ISBN: 978-3-95450-233-2 ISSN: 2673-5504 doi:10.18429/JACoW-SRF2021-MOPCAV004

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

A. Kumar†, T. Saeki, T. Dohmae, S. Michizono, M. Yamanaka, Y. Watanabe, K. Abe, A. Yamamoto
High Energy Accelerator Research Organization (KEK), Tsukuba, Japan
N. Lannoy, A. Fajardo, ATI Specialty Alloys and Components, Albany, Oregon, USA
G.R. Myneni[1], Jefferson Laboratory, Newport News, Virginia, USA
[1]also at, BSCE Systems Inc., Yorktown, Virginia, USA

Abstract

At KEK, research is being conducted to manufacture cost-effective 1.3 GHz superconducting radio frequency cavities based on the fine grain (FG) and large grain (LG) Niobium (Nb) materials. Medium grain (MG) Nb has been proposed and developed as an alternative to the FG and LG Nb, being expected to have better mechanical stability with a cost-effective and clean manufacturing approach. MG Nb has an average grain size of 200 - 300 µm, which is approximately 100 times smaller than the LG Nb, however, there are occasional grains as large as 1-2 mm. As such, it is expected to have isotropic properties rather than the anisotropic properties of LG Nb. In this paper, we will outline the mechanical properties of the directly sliced high RRR MG Nb material (manufactured by ATI), and a comparative study will be presented with respect to FG and LG Nb. Moreover, the viability of MG Nb for the global high-pressure regulation for 1.3 GHz SRF cavity 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. P. Kneisel et al. reviewed the Niobium Ingot material for SRF cavities and has detailed the Niobium Ingot manufacturing for various grades of Niobium [3]. In this paper, we would like to introduce a cost-effective alternative grade of Niobium for the SRF cavity manufacturing.

Various Grades of Nb for 1.3 GHz SRF Cavities

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

1. Residual Resistivity Ratio – Low (< 100), Medium (100 to 300) and High (> 300).
2. Grain Size – Fine Grain (< 50 µm) and Large grain (few millimetres to centimetres).

After the Nb is extracted from mines, it is melted by electron beam melting method under vacuum to remove interstitial impurities such as H, C, O and N to form it in an ingot, which is largely in LG Nb form [1]. The fine grain (FG) Nb is then manufactured by a series of forging, rolling, annealing and etching process reducing the ingot in sheet forms, producing grains with size < 50 µm, hence it has isotropic mechanical properties [1]. Research on SRF cavities manufactured with FG Nb has been carried out extensively but the cost of the material is high due to its manufacturing process. Some of the renowned research on determining the mechanical properties of FG Nb has been conducted by G. R. Myneni et al., Nakai et al. etc [4, 5].

Large Grain (LG) Nb was developed as a clean and low-cost alternative, where the LG Nb Ingot is directly sliced into disks. Its grain size usually varies from a few mms to several cms, 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 [6-9].

ATI MG Nb

There is another type of material that the authors would like to introduce in the second category called as Medium Grain (MG) Niobium [1, 10]. It has an average grain size of 200 - 300 µm with occasional grains as large as 1-2 mm and was manufactured by ATI in 2020 as a potential alternate to both FG and LG Nb, with better formability than LG Nb and potential cost reduction compared to FG Nb. It is formed by forging and annealing of the LG Nb ingot to a billet to achieve smaller grain sizes, as shown in Fig. 1. 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. The MG Nb billet manufactured by ATI and delivered to KEK is a high RRR Nb and its specifications are given in Table 1. At KEK, we have been conducting tensile tests to characterize the mechanical properties of this material at room and in liquid helium temperatures.

ISBN: 978-3-95450-233-2 ISSN: 2673-5504 doi:10.18429/JACoW-SRF2021-MOPCAV004

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

ATI billet location	Y.S [MPa]	T.S [MPa]	Elongation [%]
Top	56	146	52
Bottom	61	141	52

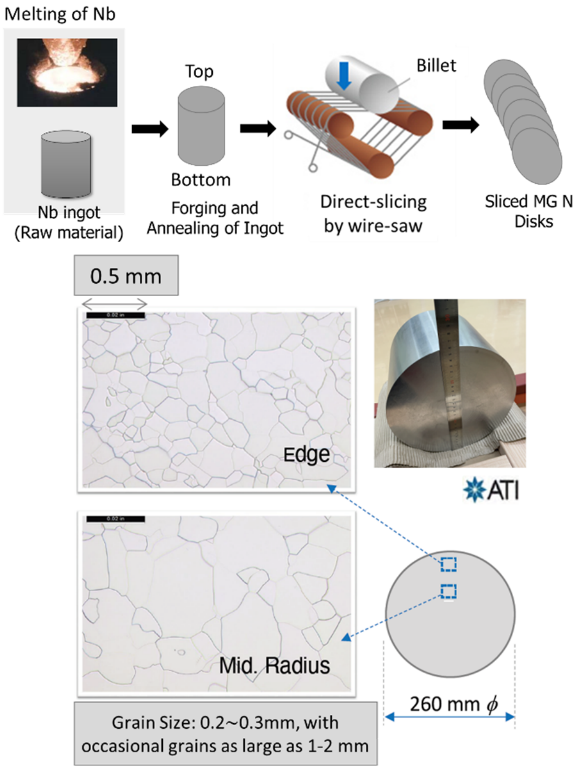

Figure 1: ATI MG Nb billet manufacturing process with direct slicing in disk form (up) and microscopic view of its grains (below) [10].

METHODOLOGY AT KEK

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, as shown in Fig. 2. 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 nominal strain rate of 4.4E-4 s^{-1}. Kyowa strain gages were bonded on the specimens to determine strain with respect to 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 the tensile test 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. In this cryostat three specimens can be tested for one cycle of cooldown. The obtained mechanical properties for MG Nb from the tensile tests are shown in the next section.

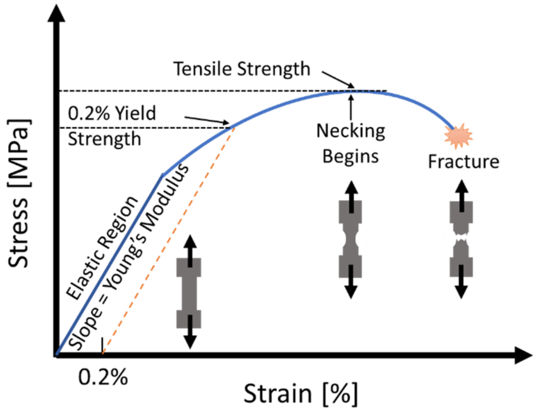

Figure 2: General stress-strain curve for metals.

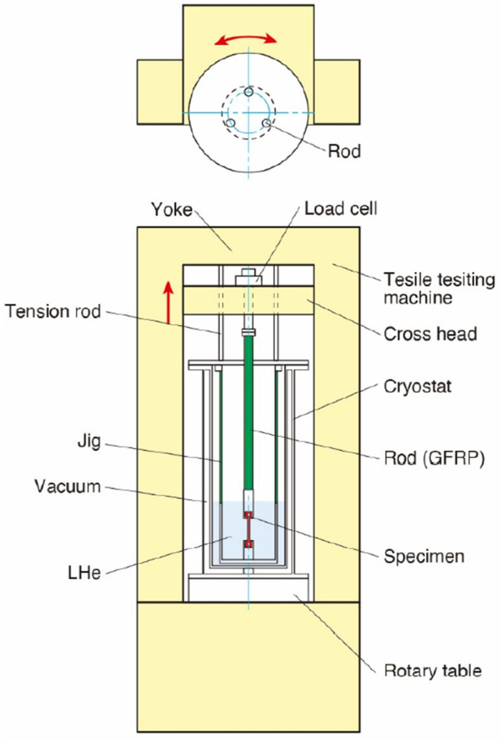

Figure 3: Schematic view of cryostat with tensile testing machine for liquid helium dipped testing [8].

RESULTS AND DISCUSSIONS

The mechanical properties of the material were determined from the top and bottom of the ATI MG Nb billet. The ATI MG Nb billet was received in forged and annealed condition and subsequently sliced into 65 disks, each 2.8 mm thick (t). From the directly sliced disks, two from the top (#1, #2) and two from the bottom (#64, #65) were used to conduct tensile tests, as shown in Fig. 4. The 0° mark is identified from the wire-saw cutting direction under microscopic view. 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. 5, 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 layout of all the specimens cut from the disks and their naming scheme can be seen in Fig. 6.

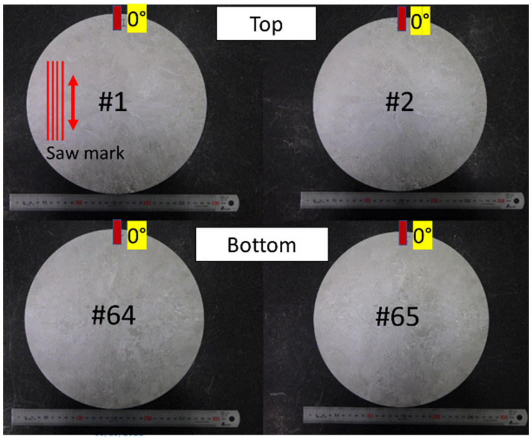

Figure 4: Sliced disks from top and bottom of the billet.

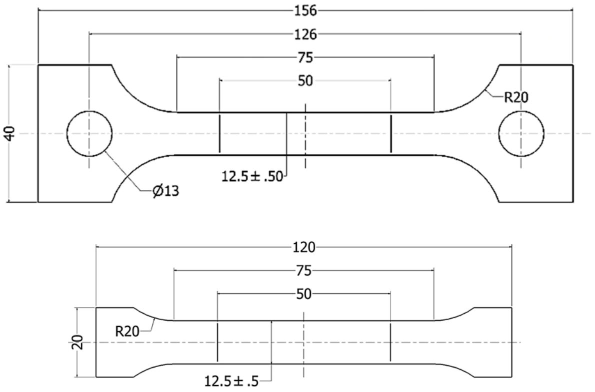

Figure 5: Specimen for tensile tests: Liquid helium or room temperature tests (up) and Room temperature specific tests (below) (JIS Z 2241 13B with t = 2.8 mm).

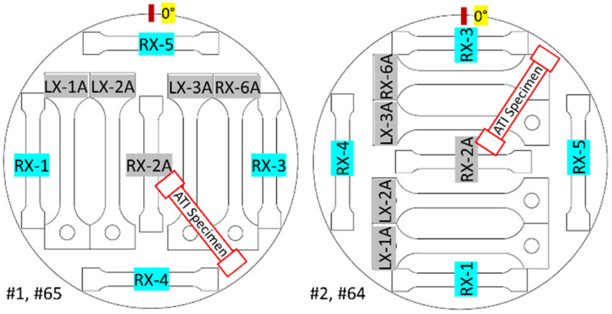

Figure 6: Specimen cut-out from the sliced disk from top and bottom two disks (R – room temperature, L – liquid helium temperature, X – Disk number, A – ASR).

Mechanical Properties at Room Temperature

The measured mechanical properties for the ATI MG Nb specimen from top and bottom of the billet are shown in Table 2, for Annealed and ASR specimens. The properties are uniform throughout the billet with minimal standard deviation between annealed specimens, as also seen in Fig. 7. However, the orientation of the annealed specimen was different than the ASR specimens. The annealed specimens are tangential on the outer radius, while the ASR samples are in a different orientation (locations 2A and 6A). The Y.S and T.S reduces by 15% and 7%, respectively, from the top to the bottom of the billet for ASR specimens. As expected, the annealed specimens showed a reduction in Y.S and T.S with respect to specimens in ASR condition, which was approximately 8% for T.S and 11% in Y.S (bottom), and maximum was 26% in Y.S (top). This behaviour has been observed for 800 °C annealing with FG Nb by W. Singer et al. and S.R. Agnew et al. [11, 12]. Stress-strain curves for some of the specimens are shown in Fig. 8.

The ATI provided data in Table 1 shows an elongation of 52% through the length of the billet for radial specimens, different from the results measured by KEK at around 18 – 27%. Possible reasons for this discrepancy include the specimen orientation, possible contamination or mechanical and thermal stress introduced from slicing. The ATI tensile specimens were tested in the radial directions as shown in Fig. 6. Although initially thought to be isotropic it is likely that the material is anisotropic. In addition, the grain size is non-homogeneous across the radius as can be seen in Figure 1. The grain size distribution and orientation may be different in the KEK tensile specimen and the ATI tensile specimen due to the specimen orientation. Future research paths could investigate grain orientation and preferential texture along the radius of the disc to characterize its impact on formability. It is well known that specimen contamination, i.e., interstitial pick-up during processing, can meaningfully decrease elongation in high purity Nb [13], possible sources including the multi-wire sawing operation, CP, wire EDM cutting and vacuum annealing. Post fabrication chemical analysis is needed to determine if this occurred. Finally, mechanical and thermal stresses induced by the multi-wire sawing can be additional sources for the discrepancy between KEK and ATI mechanical property data. Therefore, more investigation is necessary to determine the cause of such divergence. Although, this is not a major concern as T. Dohmae et al. were able to manufacture single cell MG Nb cavities [14].

Table 2: Mechanical Properties at Room Temperature

Position	**Y.S [MPa]**	**T.S [MPa]**	**Elongation [%]**	**E [GPa]**
		Annealed Specimen		
Top	$38.1^{\pm0.5}$	$122^{\pm5.7}$	$26.4^{\pm3.7}$	$83.9^{\pm8.2}$
Bottom	$37.9^{\pm1.4}$	$125^{\pm6.6}$	$22.5^{\pm3.9}$	$89.3^{\pm3.7}$
		ASR Specimen		
Top	$48.3^{\pm7.1}$	$151^{\pm9.4}$	$17.7^{\pm4}$	$86.1^{\pm4.7}$
Bottom	$41.1^{\pm2.7}$	$141^{\pm11.1}$	$22.9^{\pm5.6}$	$86.8^{\pm8.1}$

Number of Annealed specimens - 16 (8 top and 8 bottom)
Number of ASR specimens – 8 (4 top and 4 bottom)

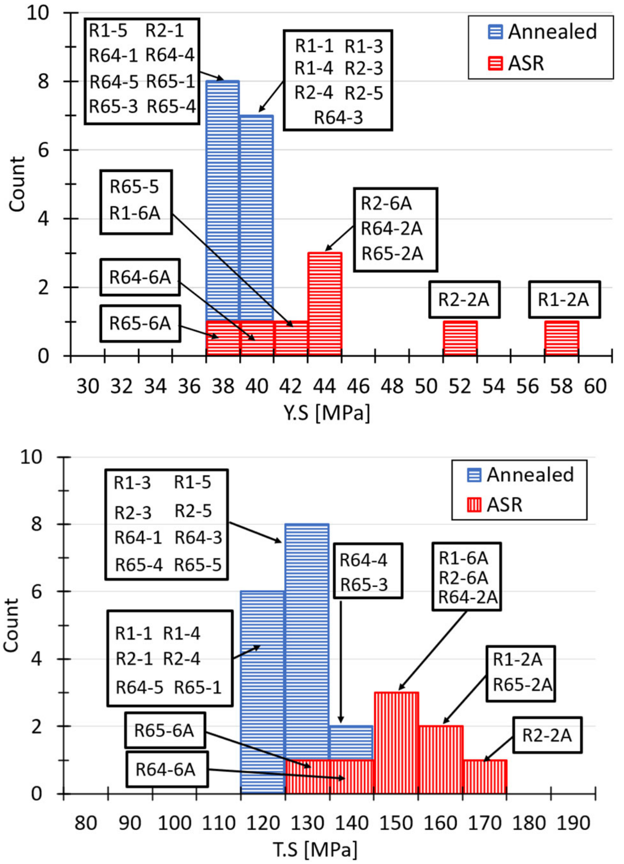

Figure 7: Frequency distribution of tested specimens for Y.S (up) and T.S (below).

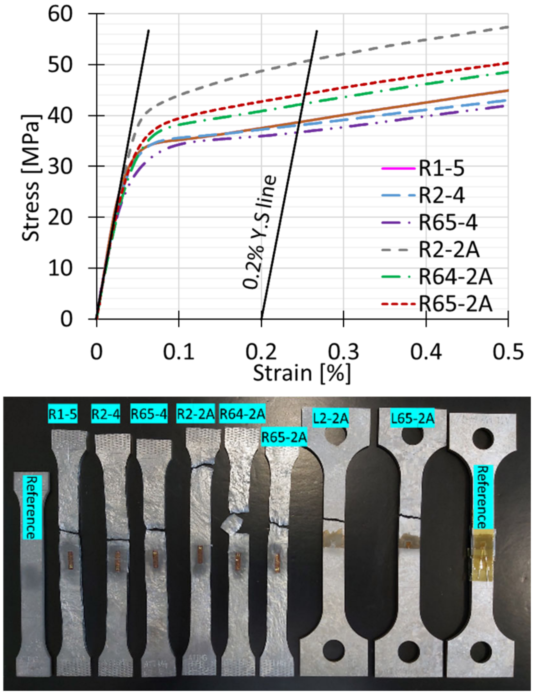

Figure 8: Stress-strain curves for some room temperature specimens (up) and their images after failure (below).

Mechanical Properties in Liquid Helium

The ASR MG Nb specimens from the top and bottom disks were tested in liquid helium. The specimens showed uniform tensile strength at cryogenic temperatures in the disks and throughout the billet, as summarized in Table 3. The standard deviation in the mechanical characteristics of the material for bottom specimens was lower than the top specimens. The elongation of the specimens was virtually non-existent, which might improve with annealing of specimens and will be considered soon. It was not possible to determine Y.S accurately due to the occurrence of serrations during yielding and most of the time the specimen or the strain gage failure occurred before the Y.S point could be reached, which usually occurs at around 0.5% strain. Images of some of the tested specimens can also be seen in Fig. 8.

Table 3: Mechanical Characteristics for ASR Specimens in Liquid Helium

Position	**Y.S [MPa]**	**T.S [MPa]**	**Elongation [%]**	**E [GPa]**
Top	-	$381^{\pm 92.7}$	$1.3^{\pm 0.1}$	$113.8^{\pm 19}$
Bottom	-	$375^{\pm 18.7}$	$2.5^{\pm 0.6}$	$114.4^{\pm 18}$

Number of ASR specimens – 6 (3 top and 3 bottom)

Comparison of MG Nb with FG and LG Nb

The MG Nb as shown in the previous section, has uniform mechanical properties throughout the length. In available literatures, it is well known that the FG Nb has isotropic properties and good formability (high elongation > 40%). 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 [7, 9]. From the obtained results and the data provided in the Fig. 9, the T.S and Y.S of the MG Nb material is closer to the FG Nb, rather than the LG Nb at room temperature. As discussed earlier, the elongation as measured by KEK is almost half of the FG Nb, while the ATI measured elongation is better than the FG Nb. The average T.S of the MG Nb is inferior to both FG and LG Nb in liquid helium, as seen in Table 4, with it being almost half of the FG Nb and almost 2/3rd of the LG Nb.

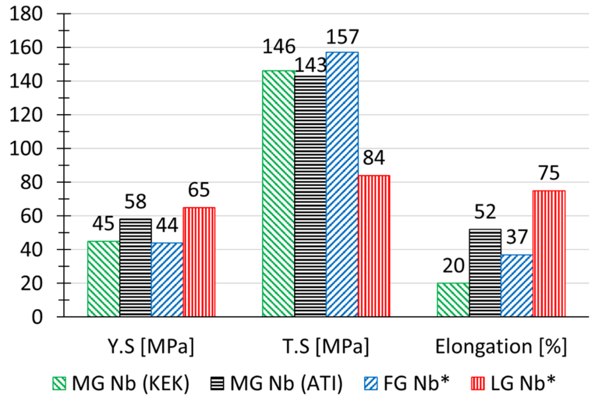

Figure 9: Comparison of mechanical properties of MG Nb with FG Nb and LG Nb at room temperature.

Table 4: Comparison of Mechanical Property of MG Nb with FG Nb and LG Nb in Liquid Helium

Material	**Y.S [MPa]**	**T.S [MPa]**	**Elongation [%]**
MG Nb	-	378	2
FG Nb*	516	832	7
LG Nb*	-	611	6

*Data is from the testing conducted at KEK on medium RRR Nb [9].

VIABILITY OF MG NB FOR 1.3 GHZ SRF CAVITY

The MG Nb mechanical properties are compared with criteria set by Eu-XFEL and KEK for SRF cavities at room temperature, as seen in Fig. 10. Nb is weakest in room temperature conditions and the mechanical strength specification is considered lower of either 2/3rd of Y.S or 1/4th of T.S, to be viable for the high-pressure safety regulation. From Fig. 10, it is noticeable that the T.S of the ATI MG Nb material is higher than the specification set for XFEL [15] and Tesla-like 1.3 GHz Nb SRF cavities by KEK [16]. For Tesla-like cavities, MG Nb clears both the Y.S and the T.S criteria. However, the material elongation is approximately 10% lower than the specification, which is set for formability of half cells and not for high pressure safety regulations. ATI's tensile test results shows the MG Nb mechanical properties clears all the necessary criteria for both facilities.

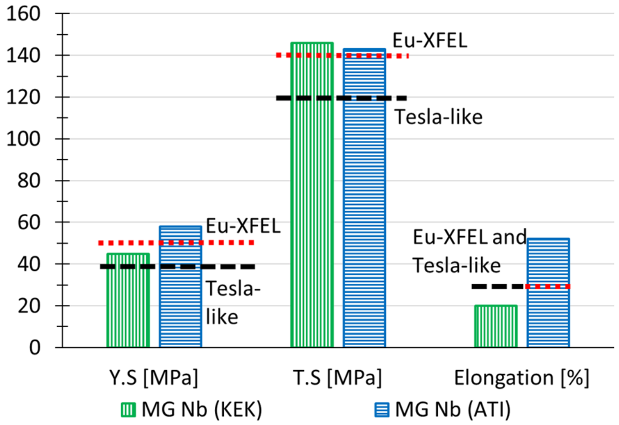

Figure 10: MG Nb room temperature properties with respect to some known Nb material property specifications.

CONCLUSION

The mechanical properties of the MG Nb have been measured and reported in this paper. Its mechanical properties are closer to FG Nb at room temperature and depends on the orientation of the tensile specimen. However, its mechanical properties being closer to FG Nb makes this material an excellent candidate to be considered as a cost-effective alternate. Moreover, the results presented in this paper suggests that the MG Nb has the mechanical strength to be accepted as a viable alternate to the FG Nb for 1.3 GHz cavity manufacturing. Of course, further studies are necessary to characterize the MG Nb mechanical properties at various annealing temperatures and to study the effect of direct slicing and other processes on its properties.

REFERENCES

[1] A. Yamamoto *et al.*, "Ingot Nb based SRF technology for the International Linear Collider", *AIP Conference Proceedings,* vol. 1687, p. 030005, 2015. `doi:10.1063/1.4935326`

[2] The International Linear Collider: A Global Project, `arXiv:1903.01629 [hep-ex]`

[3] P. Kneisel *et al.*, "Review of ingot niobium as a material for superconducting radiofrequency accelerating cavities", *Nucl. Instrum. Methods Phys. Res. Sect. A*, vol. 774, pp. 133–150, 2015. `doi:10.1016/j.nima.2014.11.083`

[4] M.G. Rao *et al.*, "Mechanical Properties of High RRR Niobium at Cryogenic Temperatures". In: *Advances in Cryogenic Engineering Materials*, ACRE, vol. 40, pp 1383-1390, Springer, 1994.

[5] H. Nakai et al., "Tensile test of materials at low temperatures for ILC Superconducting RF cavities and cryomodules", *Teion Kogaku (J. Cryo. Super. Soc. Japan)*, vol. 48, no. 8. 2013.

[6] W. Singer *et al.*, "Development of Large Grain Cavities", *Phys. Rev. Spec. Top. Accel. Beams*, vol. 16, p. 012003, 2013. `doi:10.1103/PhysRevSTAB.16.012003`

[7] Z Zhao et al., "RRR measurements and tensile tests of high purity large grain ingot niobium", *IOP Conf. Ser.: Mater. Sci. Eng.* 756 012002

[8] K. Enami *et al.*, "Tensile testing for niobium material in liquid helium", *Teion Kogaku* (2021), under submission.

[9] M. Yamanaka and K. Enami, "Tensile Tests of Large Grain Ingot Niobium at Liquid Helium Temperature", presented at the 2021 Int. Conf. on RF Superconductivity (SRF'21), virtual conference, July 2021, paper WEPFDV005, this conference.

[10] G. Myneni *et al.*, "Development of high-efficiency and cost-effective forged ingot niobium technology for science frontiers and accelerator applications", presented at SnowMass'21, AF-7 – Subgroup RF – miniWorkshop, August 2020. `https://snowmass21.org/docs/files/summaries/AF/SNOWMASS21-AF7_AF0_Ganapati_Myneni-087.pdf`

[11] W. Singer et al., "Influence of interstitials on properties of high purity niobium for RF cavities", *Matériaux & Techniques*, Vol. 91, N°7-8-9 (2003), pp. 38–44; `https://doi.org/10.1051/mattech/200391070013`

[12] S.R. Agnew et al., "Ultrasonic velocity and texture of high RRR niobium", *Matériaux & Techniques*, Vol. 91, N°7-8-9 (2003), pp. 19–22; `doi:10.1051/mattech/200391070038`

[13] G. Myneni et al., "Interstitial Solutes and Deformation in Nb and Nb Single Crystals", *AIP Conf. Proc.*, vol. 927, p. 60, 2007. `doi:10.1063/1.2770679`

[14] T. Dohmae *et al.*, "Fabrication of 1.3 GHz SRF cavities using medium grain niobium discs directly sliced from forged ingot", presented at the 2021 Int. Conf. on RF Superconductivity (SRF'21), virtual conference, July 2021, paper MOPCAV012, this conference.

[15] W. Singer, "SRF Cavity Fabrication and Materials", CAS - CERN Accelerator School: Course on Superconductivity for

Accelerators, Erice, Italy, pp. 171-207, Apr-May 2013. arXiv:1501.07142 [physics.acc-ph]; doi:10.5170/CERN-2014-005.171

[16] E. Kako *et al.*, “Cryomodule tests of four Tesla-like cavities in the superconducting RF test facility at KEK”, *Phys. Rev. Spec. Top. Accel. Beams*, vol. 13, p. 041002, 2010. doi:10.1103/PhysRevSTAB.13.041002