Experimental studies of Nb material at KEK (FY2018 – FY2021 and to FY2022)

IDT WG2 SRF-Group meeting

18 Jan. 2021

T. Saeki (KEK)

Objective of our studies at KEK

- The studies are aiming at the cost-effective Nb materials for the mass-production of ILC SRF-cavities.

- The Nb materials should promise the good performance of SRF-cavities and enough mechanical properties to pass HPGS* regulation of Japan.

*HPGS: High Pressure Gas Safety

- Focusing on:
  - * Purity/RRR (including Ta-contents)
  - * Grain-size and fabrication methods (forging, rolling, and direct-slicing etc.)
  - * Combinations to various surface treatments (standard and advanced recipes)

Definition of words in this presentation

- FG = (Fine Grain), MG= (Medium Grain), LG = (Large Grain)

  Grain size: < 0.1 mm, < 1 mm, >> 1 cm

- High-RRR = (RRR >= 300)
- Mid-RRR = (300 > RRR > 200)

- High-Ta = (Ta > 1000 ppm)
- Low-Ta = (Ta < 100 ppm)

- HPGS = High Pressure Gas Safety regulation

Overview of studies

Nb materials	Cavities	FY2018	FY2019	FY2020	FY2021	FY2022
LG Mid-RRR High-Ta		Two 9-cell cavities were fabricated	VT Eacc < 35MV/m	VT Eacc < 35MV/m		
LG High-RRR Low-Ta				Two 3-cell cavities were fabricated	VT Eacc > 35MV/m ILC spec. achieved	
LG High-RRR High-Ta				Two 3-cell cavities were fabricated	VT Eacc > 35MV/m ILC spec. achieved	
					Two 9-cell cavities are under fabrication	VT
MG High-RRR Low-Ta				Two 1-cell cavities were fabricated	VT Eacc > 35MV/m ILC spec. achieved	
						One 9-cell cavity will be fabricated

RRR and Ta-fraction studies

Overview of studies

Nb materials	Cavities	FY2018	FY2019	FY2020	FY2021	FY2022
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MG High-RRR Low-Ta				Two 1-cell cavities were fabricated	VT Eacc > 35MV/m ILC spec. achieved	
						One 9-cell cavity will be fabricated

Mid-RRR (LG-Nb, High-Ta)

300 > RRR > 200

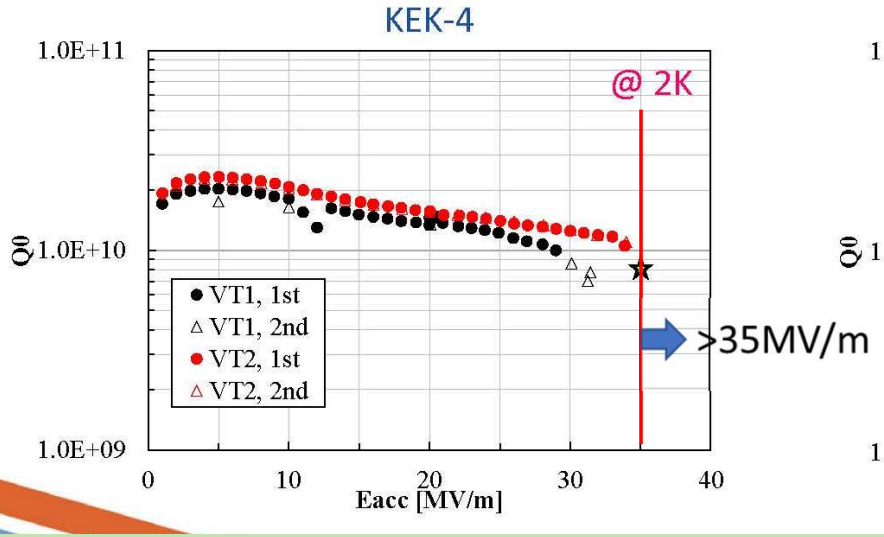
Studies from FY2018 to FY2020 → Mid-RRR (LG-Nb, High-Ta) 9-cell cavities have not reached 35 MV/m.

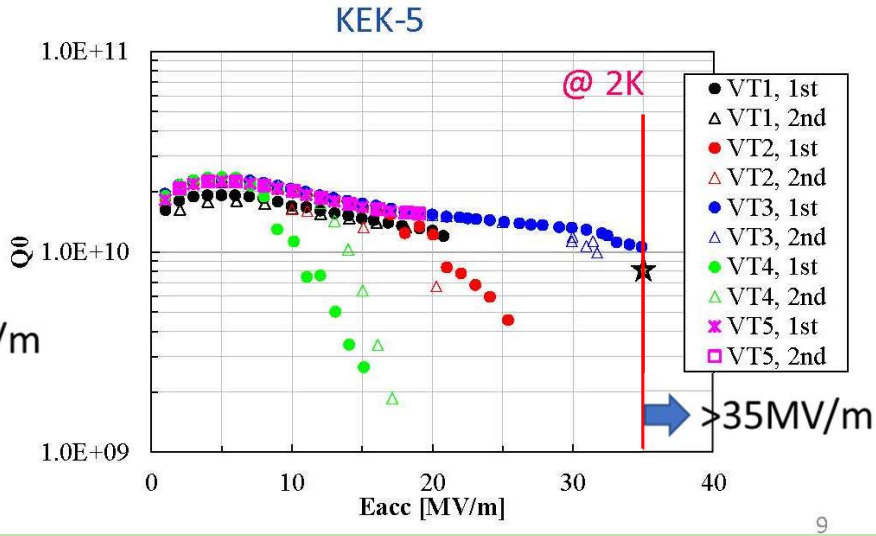
KEK-4, KEK-5

Nb Venders: C, Mid-RRR (LG-Nb, High-Ta)

Two of them have not achieved ILC spec. Eacc =34 MV/m

Slide by Dohmae 2020-Dec IHEP meeting





We have concluded that High RRR is necessary for high SRF performance.

Overview of studies

Nb materials	Cavities	FY2018	FY2019	FY2020	FY2021	FY2022
LG Mid-RRR High-Ta		Two 9-cell cavities were fabricated	VT Eacc < 35MV/m	VT Eacc < 35MV/m		
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MG High-RRR Low-Ta				Two 1-cell cavities were fabricated	VT Eacc > 35MV/m ILC spec. achieved	
						One 9-cell cavity will be fabricated

High-RRR (LG-Nb, Low-Ta)

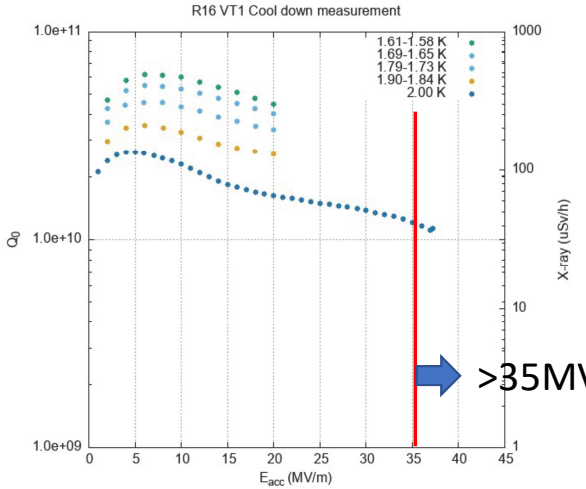
RRR > 300

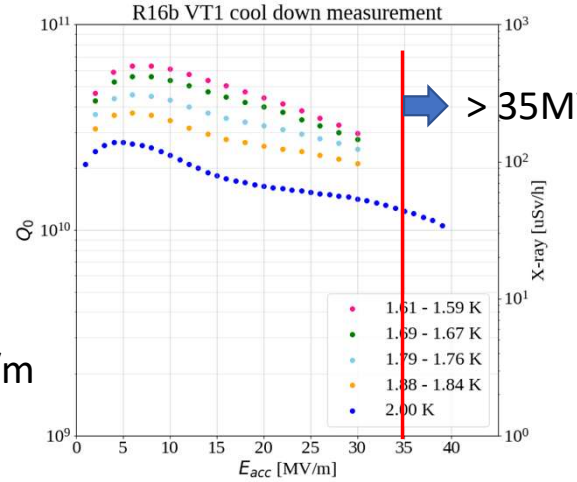
Studies from FY2020 to FY2021 (now) → High-RRR (LG-Nb, Low-Ta) 3-cell cavities have reached > 35 MV/m.

Two 3-cell cavities were fabricated in Mar. 2021.

R16, R16b

Nb Venders: A + C, High-RRR (LG-Nb, Low-Ta)





R-16: VT1 (25 Nov. 2021), Eacc = 37 MV/m (Initial Eacc = 41 MV/m).
R-16b VT1 (16 Dec. 2021), Eacc = 39 MV/m (Initial Eacc = 40 MV/m).

We have decided to focus on Nb material with RRR > 300.

Overview of studies

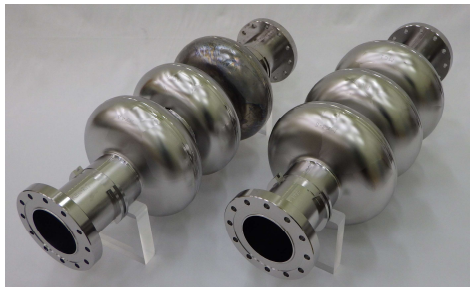
Nb materials	Cavities	FY2018	FY2019	FY2020	FY2021	FY2022
LG Mid-RRR High-Ta		Two 9-cell cavities were fabricated	VT Eacc < 35MV/m	VT Eacc < 35MV/m		
LG High-RRR Low-Ta				Two 3-cell cavities were fabricated	VT Eacc > 35MV/m ILC spec. achieved	
LG High-RRR High-Ta				Two 3-cell cavities were fabricated	VT Eacc > 35MV/m ILC spec. achieved	
					Two 9-cell cavities are under fabrication	VT
MG High-RRR Low-Ta				Two 1-cell cavities were fabricated	VT Eacc > 35MV/m ILC spec. achieved	
						One 9-cell cavity will be fabricated

High-RRR (LG-Nb, High-Ta)

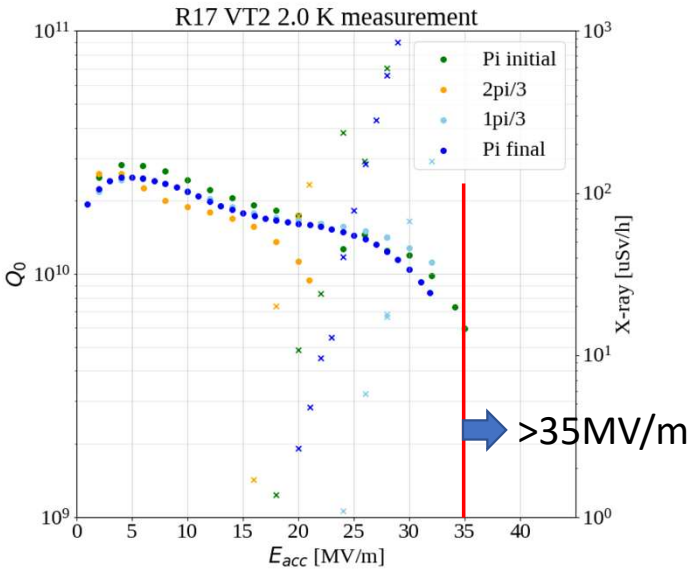
RRR > 300

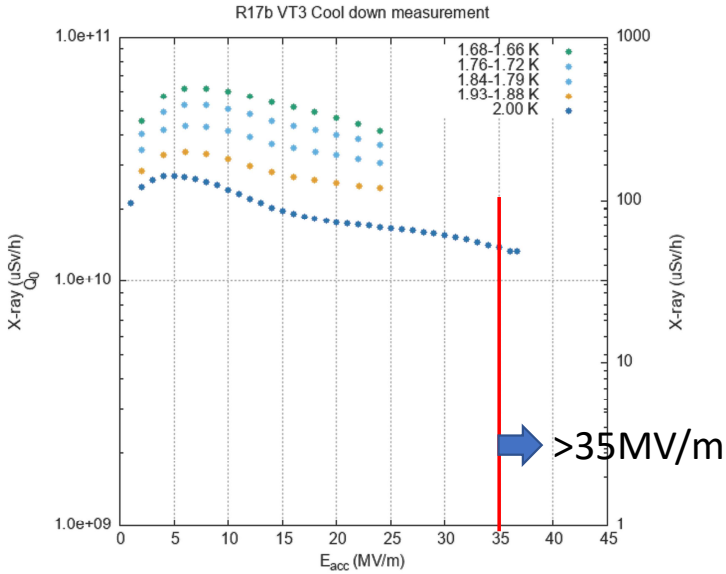
Studies from FY2020 to FY2021 (now) → High-RRR (LG-Nb, High-Ta) 3-cell cavities have reached > 35 MV/m.

Two 3-cell cavities were fabricated in Mar. 2021.



R17, R17b
Nb Venders: B + C, High-RRR (LG-Nb, High-Ta)





R-17: VT2 (09 Spt. 2021) Eacc = 35 MV/m
R-17b: VT3 (28 Spt. 2021) Eacc = 37 MV/m (Initial Eacc = 42MV/m)

Ta-fraction is not sensitive to SRF performance if RRR > 300.

Overview of studies

Nb materials	Cavities	FY2018	FY2019	FY2020	FY2021	FY2022
LG Mid-RRR High-Ta		Two 9-cell cavities were fabricated	VT Eacc < 35MV/m	VT Eacc < 35MV/m		
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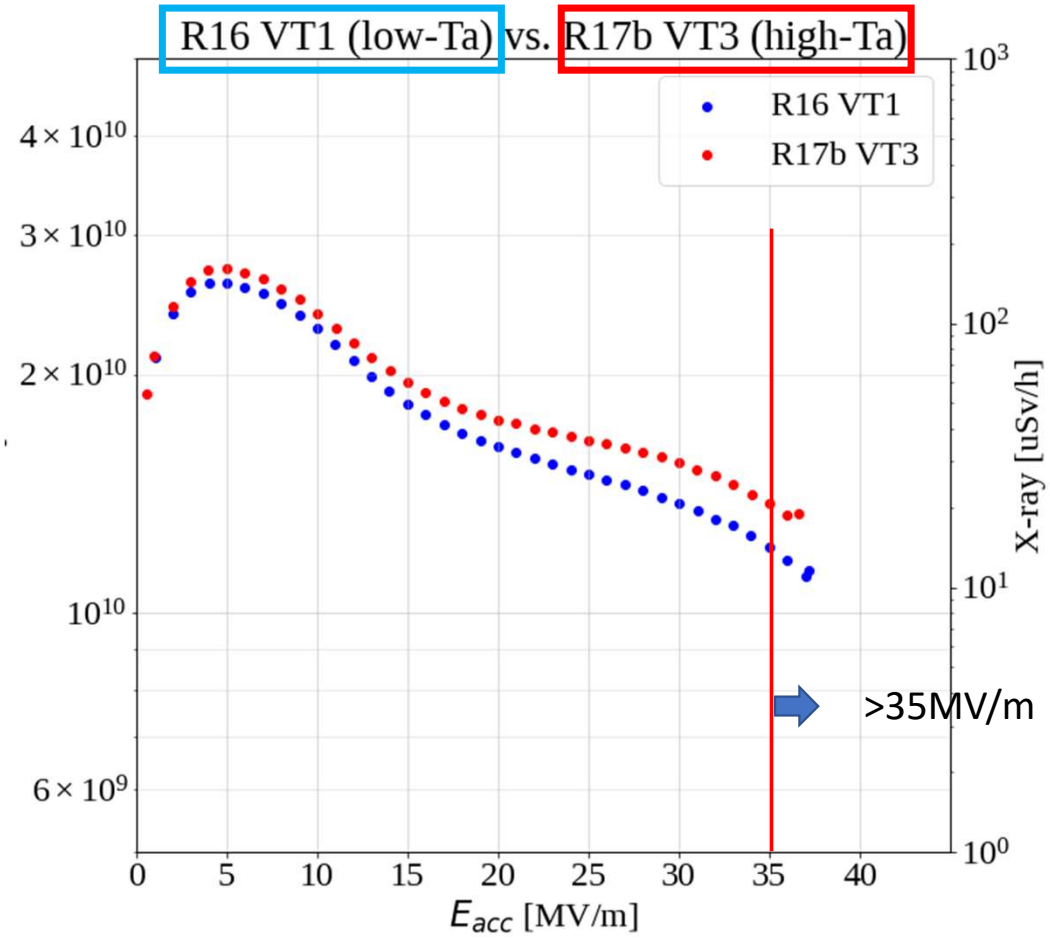
MG High-RRR Low-Ta				Two 1-cell cavities were fabricated	VT Eacc > 35MV/m ILC spec. achieved	
						One 9-cell cavity will be fabricated

Low-Ta vs. High-Ta (LG, High-RRR)



Comparison of Low-Ta vs High-Ta

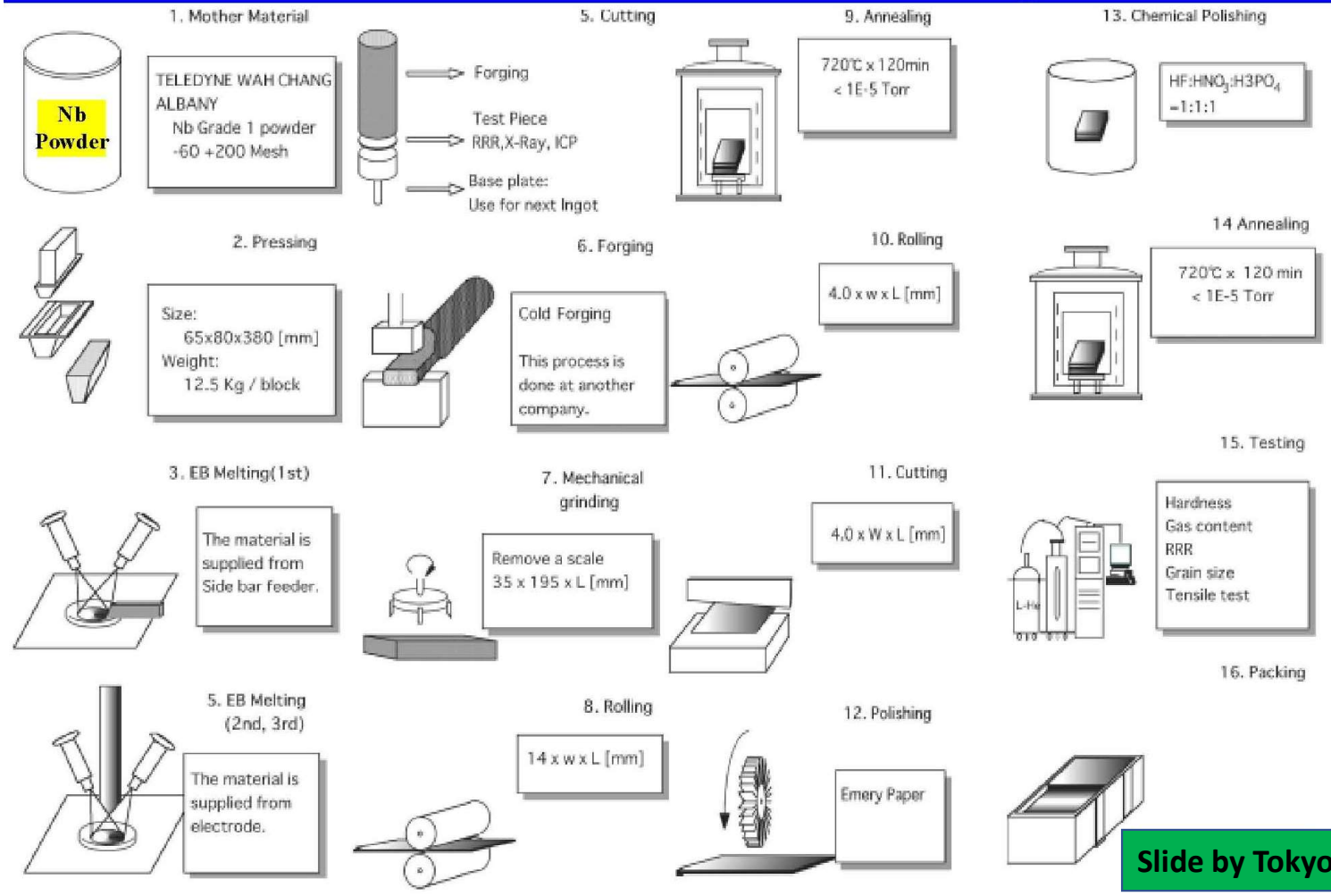
Ta-fraction is not sensitive to the SRF performance if RRR > 300.

Difference of Q values are within the measurement error.

Grain-size studies

Process flow of the industrial Nb production

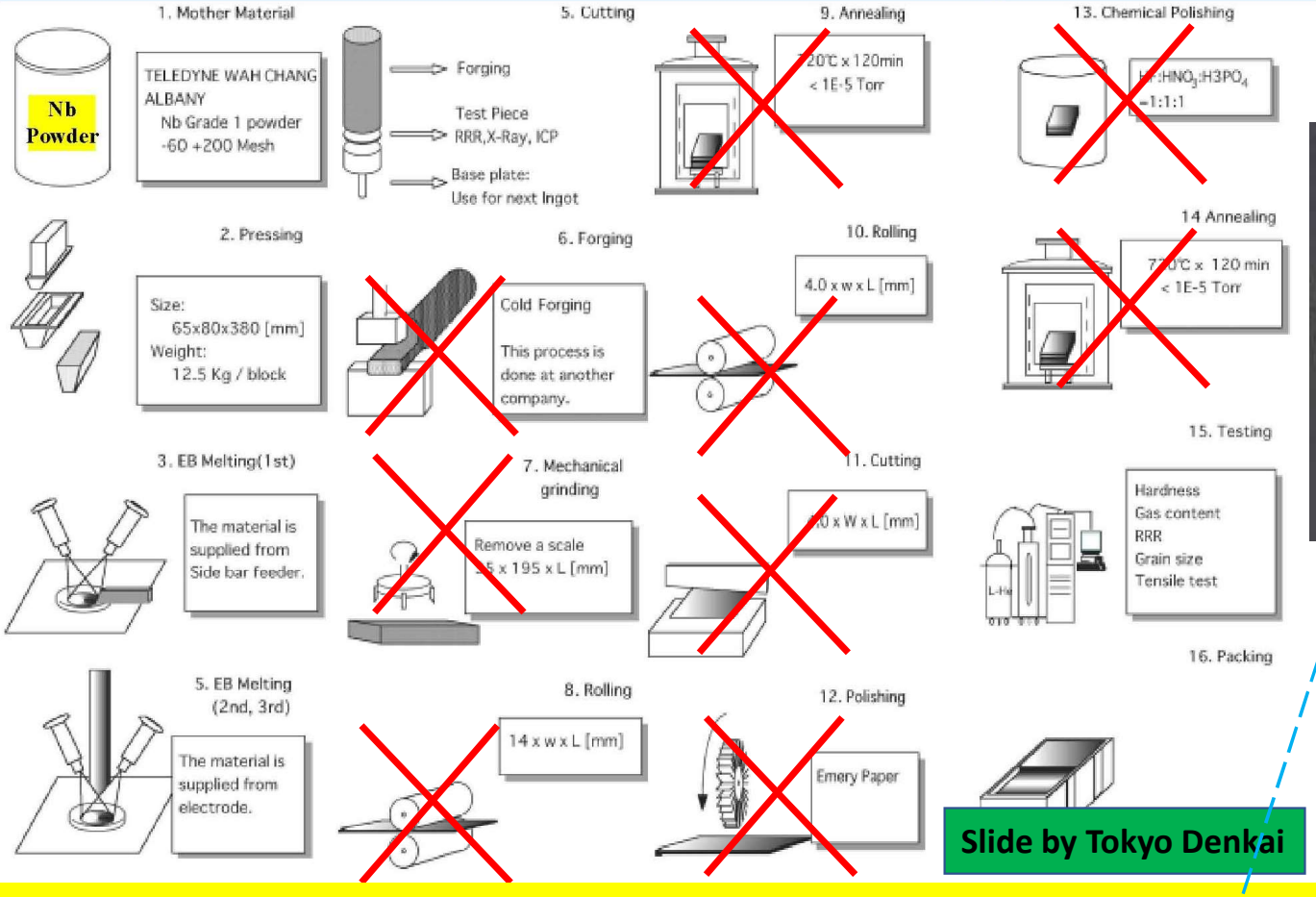
FG Nb



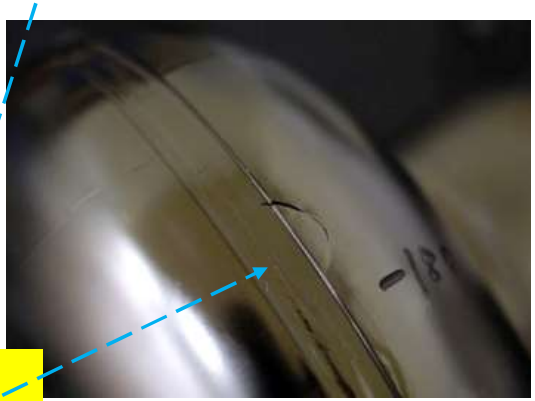
Slide by Tokyo Denkai

LG Nb

Process flow of the industrial Nb production



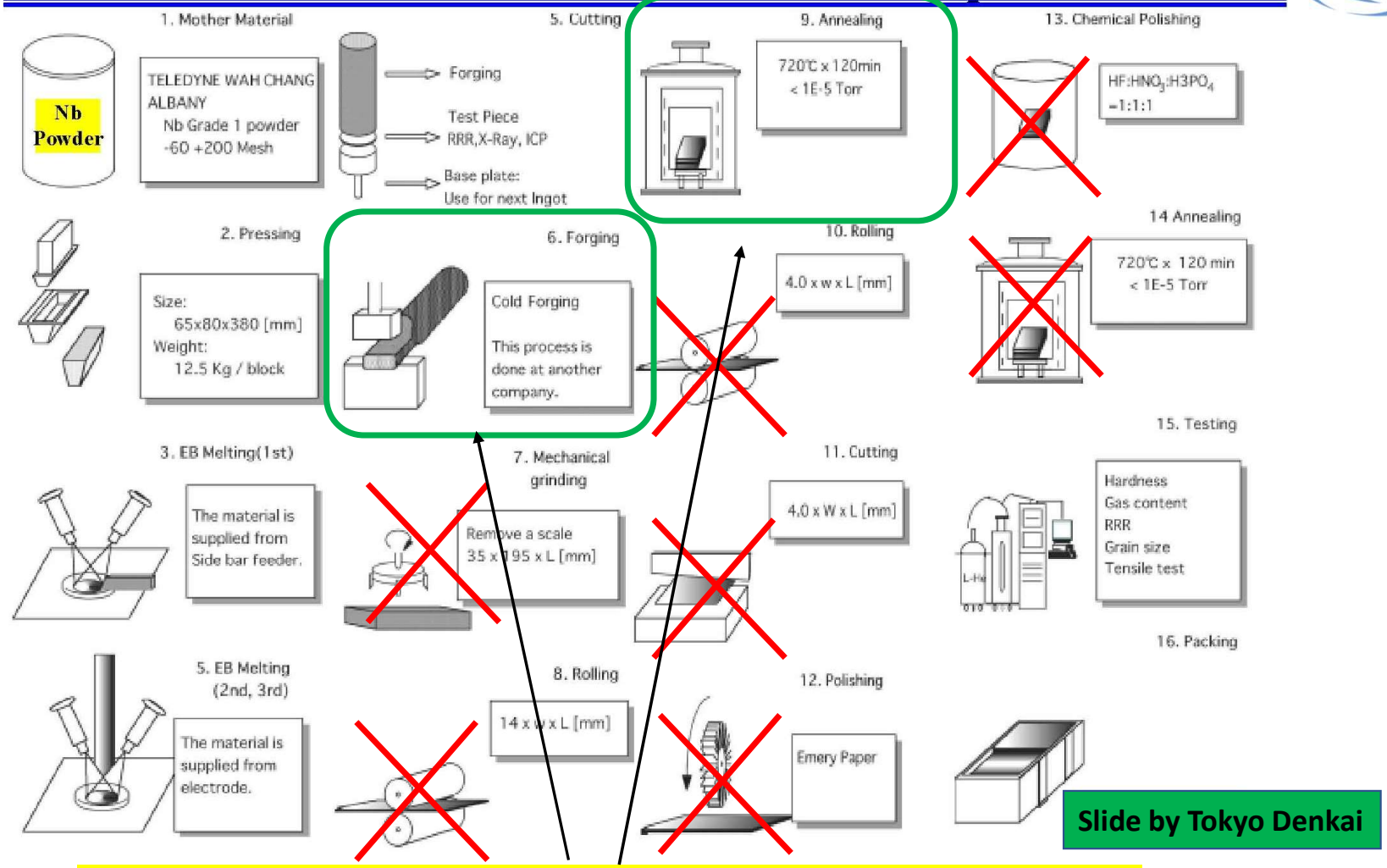
Picture of half cup



LG-Nb realized very good cost performance.
But LG-Nb material causes broad mechanical strength distribution (less uniformity).
(Minimum strength becomes low compared with that of FG-Nb material.)

Process flow of the industrial Nb production

MG Nb

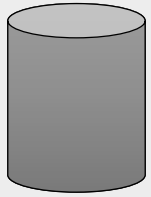


Slide by Tokyo Denkai

Uniformity of Nb material is achieved by Medium-Grain (MG) structure.

Manufacture methods of Large-Grain / Medium-Grain Nb discs

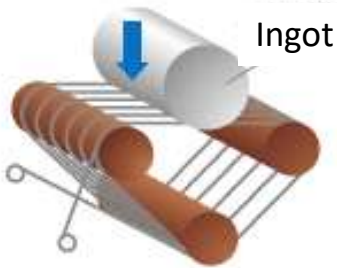
Nb melting



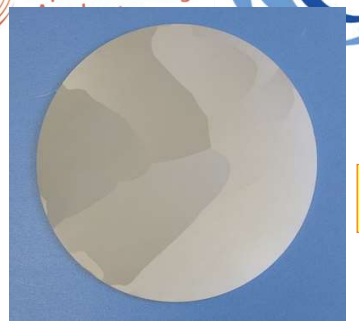
Niobium ingot
(Raw material)

Direct slice of LG ingot

Ingot, as it is



Slicing image
by wire-saw



LG Nb

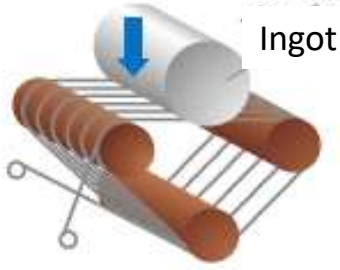
Large Grain (LG) Disc

Grain Size >> 1 cm

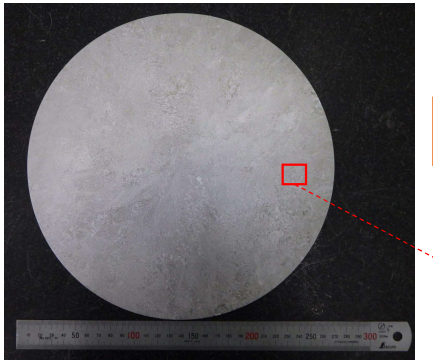
- Aiming for clean, mechanically stable, and cost-effective SRF cavity production.

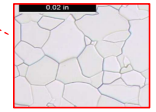
Direct slice of MG ingot

Ingot, forged
and annealed



Slicing image
by wire-saw





MG Nb

A New Approach:

Medium Grain (MG) Disc

Grain Size **< 1 mm**

* The “Nb forged ingot” technology originated by **ATI**, and SRF (GHz) cavities planned to be fabricated and RF tested by **KEK** and **JLab**, to qualify this approach, in collaboration of **ATI, ODU/BSCE, JLab, and KEK.**

MG-Nb demonstration by 1.3 GHz SRF Cavity at KEK

Courtesy: ATI

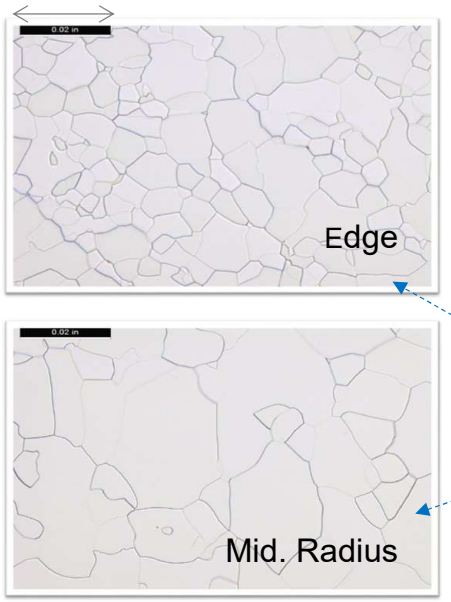
"**Nb RRR**" Billet, annealed

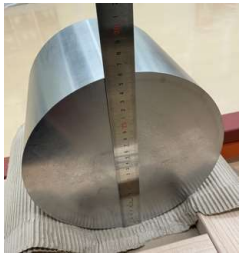
Sample location from forged ingot

Parameters	Nb sheet (FG) (Spec. Eu-XFEL as Reference)	Nb forged ingot (MG) Measured
RRR	$R_{RT}/R_{4.2K}$ ≥ 300	R_{RT}/R_{TC} 450 523
Re-crystallization	100 %	100 %
Grain size (ASTM) Edge, Mid., Center	4 ~ 6	2 , 1, 1.5
Grain size (mm) Edge, Mid.,Center	< 0.05	0.2, 0.25, 0.21
Y.S.-0.2% (RT)	≥ 50 MPa	61 MPa
T.S. (RT)	≥ 140 MPa	141 MPa

MG-Nb: Good mechanical uniformity

0.02 inch (50 μm)





ATI

Grain Size: 0.2 ~ 0.3 mm
Mechanical stability may be expected

Overview of studies

Nb materials	Cavities	FY2018	FY2019	FY2020	FY2021	FY2022
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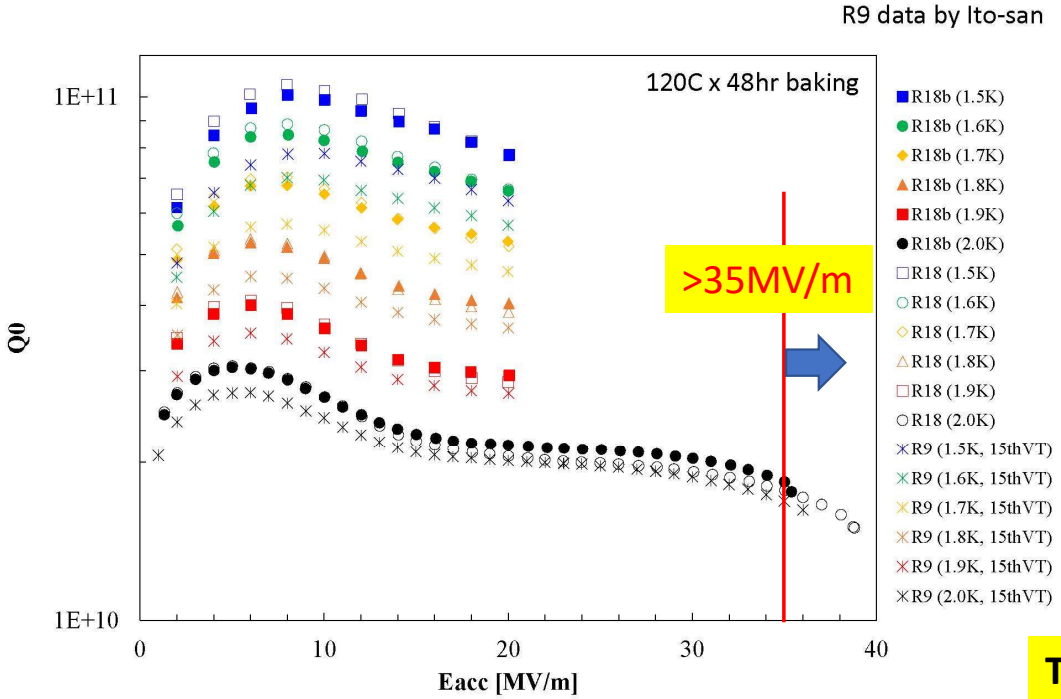
LG High-RRR High-Ta				Two 3-cell cavities were fabricated	VT Eacc > 35MV/m ILC spec. achieved	
					Two 9-cell cavities are under fabrication	VT
MG High-RRR Low-Ta				Two 1-cell cavities were fabricated	VT Eacc > 35MV/m ILC spec. achieved	
						One 9-cell cavity will be fabricated

MG-Nb material （High-RRR, Low-Ta）

Results of MG single-cell cavities.
R18, R18b (vs. R9 / FG single-cell cavity).

R18 vs R18b vs R9



- R18, R18b were fabricated using MG discs
- Inner surface of R18 was mechanically polished during fabrication (R18b was not)
- VT of R18: 38.8 MV/m with Q_0=1.5 × 10^{10}
- VT of R18b: 35.4MV/m with Q_0=1.76 × 10^{10}

Two MG-Nb single-cell cavities have reached > 35 MV/m

Mechanical properties of Nb material

Objective: To pass HPGS regulation

MG Nb material / Tensile tests etc.

Mechanical strength of MG-Nb samples were tested and the results were presented in SRF2021.

The MG-Nb samples were BCP'ed (chemical polished) before tests. There was some brittleness and low elongation of MG-Nb samples in the low temperature tensile-tests (hydrogen absorption problem?).

SRF2021
MOPCAV004
A. Kumar
(July 2021)

MECHANICAL PROPERTIES OF DIRECTLY SLICED MEDIUM GRAIN NIOBIUM FOR 1.3 GHZ SRF CAVITY

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High Energy Accelerator Research Organization (KEK), Tsukuba, Japan
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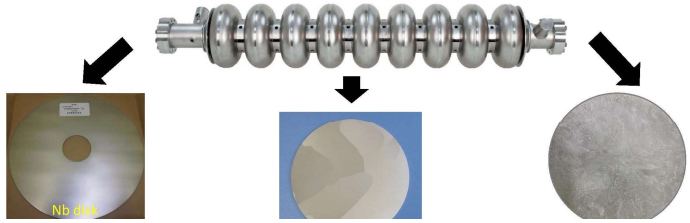
SRF 2021
MOPCAV004

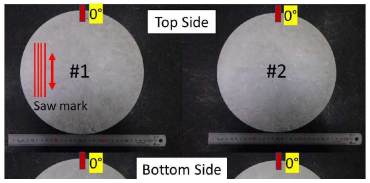
Introduction

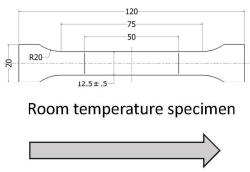
- ILC-250 is an electron-positron collider that requires 7800 1.3 GHz SRF cavities.
- The TDR is already published but the cost of its construction is a major hurdle.
- High purity Niobium (costly) is used to manufacture 9-Cell 1.3 GHz SRF cavity.

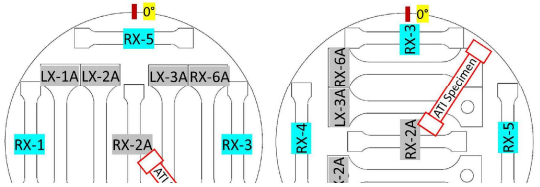


Results and Discussion

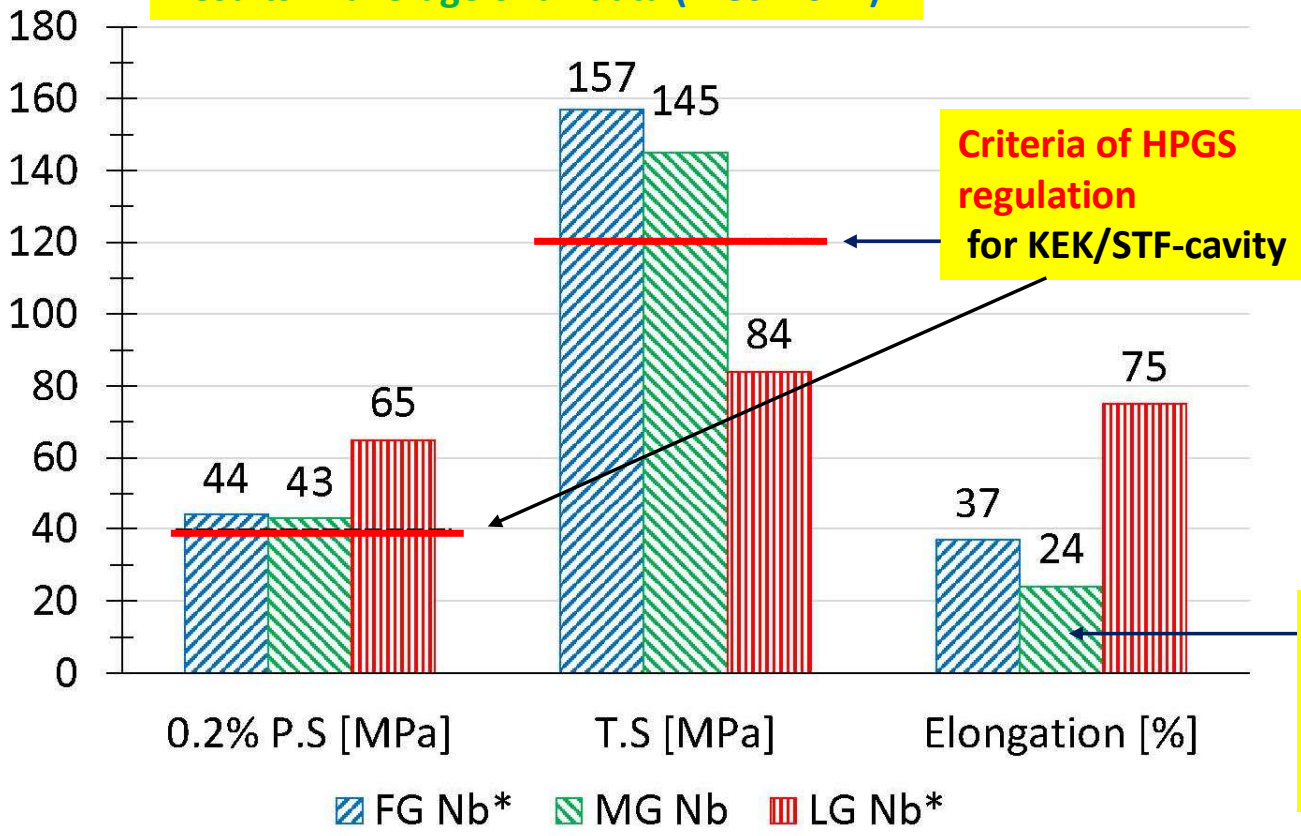
- ATI MG Nb billet was sliced into 65 disks and specimens from top two and bottom two disks were cut for tensile testing.
- All disks were chemically polished, specimens were wire EDM cut and then chemically polished again (see fig below).
- A set specimens were annealed at 800 °C for 3 hrs and the remaining ones were not, considered as in As-received condition (ASR).
- Tensile tests were performed in room temperature and in liquid helium.







Mechanical strength comparison of FG/MG/LG at RT



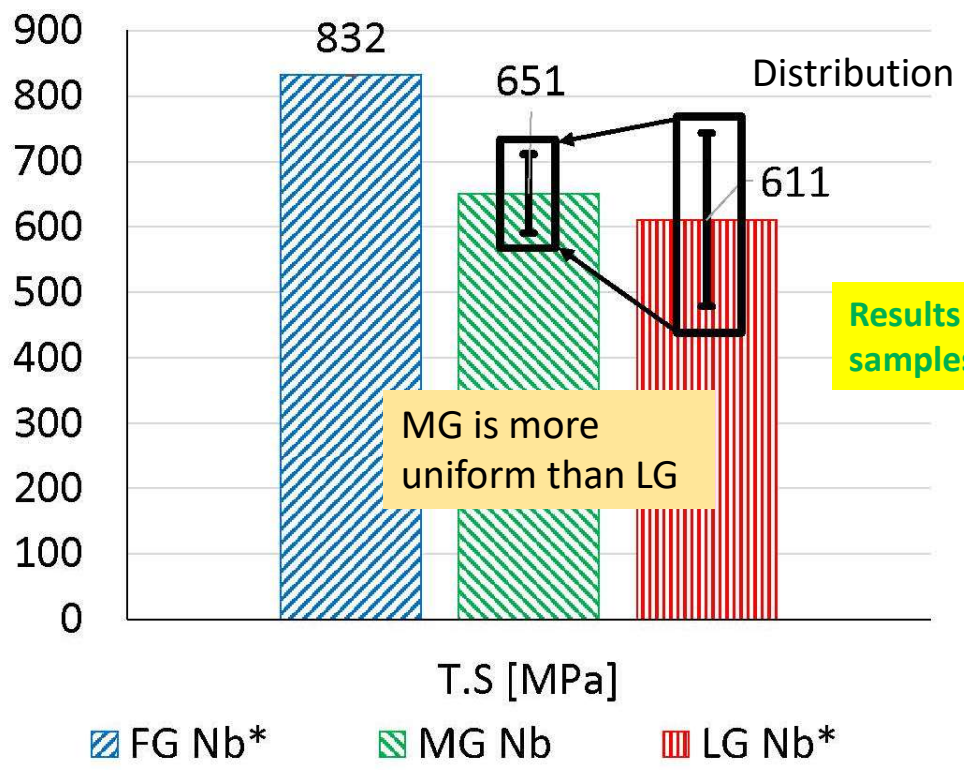
Criteria of HPGS regulation for KEK/STF-cavity

- MG Nb closer to FG Nb than LG Nb at room temperature.
- Elongation is not considered for high pressure code.
- High elongation necessary for press forming of Nb half cells.

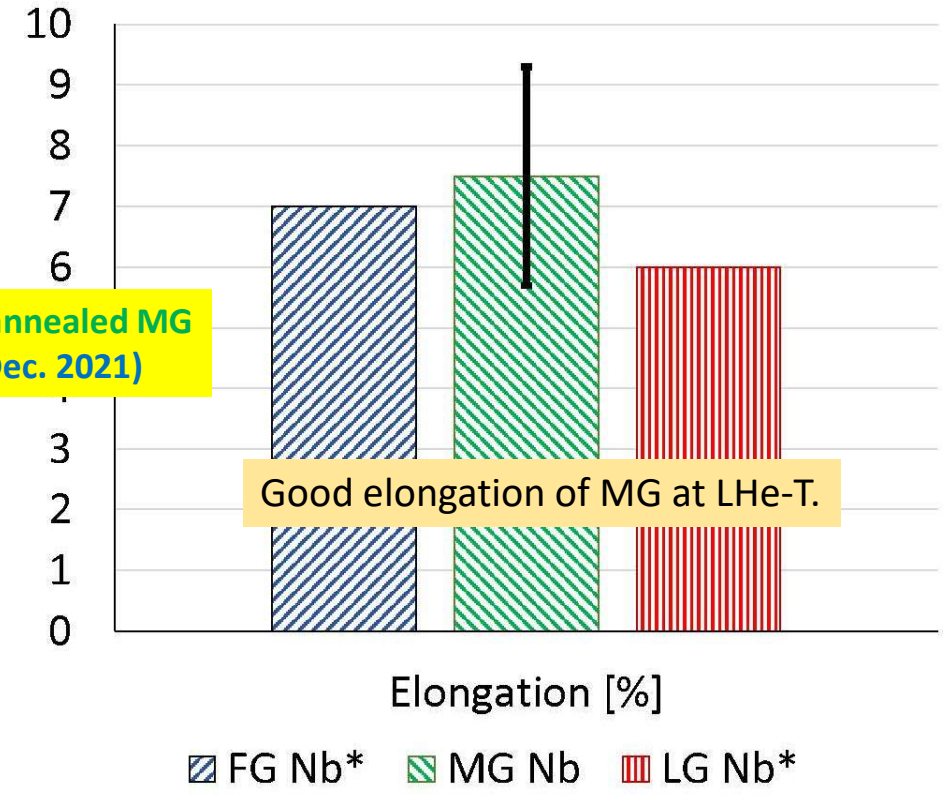
There is a problem of low elongation at RT. This is related to the yield rate of press-forming of half-cups (Elongation is not related to HPGS regulation).

Mechanical strength of MG-Nb achieved the criteria of HPGS regulation.

Mechanical strength comparison of FG/MG/LG at LHe-T



Results of annealed MG samples (Dec. 2021)



Strength of MG-Nb at Lhe-T is better than LG-Nb.
Brittleness and low-elongation of MG-Nb are not observed at LHe-T after annealing process.

Plan in FY2022

Overview of studies

Nb materials	Cavities	FY2018	FY2019	FY2020	FY2021	FY2022
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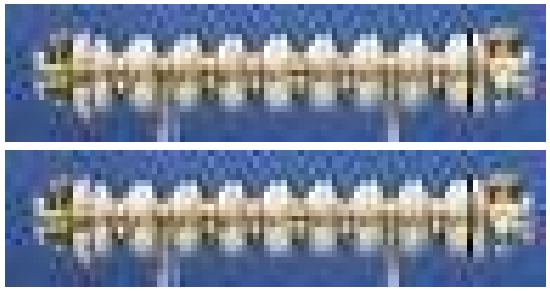
						One 9-cell cavity will be fabricated

Plan-1: Demonstration of Nb material of High-RRR and High-Ta.

LG
High-RRR
High-Ta



Two high-RRR/high-Ta 9-cell cavities will be fabricated and Vertically-Tested (VT) in FY2022.

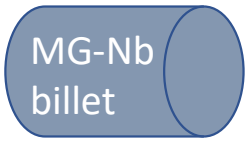
Nb material of high-RRR/high-Ta is cost-effective, and has good SRF performance.

Plan-2: Demonstration of MG-Nb 9-cell cavity SRF performance

MG
High-RRR
Low-Ta

One MG 9-cell cavities will be fabricated and Vertically-Tested (VT) in FY2022.

MG-Nb material is cost-effective, and has both good SRF performance and stable mechanical properties to pass HPGS regulation.



Plan-3: Reproducibility verification of mechanical properties of MG-Nb billet/disk

MG-Nb billet#	Delivery to KEK	Billet dimension	Studies
Billet #1	Feb. 2021	D 260 mm L 200 mm	Mechanical properties were already tested. (Tensile tests at RT and LHe-T)
First Trial of MG-Nb Forging + HT			One TESLA-shape 9-cell cavity will be fabricated.
Billet #2	Mar. 2022	D 260 mm L 270 mm	Mechanical properties will be tested. (Tensile tests at RT and LHe-T + charpy impact tests)
Objective: **Reproducibility**			Two TESLA-shape 9-cell cavities will be fabricated (not scheduled yet).
Billet #3	Dec. 2022	D 260 mm L 270 mm	Trial to improve elongation in production process in vender.
Objective: **Improvement**			Mechanical properties will be tested. (Tensile tests at RT and LHe-T + charpy impact tests)
			Two TESLA-shape 9-cell cavities will be fabricated (not scheduled yet).

Summary

- Nb: High RRR is necessary for high SRF performance.
- Nb: Ta-fraction is not sensitive to the SRF performance.

- MG-Nb: Cost effectiveness, good SRF performance & mechanical properties to pass HPGS regulation are demonstrated.
  - SRF Performance of MG single-cell cavities reached the ILC spec. as well as LG cavities.
  - MG elongation improvements is anticipated for ILC mass-production (yield rate of press-forming of half-cell).

- LG-Nb: Cost effectiveness & good SRF performance, but the mechanical strength stability is the issue to be improved to pass HPGS regulation.