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DEVELOPMENT OF THE DIRECTLY-SLICED NIOBIUM MATERIAL FOR HIGH PERFORMANCE SRF CAVITIES

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Abstract

For cost reduction studies for the ILC, KEK has been conducting R&D on direct sliced Nb materials such as large grain and medium grain Nb. 1-cell, 3-cell, and 9-cell cavities have been manufactured, and each has demonstrated a high-performance accelerating gradient exceeding 35 MV/m. The results of applying high-Q/high-G recipes, such as two-step baking and furnace baking to these cavities are also shown. Moreover, mechanical tests have been carried out for the beforementioned materials to evaluate their strength for application to the High-Pressure Gas Safety Law. The status of development of large grain and medium grain Nb will be presented.

INTRODUCTION

The International Linear Collider (ILC) is an electron-positron collider accelerator, that requires approximately 7800 1.3 GHz Niobium 9-cell cavities to attain 250 GeV centre-of-mass energy and is extendable up to 1 TeV [1, 2]. The ILC design update for the ILC-250 (GeV) has been already published [2] but the cost of its construction is a major hindrance. Cost reduction studies are being carried out at KEK and other facilities all over the world for the realization of ILC. A part of the cost-reduction studies at KEK is to research on various grades of Niobium, to reduce the manufacturing cost of the SRF cavity. Research on SRF cavities manufactured with fine grain Nb (FG Nb) has been carried out extensively but the cost of the material is high due to its manufacturing process. In this paper, we would like to introduce current progress regarding cost-effective alternative grades of Nb for the SRF cavity manufacturing.

The operational requirement for the ILC's 1.3 GHz 9-cell cavities is accelerating gradient (E_{acc}) > 31.5 MV/m with quality factor (Q_0) > 1E10, such specification generally requiring Niobium with high purity (RRR >300). However, the mechanical strength of Nb generally deteriorates with higher purity (Table 1). The Nb material in the SRF community is usually classified in two categories [1, 3, 4]:

1. Residual Resistivity Ratio (RRR) – Low (< 100), Medium (100 to 300) and High (> 300).
2. Grain Size – Fine Grain (< 50 µm), Medium Grain (MG) (200 - 300 µm with occasional grains as large as 1-2 mm) and large grain (LG) (few millimetres to centimetres) [1, 3, 4].

DIRECTLY-SLICED NIOBIUM

Large Grain (LG) Nb was developed as a clean and low-cost alternative, where the LG Nb Ingot is directly sliced into disks, as shown in Fig. 1. Its grain size usually varies from a few mms to several cms, as seen in Fig. 2, due to which it has anisotropic mechanical properties causing its 0.2% Yield Strength (Y.S) and Tensile Strength (T.S) to sometimes fall short of the mechanical property requirement set for 9-Cell 1.3 GHz SRF cavities. W. Singer et al., Zhao et al., Enami et al., Yamanaka et al., has conducted in depth research on determining the mechanical properties of the LG Nb at various temperatures and strain rate ranges [5-8].

Medium Grain (MG) Niobium has an average grain size of 200 - 300 µm with occasional grains as large as 1-2 mm as a potential alternate to both FG and LG Nb, and potential cost reduction compared to FG Nb [1, 3, 4]. It is formed by forging and annealing of the LG Nb ingot to a billet to achieve smaller grain sizes. The forged billet is then directly sliced in disk forms, which lowers the number of manufacturing steps that are involved with FG Nb, such as rolling, etching, annealing etc., elimination of these steps having the potential to reduce material cost [1, 3 ,4], as seen in Fig.1.

Table 1: Chemical Composition and Mechanical Properties Measured by ATI (Unit of Chemical Composition: wt ppm)

C	H	O	N	RRR	Hardness (HV10)
<20	<3	<50	<20	> 300	~ 41

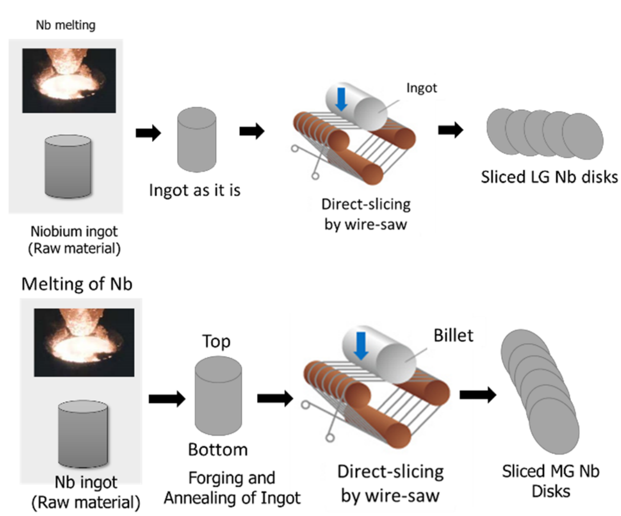

Figure 1: LG Nb manufacturing process (up) and ATI MG Nb manufacturing process (below) [3, 4].

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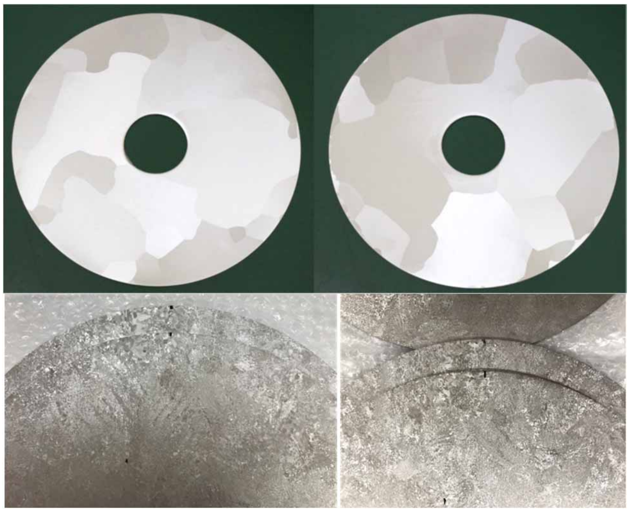

Figure 2: LG Nb sliced disk (up) [9] and ATI MG Nb sliced disk (below).

MECHANICAL PROPERTIES OF DIRECTLY-SLICED NIOBIUM

Tensile Test and Its Setup

Tensile testing is a methodology where a material is subjected to uni-axial tension until failure to obtain mechanical properties, such as, Young's modulus (E), 0.2% Yield Strength (Y.S), Tensile Strength (T.S) and Elongation of the material. Shimadzu's Autograph AG-5000C tensile test machine was utilized to conduct tensile tests at room and cryogenic temperature (in liquid helium).

The cross-head speed is kept constant at 2 mm/min for all tests with a maximum nominal strain rate of 4.4E-4 s^{-1}. Kyowa strain gages were bonded on the specimens to determine strain w.r.t the applied load. Kyowa strain amplifier DPM-911B measured the strain produced in the strain gages during tensile tests. Elongation is determined by measuring the percentage change in the gage length (50 mm) of the specimen before and after failure. For testing in liquid helium, a custom-built cryostat, as shown in Fig. 3, is used where test pieces are dipped in liquid helium during the tensile test. With this cryostat three specimens can be tested for one cycle of cooldown.

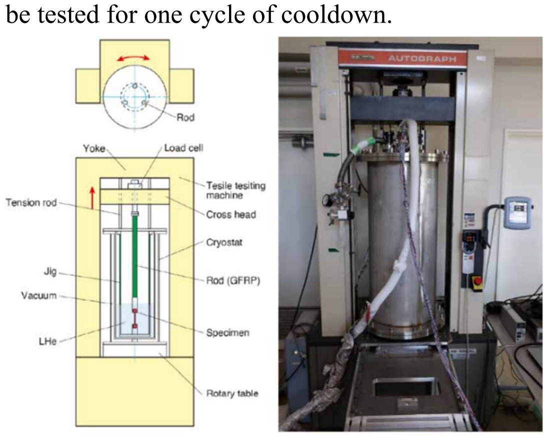

Figure 3: Schematic view of cryostat with tensile testing machine for liquid helium dipped testing [7].

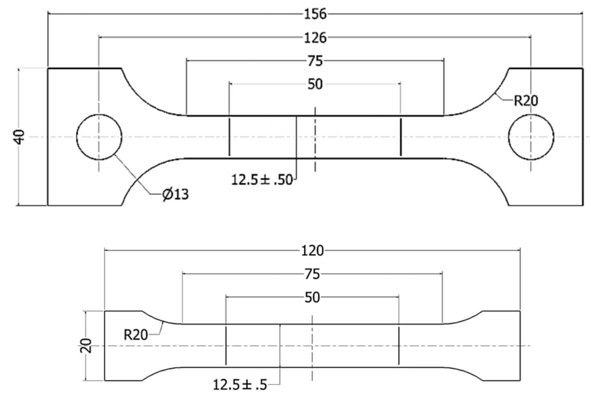

Figure 4: Specimen for tensile tests: Liquid helium or room temperature tests (up) and Room temperature specific tests (below) (JIS Z 2241 13B with t = 2.8 mm).

Mechanical Properties at Room Temperature

The mechanical properties of the MG Nb material were evaluated from the top, middle and bottom of the ATI MG Nb billet. The sliced disks were chemically polished (CP), and the specimens were wire EDM cut from the polished disks. A set of specimens went through another round of CP to be annealed at 800 °C for 3 hours at vacuum pressure of 2×10^{-5} Torr and the remaining were tested without annealing generally known as in As-received (ASR) condition. The specimens were cut according to Japanese Industrial Standards JIS Z 2241, as shown in Fig. 4, and the tests were conducted in accordance with JIS Z 2241 for room temperature tensile tests and JIS Z 2277 for the tensile test of metallic materials in liquid helium. The total number of MG Nb specimens that were tested at room temperature were 39 (18 Annealed and 21 ASR). The details for specimen cut-out are already reported by Kumar et al. [3].

Table 2: Mechanical Properties at Room Temperature for Various Grades of Niobium

Material	Y.S [MPa]	T.S [MPa]	Elongation [%]
FG Nb (Annealed)	44	157	> 35
MG Nb (Annealed)	$39^{\pm2}$	$123^{\pm5}$	$25^{\pm3}$
MG Nb (ASR)	$43^{\pm4}$	$145^{\pm7}$	$24^{\pm4}$
LG Nb (Annealed)	65	84	75

In available literatures, it is well known that the FG Nb has isotropic properties and good formability (high elongation > 35%). Although, the same cannot be said for the LG Nb which is known to have anisotropic properties dependent on its grain orientation but is also known to have good formability [5, 8]. The room temperature mechanical properties for the MG Nb are summarized in Table 2 and compared with already reported properties of FG and LG Nb. The MG Nb mechanical properties are uniform throughout the billet with minimal standard deviation. The Y.S and T.S of the MG Nb is much closer to FG Nb, when it comes to material in ASR condition. However, the elongation of the material is lower than both FG and LG Nb.

Mechanical Properties in Liquid Helium

The trend with anisotropic behaviour for LG Nb continues at cryogenic temperatures too, as reported by Yamanaka and Enami et al. [6, 7]. A large variation in T.S is observed for the LG Nb material, as seen in Table 3 and Fig. 5 [9]. The annealed MG Nb specimens (11 specimens) showed better isotropic behaviour w.r.t LG Nb, as seen in Table 3 and Fig. 5. The T.S and the elongation of the MG Nb recovered after annealing at 800 °C for 3 hours, compared to previously reported data [4]. The T.S of the MG Nb material shows 55% reduction in its standard deviation compared to LG Nb, hence, showing much better isotropic behaviour than LG Nb. Although, the average T.S of the material is roughly 80% of the FG Nb material. The elongation of the MG Nb material is similar to FG Nb material.

Table 3: Mechanical Properties of Annealed (800 °C for 3 hrs) FG Nb, MG Nb and LG Nb in Liquid Helium

Material	Y.S [MPa]	T.S [MPa]	Elongation [%]
FG Nb*	517	830	7
MG Nb**	$283^{\pm 34}$	$651^{\pm 60}$	$7.5^{\pm 1}$
LG Nb*	-	$611^{\pm 132}$	6

*Data is from the testing conducted at KEK for FG and LG Nb [7, 8].

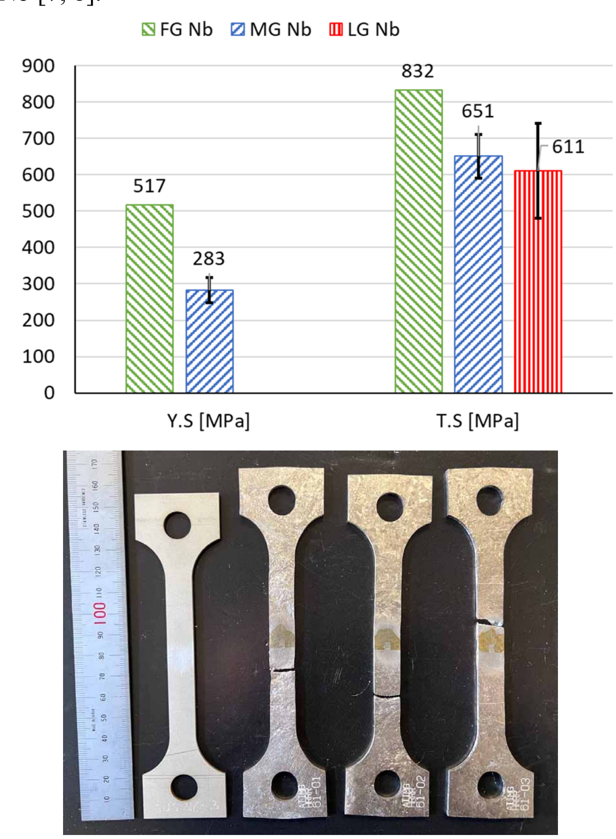

Figure 5: Mechanical properties of various grades Niobium in Liquid helium (up); MG Nb annealed specimens tested in liquid helium (below).

PERFORMANCE OF LG NB CAVITIES TESTED AT KEK

LG Nb Study with High and Low Tantalum

At KEK, four 3-Cell HRRR LG Nb Tesla cavities were manufactured as a part of the cost reduction study, as seen in Fig 6. A part of this study was to determine the effect of tantalum content in LG Nb on the performance of SRF cavities. Basically, the material with high Ta content is cheaper than the material with low Ta content. The specification of the LG Nb material with which the cavities were manufactured are shown in Table 4. The cavities inner surface was treated with Pre EP (5 µm), Bulk EP-1 (100 µm), Annealing (800 °C for 3hrs) and final EP-2 (20 µm). The final EP-2 condition varies for cavities from 5-30 µm followed with high pressure rinsing (HPR)[9].

Table 4: Chemical Composition of the LG Nb Material

Material	RRR	Ta [ppm]	Cavity
ILC TDR	>300	<500	-
HRRR Low Ta	500	20	KEK-R16, R16b
HRRR high Ta	363	1390	KEK-R17, R17b

Figure 6: LG Nb cavities manufactured at KEK [9].

The vertical test (VT) results of the cavities are shown in Fig. 7 and it was concluded that the content of Ta in LG Nb ingot did not affect the performance of the LG Nb cavities. Moreover, with initial pi mode measurement all cavities satisfied the ILC specification (Eacc = 35 MV/m at Q_0 = 1.0E10) for the standard surface treatment of 120 °C baking for 48 hrs. All cavities reached Eacc = 40 MV/m or above with Q_0 of 1.0E10 [9]. However, performance degradation in the Eacc for final Pi mode measurement (measurement after quench) was noticed with all cavities, albeit to varying degrees, as summarized in Table 5. The main cause for this is not clear, it is possible that the enviornmental megnetic field is locally trapped at the quench location. A change of 60 mG was noticed at the flux gate sensors on the outer surface of cavity [9]. However, after warmup the performance of the R17b cavity was

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recovered, so it was concluded that the initial Pi mode performance of the LG Nb cavity can be recovered.

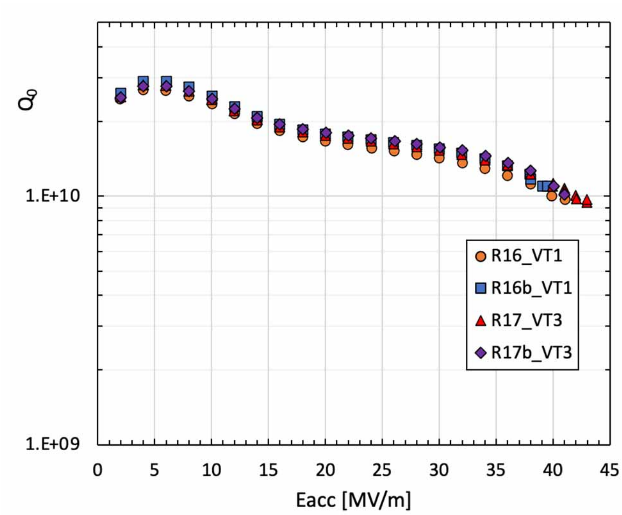

Figure 7: VT test results at 2.0 K for the LG Nb cavities which went through standard surface treatment.

Table 5: Eacc before and after Quench during VT

Cavity	Eacc initial [MV/m]	Eacc final [MV/m]	Delta
R16 VT1	41.4	37.2	-4.2
R16b VT1	39.7	39.5	-0.2
R16b VT2	41.7	35.4	-6.3
R16b VT3	40.6	34.7	-5.9
R17 VT3	43.1	30	-13.1
R17b VT3	41.9	36.6	-5.2

LG Nb for High Q – High G

Recently, the 3-cell LG Nb cavity R17b was vertically tested with high Q- high G surface treatment such as, 2-step baking and furnace baking at 200 °C for 3 hrs. In 2-step baking, the cavity (being pumped) is baked for 70 °C for 4 hrs and then 120 °C for 48 hrs, continuously, with ribbon heaters. In the case of furnace baking, the dried cavity (after HPR) is baked in a vacuum furnace [10]. The pressure in the furnace is maintained at 1E-6 Pa at room temperature and kept to < 10^{-4} Pa during baking. The ramp rate to desired baking temperature is 200 °C/hr.

For 2-step baking, the improvement in Q_0 was within the error range. However, for furnace baking, this cavity reached Eacc > 40 MV/m with appreciable improvement in Q_0. At Eacc = 36 MV/m, the Q_0 of the cavity improved from 1.36E10 -> 1.50E10 (2-step baking) -> 1.75E10 (furnace baking). Moreover, the residual resistance for the furnace baked cavity was the least, as seen in Fig. 8.

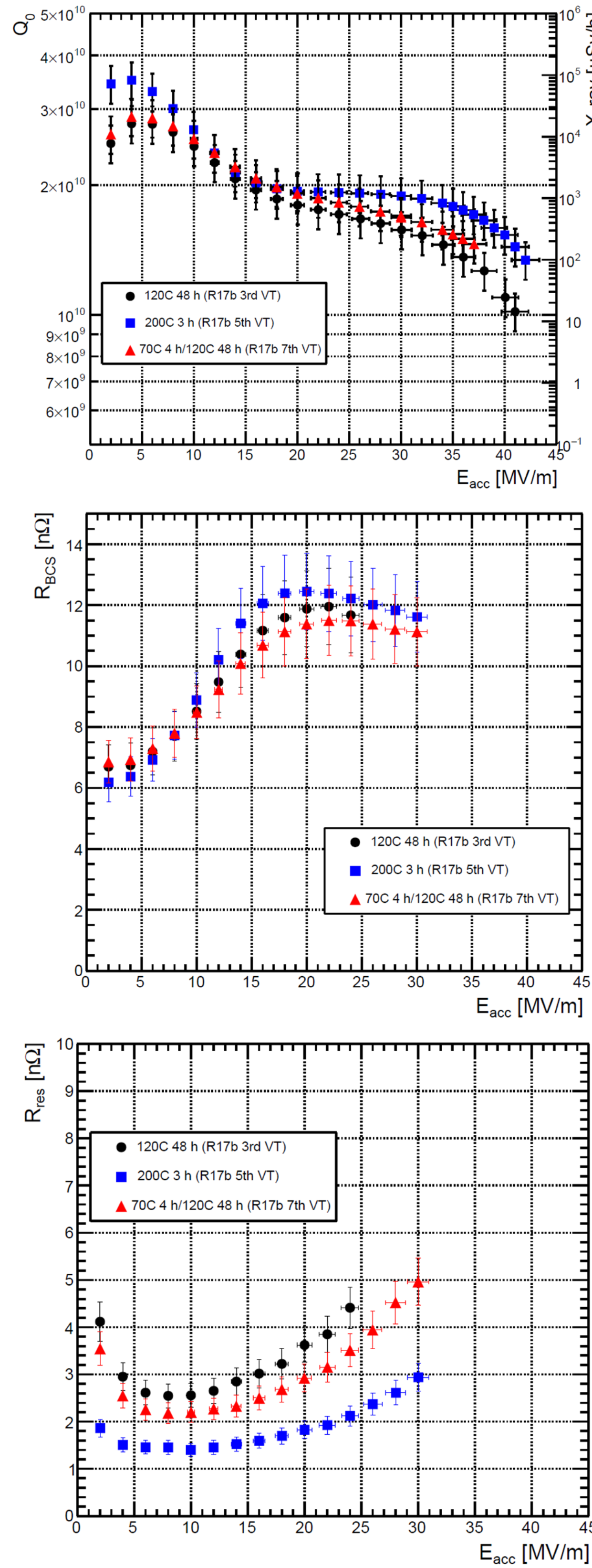

Figure 8: Eacc-Q_0 curve for the 3-Cell LG Nb (R17b) cavity for the standard baking, 2-step baking, and furnace baking surface treatment at 2.0 K (Up); Resistance deconvolution for the 3-Cell R17b cavity for the standard baking, 2-step baking, and furnace baking recipe (middle and below).

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A 9-Cell LG Nb Tesla cavity (KEK-5) was also tested with standard and Furnace baking (200 °C for 3 hrs) surface treatment. This cavity has reached Eacc = 32 MV/m with Q_0 = 7.6E9, with standard baking surface treatment. However, with furnace baking at 210 °C for 3 hrs, its Q_0 improved to 1.45E10, as seen in Fig. 9, clearing the ILC operational specification of Eacc = 31.5 MV/m with Q_0 = 1.0E10.

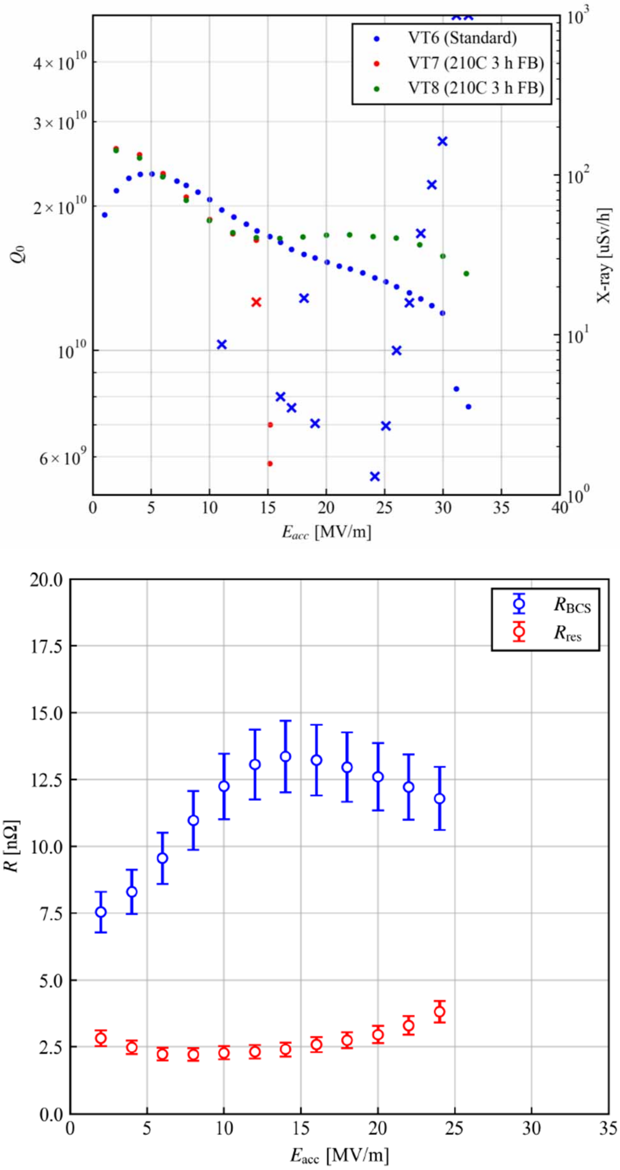

Figure 9: Eacc-Q_0 curve for the 9-Cell LG Nb KEK-5 cavity at 2.0 K (up); Resistance deconvolution for the KEK-5 cavity for the furnace baking recipe (below).

PERFORMANCE OF MG NB CAVITIES TESTED AT KEK

At KEK, two 1-cell high RRR MG Nb Tesla cavities were manufactured named as R18 and R18b, as seen in Fig. 10. Initially, the R18 cavity was treated with Pre EP (5 µm), Bulk EP-1 (100 µm), Annealing (800 °C 3hrs), final EP-2 (20 µm), HPR and Standard Baking (120 °C for 48 hrs). The VT results for standard baked R18 cavity is presented by T. Dohmae et al. [11]. In summary, the cavity reached Eacc= 38.8 MV/m at Q_0 = 1.5E10 with the standard surface treatment, without any multipacting and field emmision.

Figure 10: MG Nb fabricated cavity at KEK[14].

MG Nb for High Q – High G

Recently, R18 cavity has been vertically tested with 2-step baking and Furnace baking (200 °C for 3 hrs). The cavity surface was refreshed with Cold EP-2 (10 µm) followed with HPR and the required surface treatment. During cooldown, a solenoid coil was used to cancel out the environmental magnetic field and beam pipe heater was operated to provide thermal gradient for better flux expulsion. The performance of this cavity improved with respect to its Q_0, constantly staying above 1.5E10 at 35 MV/m, as seen in Fig 11. The Q_0 improved by approximately 10% at the ILC operational specification Eacc = 31.5 MV/m with high Q - high G surface treatment, as seen in Fig. 10. However, no improvement is noticed with respect to its Eacc, although, still stayed at > 35 MV/m for each surface treatment. The summary of the VT results are shown in Table 6, and during the VT no multipacting or field emmision occurred.

Table 6: VT Summary of MG Nb R18 Cavity at 2.0 K

Surface treatment	Eacc max. [MV/m]	Q_0 at Eacc = 35 MV/m	Q_0 Max.
Standard baking	38.8	1.77E10	3.06E10
2-Step baking	35	1.75E10	3.47E10
Furnace baking (200 C * 3hrs)	37	2.02E10	3.89E10

Flux Sensitivity Study for High Q – High G Techniques

Flux sensitivity studies were also conducted for the R18 MG Nb cavity. For this study, a constant magnetic flux of 20 mG is trapped during cooldown (superconducting transition) of the cavity, with the help of surrounding solenoid coil. The VT results for the flux trapped cavity, with 2-step baking and furnace baking (200 °C for 3 hrs) surface treatment, are shown in Fig 12.

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No degradation was observed in Eacc for each treatment, when compared to VT without trapped flux, as seen in Figs. 11 and 13. However, there was degradation in Q_0 of the cavity with respect to without trapped flux condition, as also observable from Figs. 10 and 12. The Q_0 of the cavity degraded from approximately 1.75E10 to 7.70E9, at Eacc = 35 MV/m. The effect of trapped flux on the surface resistance of the MG Nb 1-Cell cavity is approximately 1.2 n^ /mG at Eacc = 35 MV/m. This is similar to the data provided by Ito et al. for a standard baked FG Nb 1-Cell cavity [10].

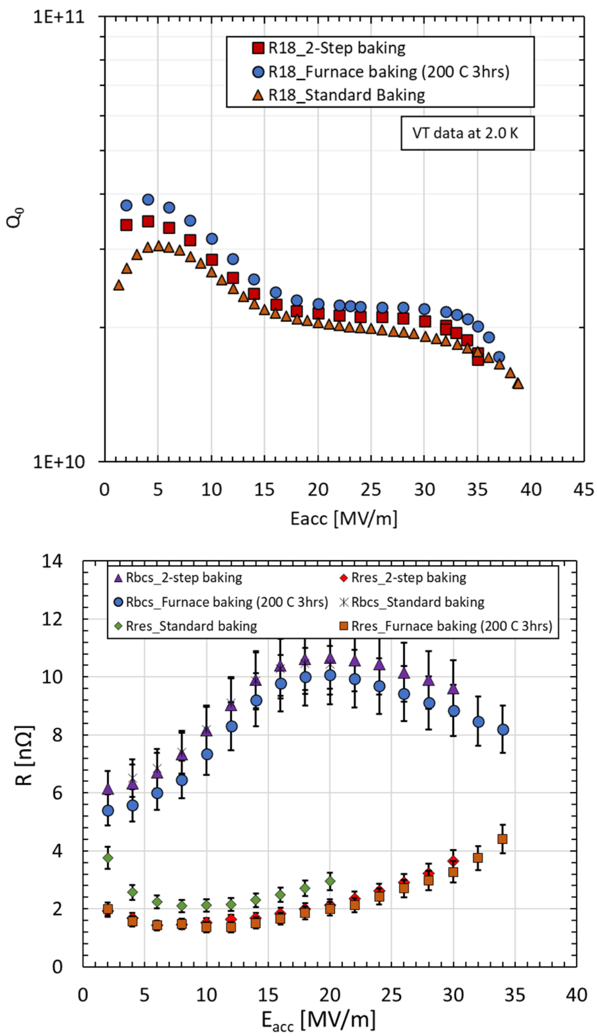

Figure 11: Eacc-Q_0 curve for the 1-Cell R18 cavity for the standard baking, 2-step baking, and furnace baking surface treatment at 2.0 K (up); Resistance deconvolution for the R18 cavity for the standard, 2-step, and furnace baking surface treatment (below).

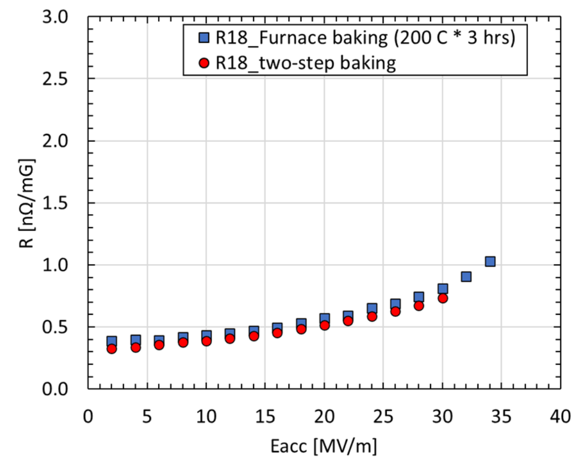

Figure 12: Flux sensitivity for 1-Cell MG Nb (R18) cavity for 2-Step baking and Furnace baking (200 °C for 3 hrs).

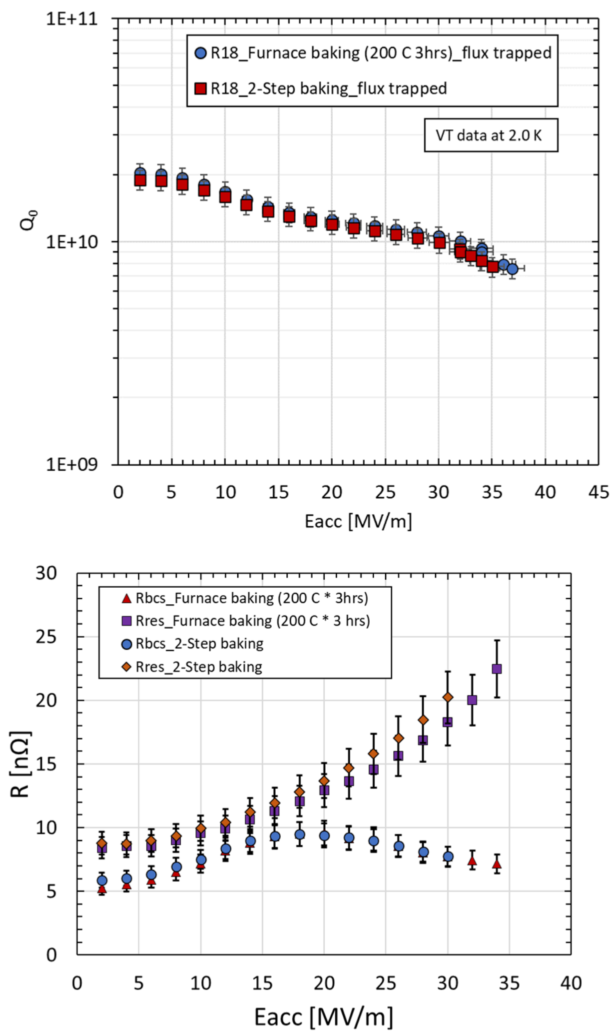

Figure 13: Eacc-Q_0 curve for the flux trapped (20 mG) 1-Cell R18 cavity for the 2-step baking and furnace baking surface treatment at 2.0 K (up); Resistance deconvolution for the flux trapped (20 mG) R18 cavity for the 2-step baking and furnace baking surface treatment (below).

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CONCLUSION

At KEK, directly-sliced Nb materials such as LG and MG Nb have been studied extensively. The LG Nb cavities have attained ILC specification consistently with standard and high Q – high G surface treatments. However, LG Nb material's anisotropic mechanical properties currently makes it difficult to apply for ILC. However, MG Nb is an exciting new material for application to SRF cavities. Its 1-cell cavity has attained ILC specification with standard and high Q – high G surface treatment. Moreover, it is much better isotropic mechanical properties make it a cost-effective alternate to FG Nb. At KEK, a 9-Cell MG Nb Tesla cavity is planned to be manufactured this year.

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