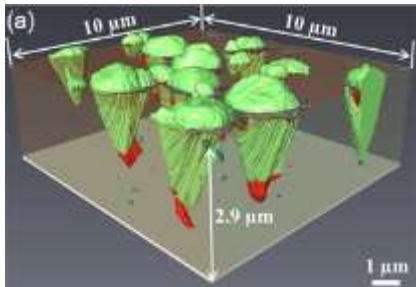
H. Su, D. O. Welch, and W. Wong-Ng, Phys. Rev. B 70, 054517 (2004)

Crystal	YBCO	GdBCO	EuBCO	SmBCO
$E_{Schottky}$ (eV)	4.22	4.21	4.20	4.16
$T_{melting}$ (°K)	1250	1290	1300	1325

RE elemental dependent growth stability for BMO-REBCO

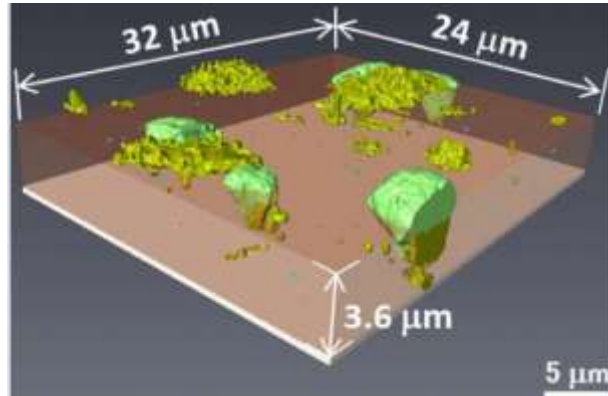
FIB-SEM 3D observation on misoriented grains (mainly a-axis aligned normal) for thick BMO-REBCO films

BaHfO-GdBaCuO



<https://www.jfcc.or.jp/result/16r33.html>

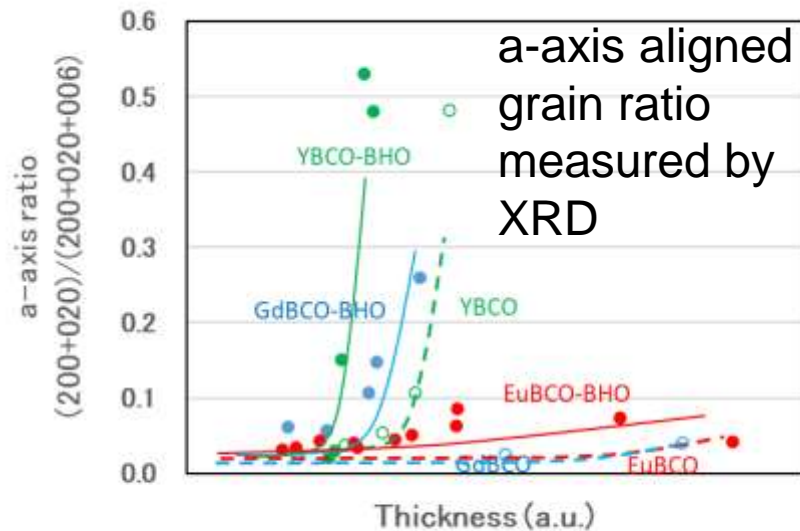
BaHfO-EuBaCuO



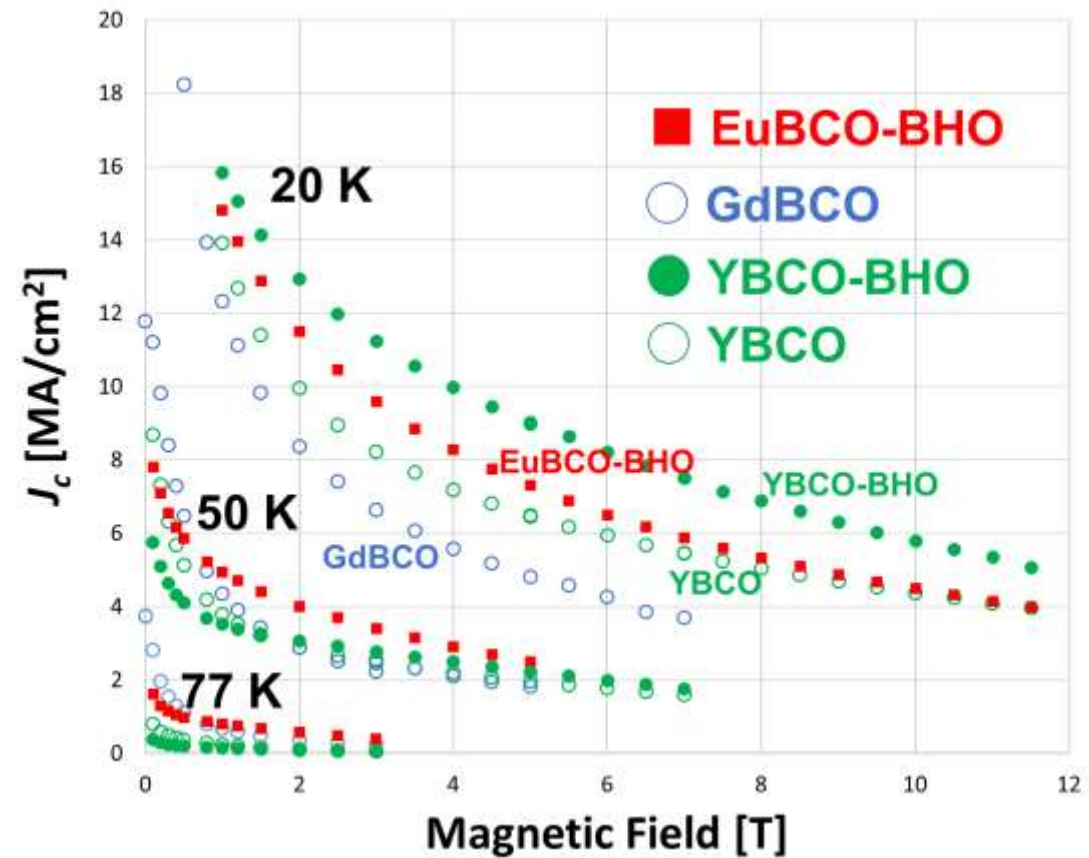
D. Yokoe et al., Supercond.Sci.Technol. **33** (2020) 024002
T. Yoshida et al., Physica C **504** (2014) 42

Oriented growth stability

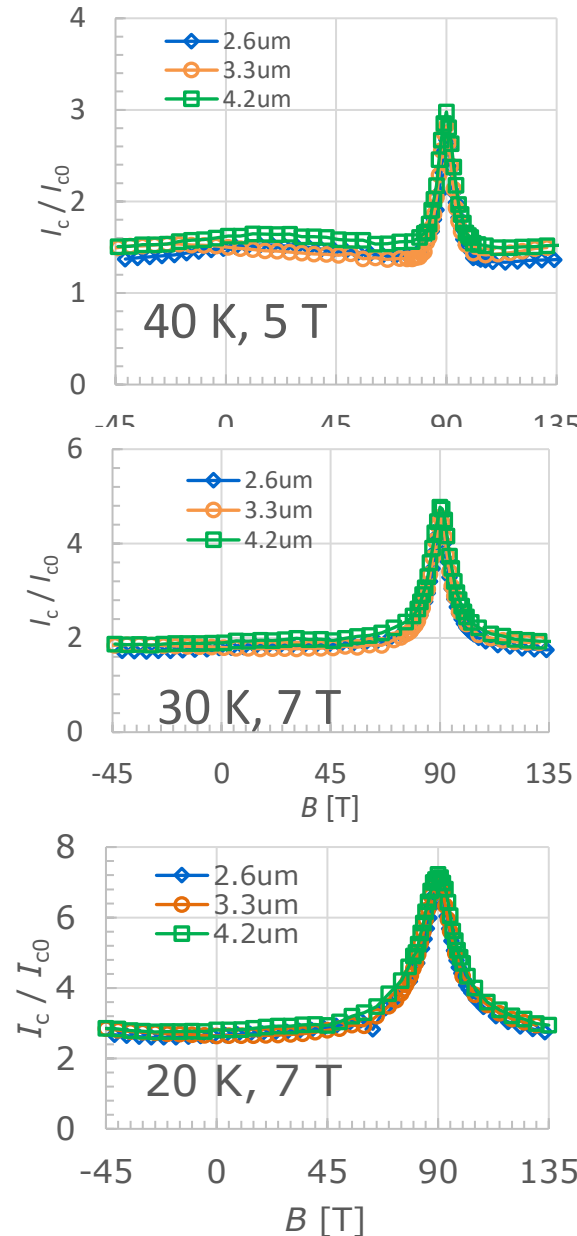
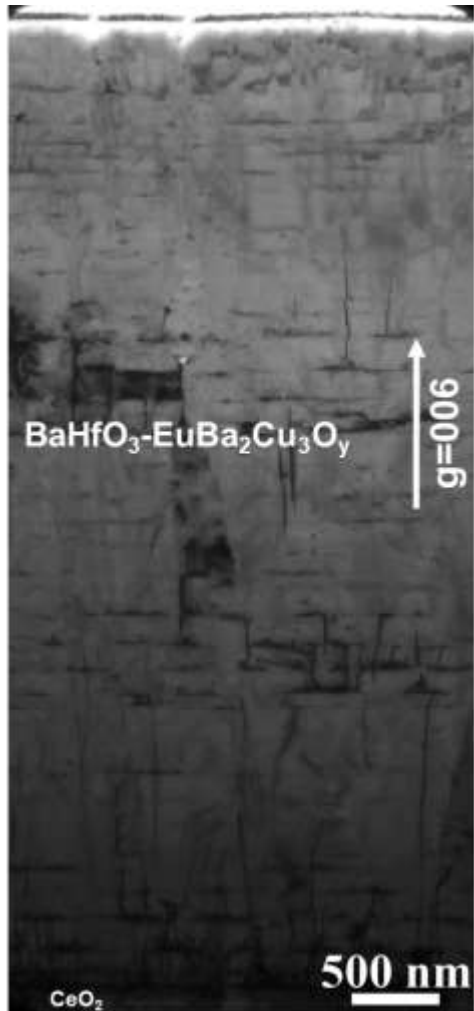
EuBCO
>GdBCO
>YBCO



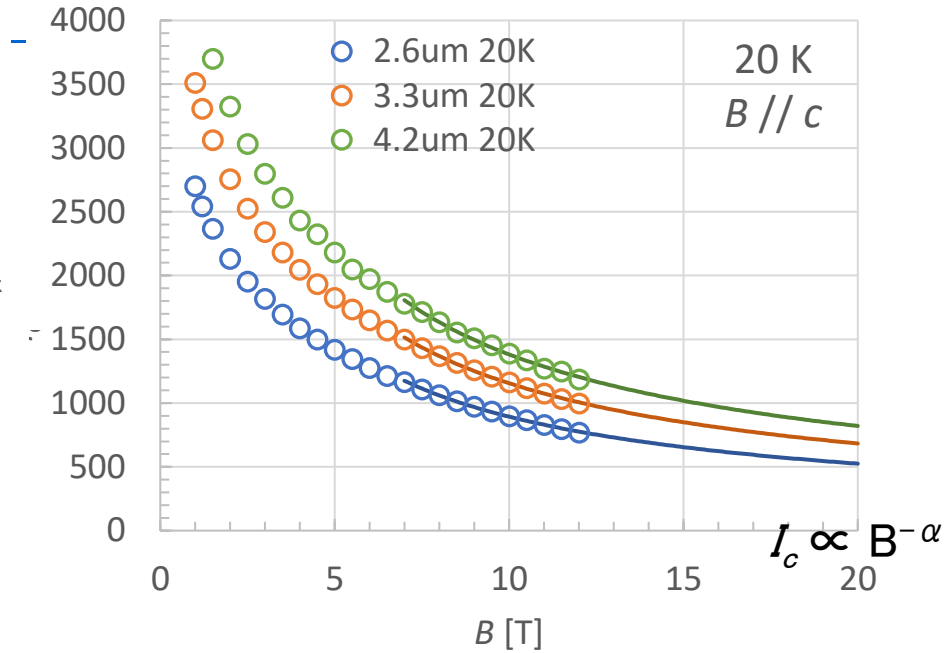
Typical J_c -B characteristics for BHO-EuBCO and pristine GdBCO, YBCO films



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



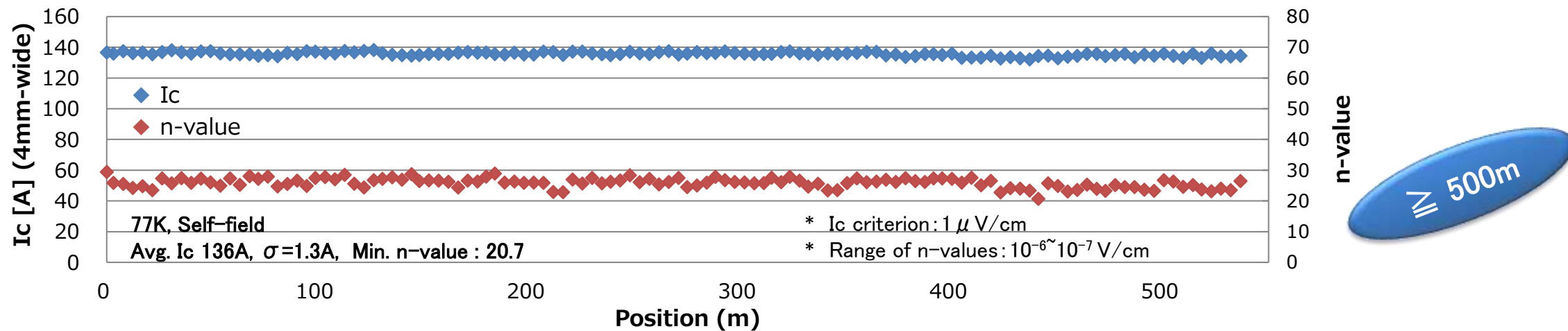
Almost the same I_c - B - θ shape up to 4.2 μm thick



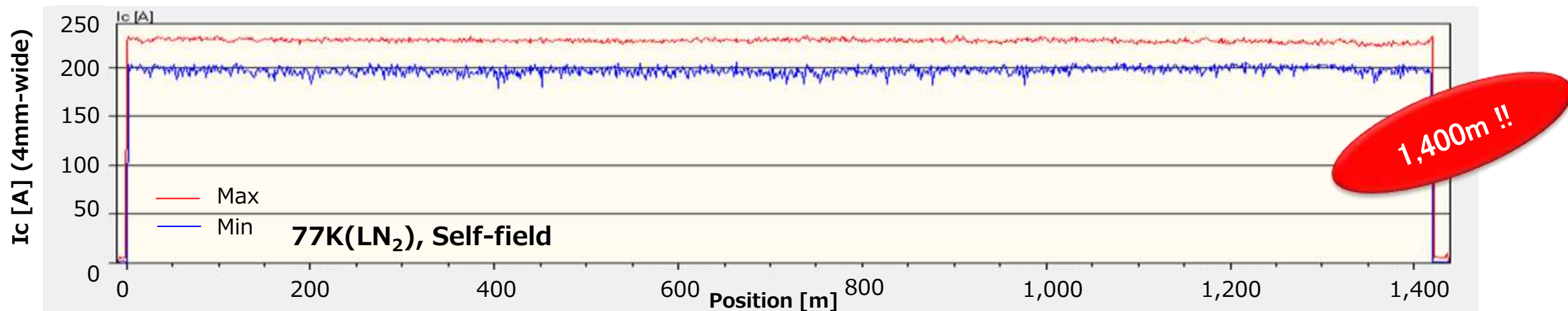
$J_e = 595 \text{ A/mm}^2$ ($I_c = 357 \text{ A}$, 4mm wide)
@30K, 7T
obtained with 2.5 μm thick
at high throughput

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

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



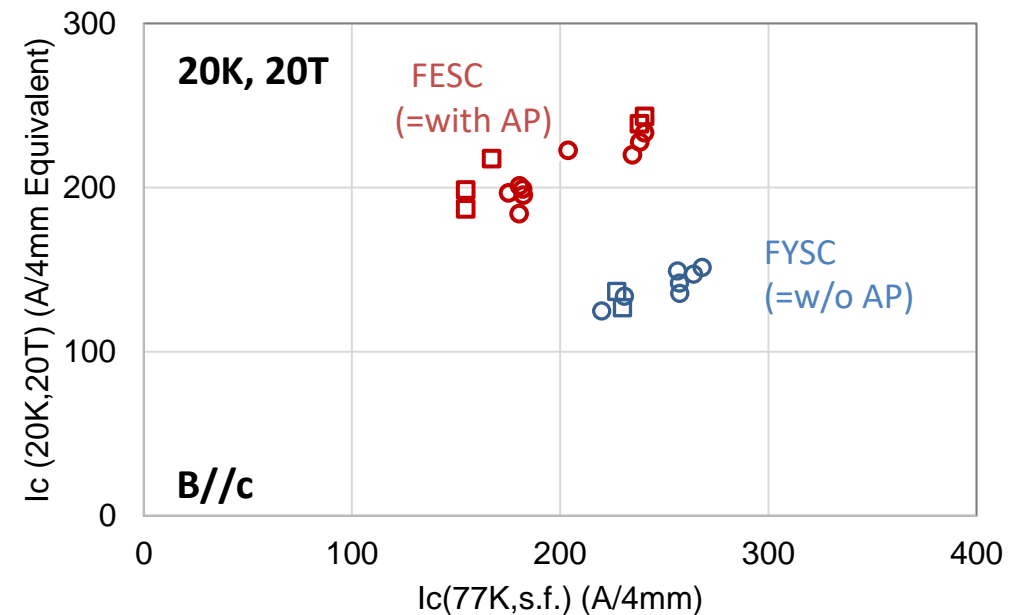
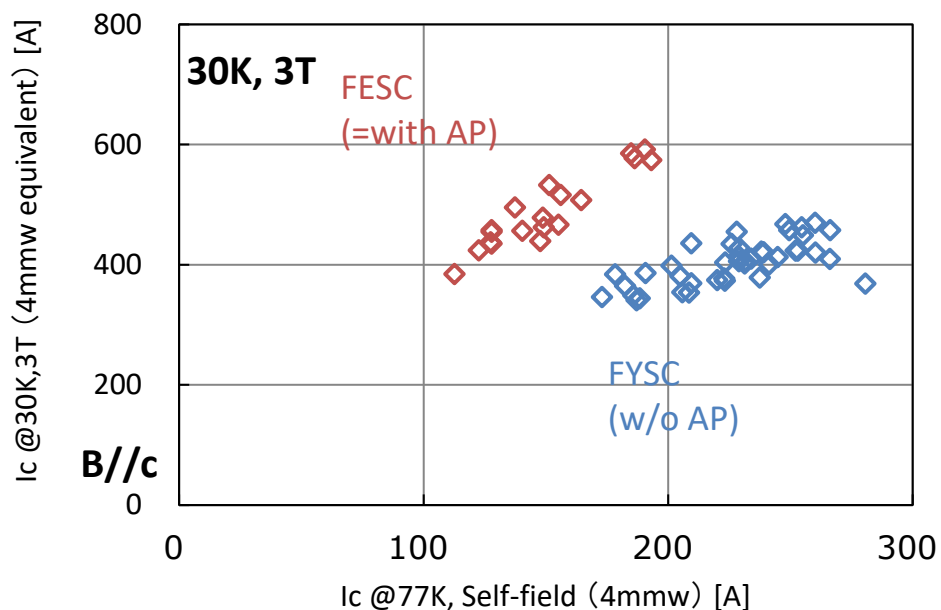
- Magnetic measurement @Tapestar™ (4mm-wide with APC / FESC-SCH04)



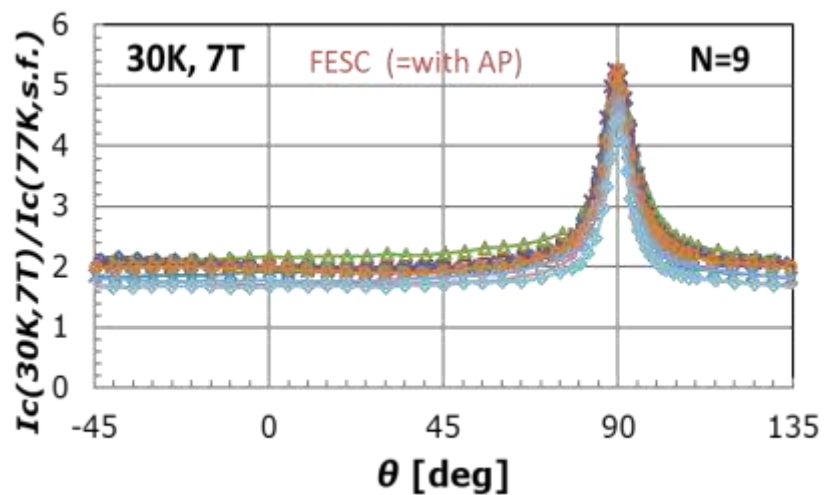
quite uniform I_c with artificial pinning tape and $\sim 1,400$ m are obtained

Lot-to-lot in-field I_c distribution of 4 mm^w wire

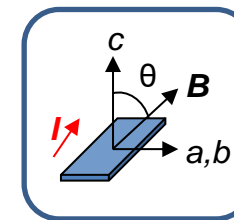
■ rot-to-rot variation of in-field I_c / I_c (77 K, s.f.)



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



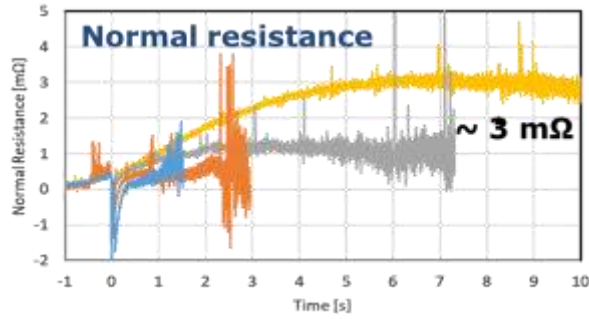
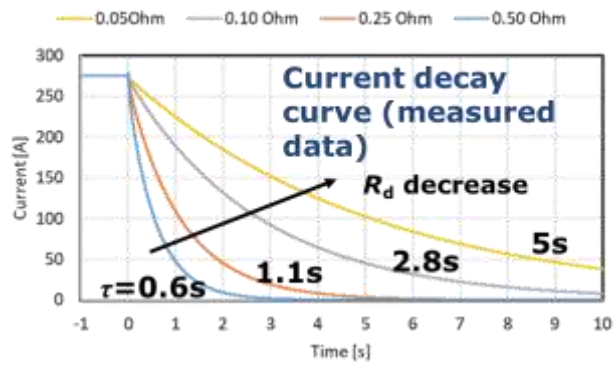
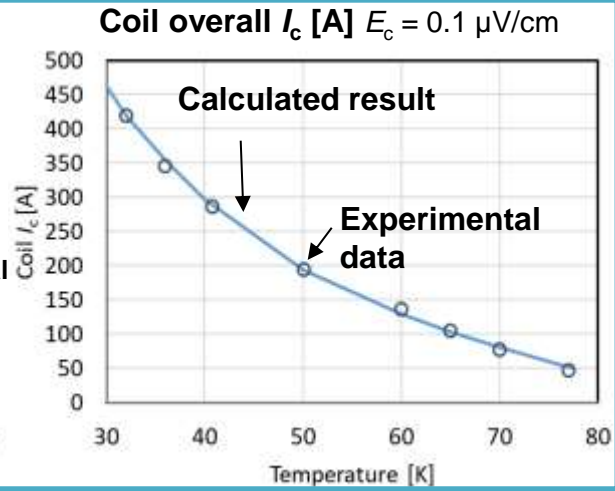
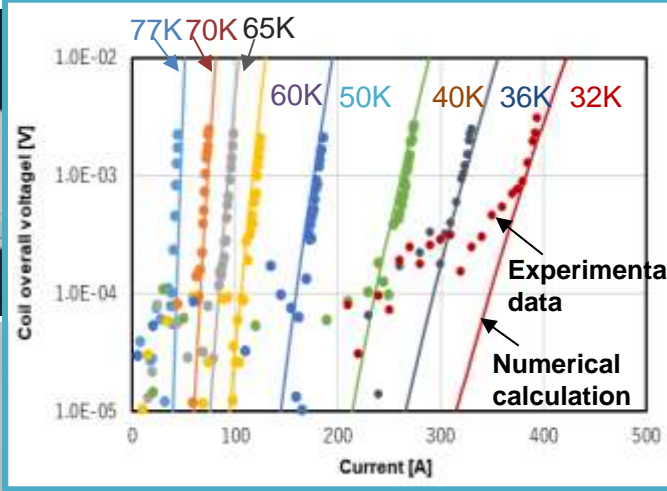
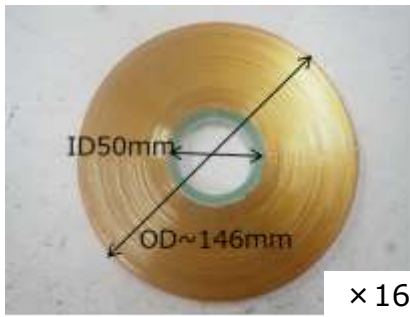
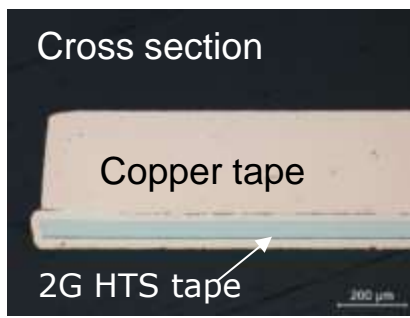
◇○ measured at Fujikura, and exploited values with I_c
□ in-field I_c measured at Tohoku university



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

Parameters	REBCO tape
Substrate thick.	50 μm
Copper thick.	20 $\mu\text{m} \times 2$ (plates) + 300 μm
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%

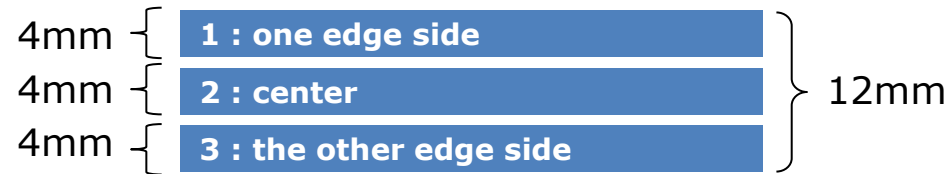


- **Good agreement** between experimental and calculated results for coil I_c
- **No degradation** observed after quench test.

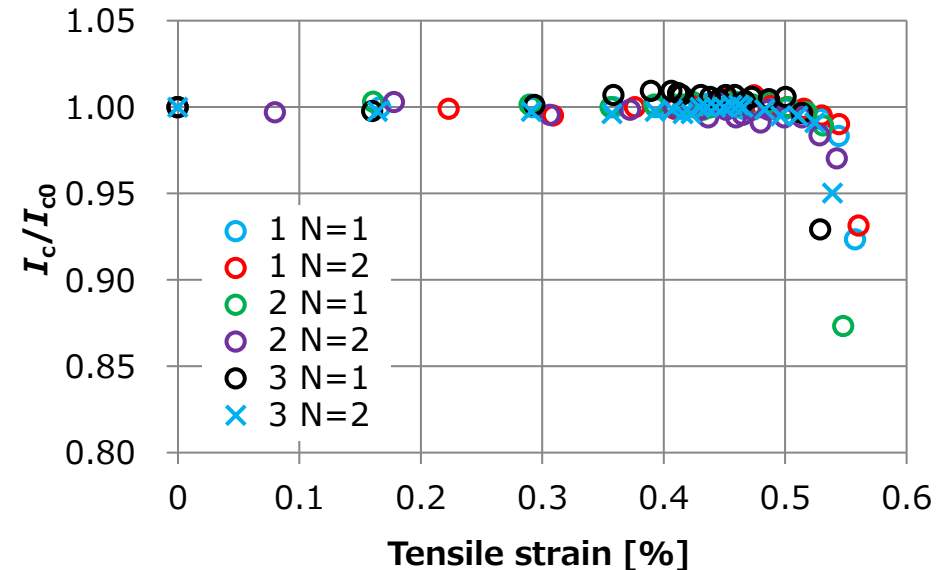
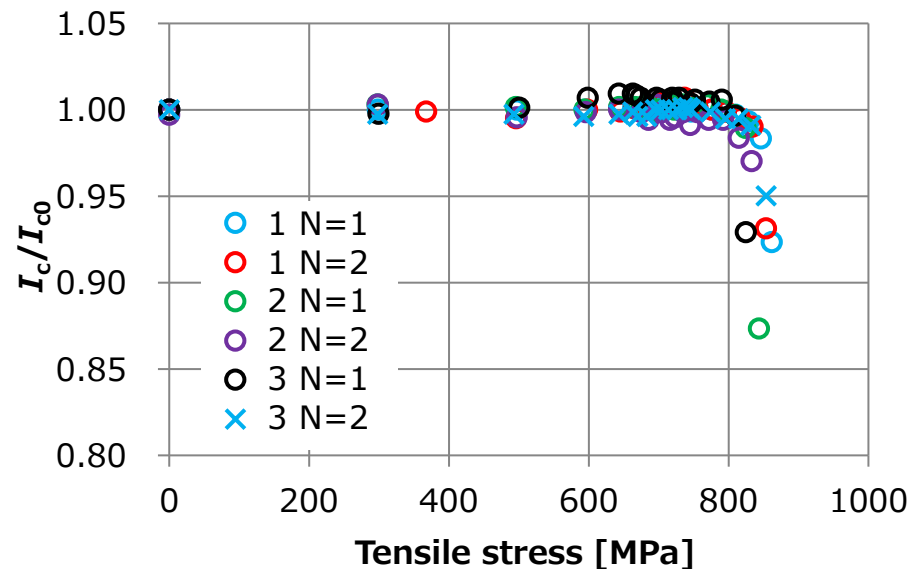
Tensile Properties Evaluation of Divided 4 mm-wide

- Tensile properties of 3 parts of 4 mm-wide tapes divided from 12 mm-wide tape in LN₂

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



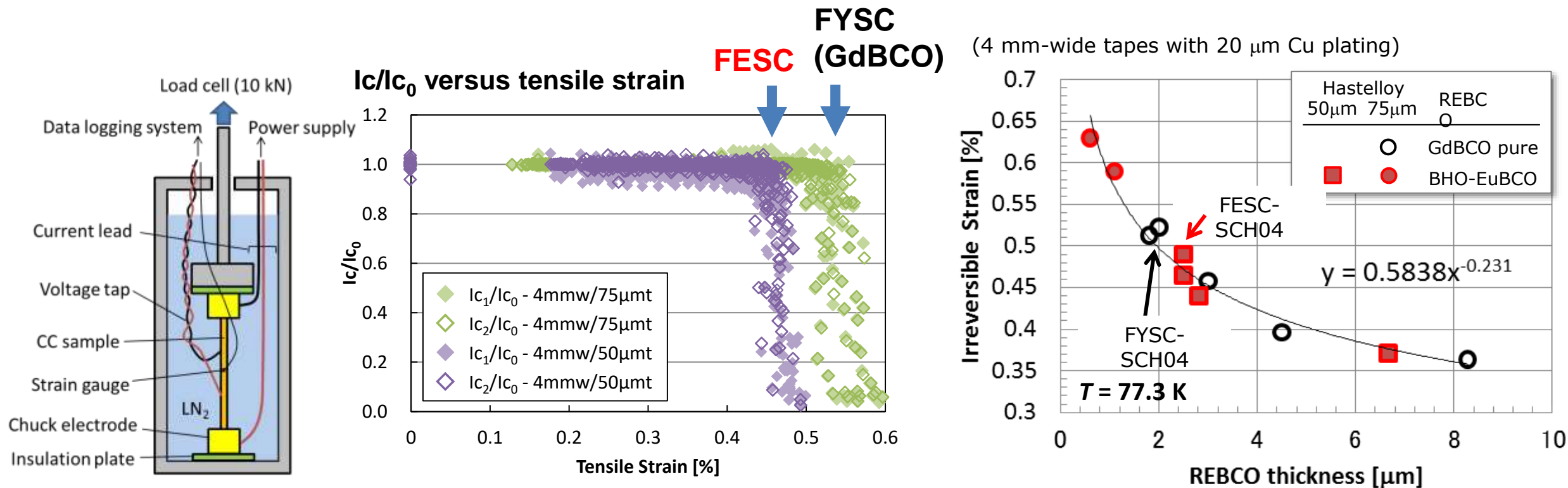
* Reversible I_c is defined at $I_c/I_{c0} > 0.99$. (I_{c0} : Initial)



- 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**.

Tensile strength: REBCO thickness dependance

High quality, high J_c , thin REBCO film is favorable for stronger tensile strength



- The REBCO thickness dependence of ϵ_{irr} is due to the volume effect, which is general phenomena in ceramics.

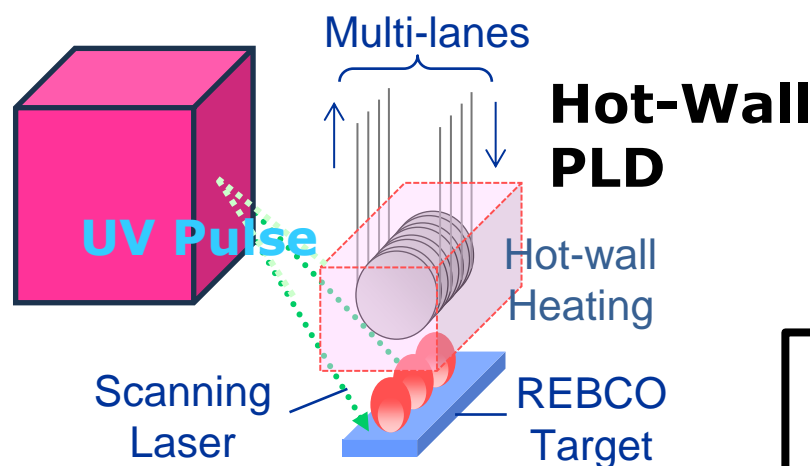
$$\bar{\epsilon} = \epsilon_0 \Gamma \left(1 + \frac{1}{m} \right) \left(\frac{V}{V_0} \right)^{-\frac{1}{m}} \propto V^{-\frac{1}{m}} \propto (\text{REBCO thickness})^{-\frac{1}{m}}$$

General evaluation of IBAD/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

◆ 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

◆ Tensile strength

- ◆ High quity Thinner film

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

Typical Specifications of 2G HTS Tape at Fujikura

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

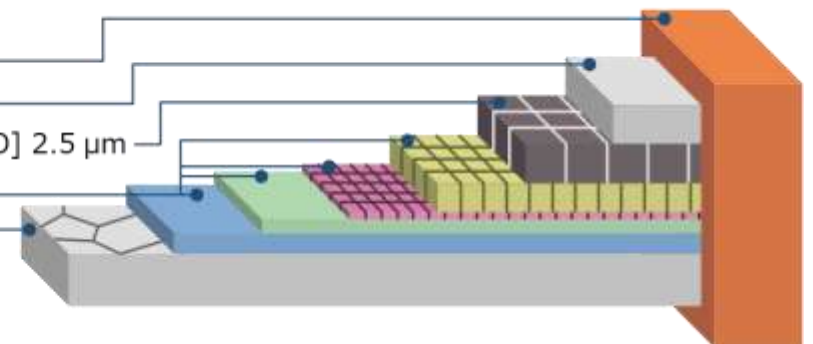
*5 If requested, an option **customizing copper plating thickness is also available**. (e.g., 5 μm , 10 μm or 40 μm)

- 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>

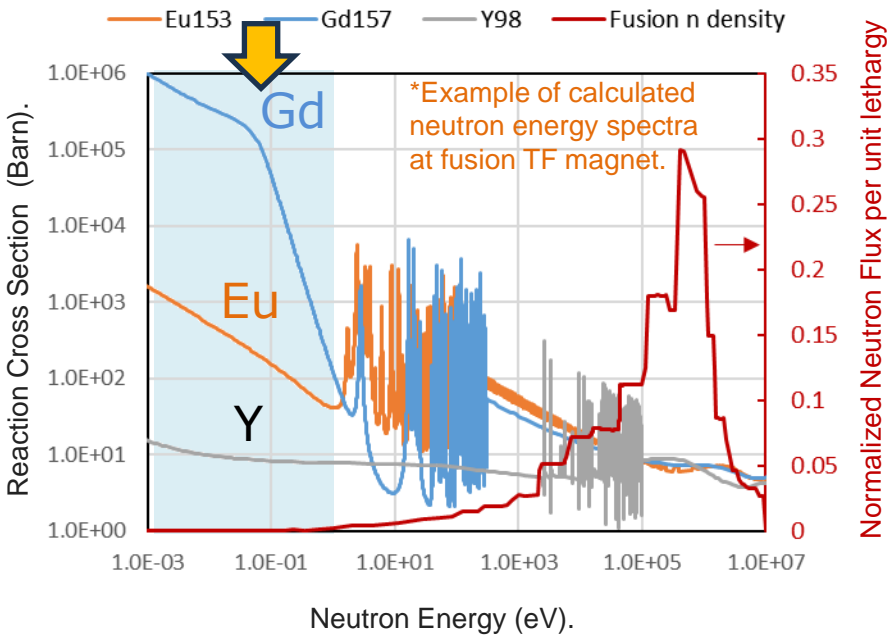
Stabilizer [Cu plating] 20 μm
 Protection layer [Ag] 2 μm
 Superconducting Layer [GdBCO] 2 μm / [EuBCO+BHO] 2.5 μm
 Buffer layer [MgO, etc.] 0.7 μm
 Substrate [Hastelloy®] 75 / 50 μm



RE elemental dependence of neutron radiation damage

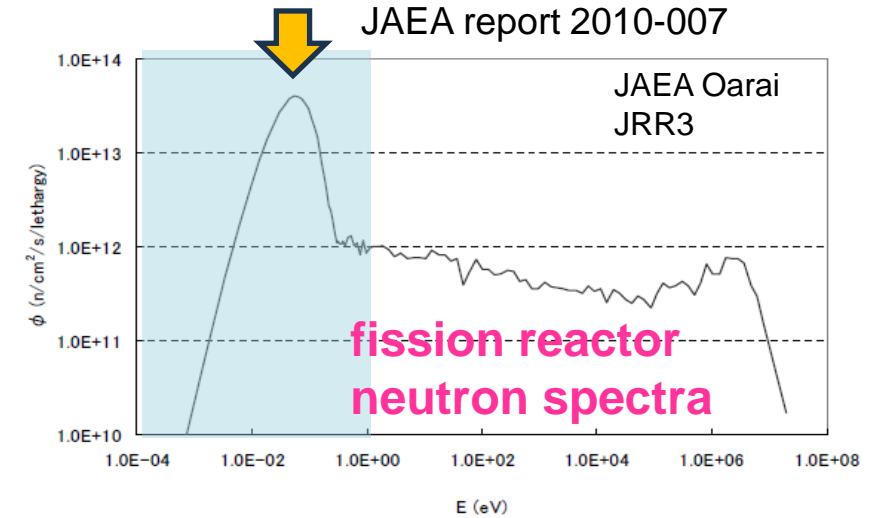
Large cross section of neutron capture for Gd/Eu isotopes ~ 1 eV anticipated to damage lattice structures by gamma decay with large recoil energy

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

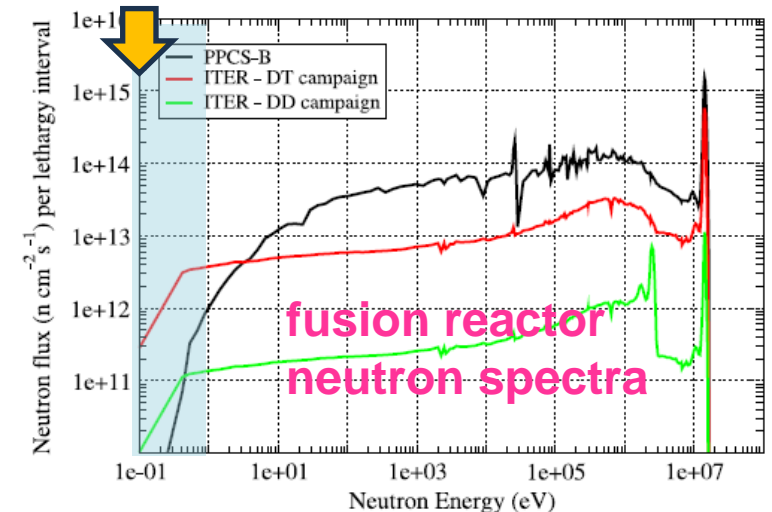


Fission reactor environment often used for irradiation tests has big peak in neutron spectra below 1 eV come from cooling water

where fusion ones estimated by calculation have quite low flux there



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



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.

Summary

- **IBAD/PLD: most reliable process to answer current business demand**
 - strong mechanical strength, high in-field J_c ,
 - with good uniformity and production reliability.

- **The advantage of Hot-wall PLD process covers laser cost well under mass production so far, with large process window**
 - **Reproducibility:** controllable near best phase point with simple parameters
 - **High in-field I_c :** small and dispersed pinning centers by vapor deposition
 - **High growth rate:** high transfer yield and adequate adatom energy
 - **Uniformity:** improved by Hot-wall architecture
 - **RE dependent growth stability:** Gd, Eu desirable from the manufacturer standpoint

- **Neutron irradiation durability of REBCO wires line-up**
 - Depends on **Thermal neutron spectra** below 1eV for compact fusion magnet

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

END

Thank you for attention