ISBN: 978-3-95450-233-2 ISSN: 2673-5504 doi:10.18429/JACoW-SRF2021-WEPFDV005

TENSILE TESTS OF LARGE GRAIN INGOT NIOBIUM AT LIQUID HELIUM TEMPERATURE

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Abstract

Tensile tests at liquid helium temperature were performed using specimen taken from high purity large grain niobium ingot produced by CBMM. The measured RRR is 242. The ingot is 260 mm in diameter and sliced by a multi wire saw to 2.8 thickness. 5 specimens were cut off from one sliced disk. 3 disks were set in same phase to obtain same grain distribution. 3 specimens each of 5 grain patterns 5, 15 in total were used for the tensile test. The tensile test stand using a cryostat and liquid He was manufactured by ourselves. The measured tensile strength varied 379 to 808 MPa. The average value is 611 MPa. The tensile strength at room temperature is 84 MPa. The strength becomes high at low temperature like a fine grain niobium. The specimen includes a grain boundary, and causes the variation of strength. The different result was obtained in same grain patterns. The relationship between crystal orientation and strength is discussed.

INTORODUCTION

The start material of high-purity niobium used for the superconducting cavity is an ingot manufactured by electron beam melting. It is a polycrystal with a grain size of 10 to 200 mm. The center cell part of the 1.3 GHz superconducting cavity shown in Fig. 1 is fabricated by press-forming a niobium sheet with a thickness of about 3 mm. A niobium sheet is usually produced by forging an ingot and rolling. The crystals are fined and the grain size becomes about 0.01 to 0.1 mm. This is called a fine grain (FG). On the other hand, there is a method in which a cylindrical ingot is sliced into a cell disk and press-formed to produce a cavity. Since the sliced disk contains large crystal grains, it is called a large grain (LG). The LG cavity has features such as a higher maximum accelerating gradient and Q value than the FG cavity [1-3]. Moreover, the slicing process is simpler than the forging / rolling process, and it is effective to reduce the secondary process cost of the low material.

The superconducting cavity is housed in a helium tank and cooled with liquid helium. These are subject to the High-Pressure Gas Safety Act. The tensile strength of the niobium material at the liquid helium temperature is required for it [4]. Therefore, the authors are conducting the tensile testing in liquid helium using a commercially tensile testing machine, a jig, and a cryostat as shown in Fig. 1 [5].

Although the tensile strength of LG niobium at room temperature has been reported [6], there is no report of measurement at liquid helium temperature. Therefore, tensile testing in liquid helium was performed using the LG niobium specimen having the same shape as the measurement result of FG niobium. The new experimental procedure and results are reported.

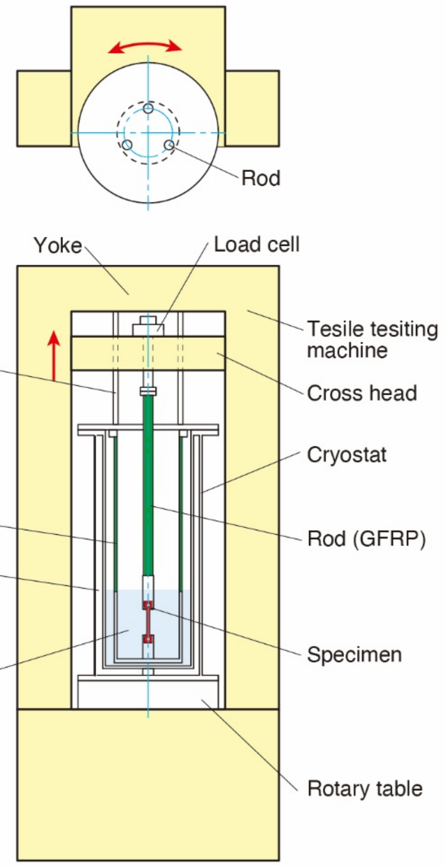

Figure 1: Schematic view of tensile testing machine with a cryostat (I.D. 317 mm) [5].

PREPARATION OF SPECIMEN

A niobium ingot with a diameter of 260 mm was sliced to a thickness of 2.8 mm using a multi-wire saw, and the JIS Z 2241 13B specimen shown in Fig. 2 was cut out by a wire EDM in the layout shown in Fig. 3. The specimens A to E were arranged so that the grain boundary was included in the center of each specimen. Using three disks with near slicing order, the grain pattern was set in the same order, and five specimens were prepared from each disk, for a total of 15 specimens. In addition, 14 13B specimens were cut out from one disk for the room temperature test. The shape of the grip part of the specimen for room temperature is different from that for the low-temperature test. The position of the grain boundary varies depending on each specimen. After cutting out, all the specimens were vacuum annealed at 800 ° C for 3 hours.

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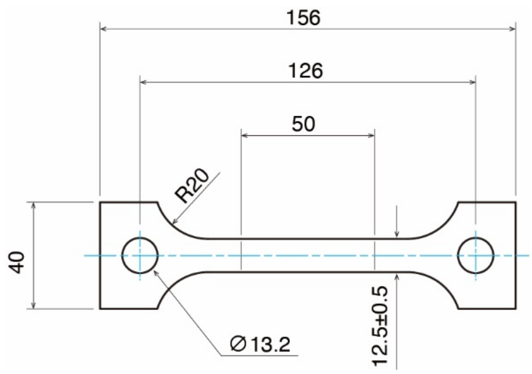

Figure 2: Tensile testing specimen (JIS Z 2241 13B) [5].

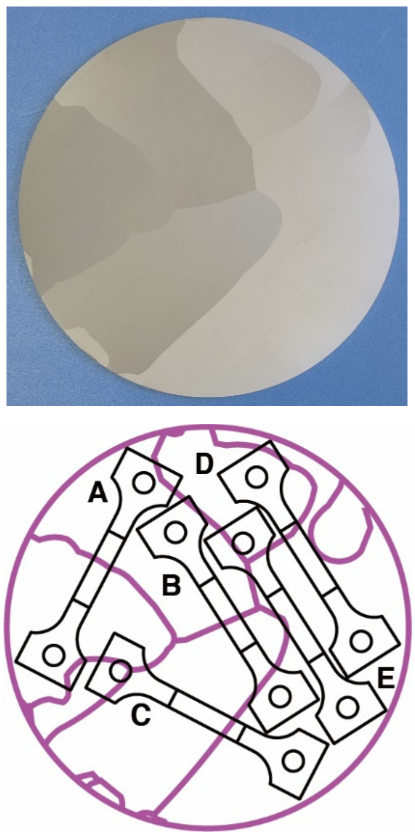

Figure 3: Layout of specimen in LG niobium disk (Dia. 260 mm, t 2.8 mm).

Table 1 shows the measured values of the chemical compositions and residual resistivity ratio (RRR) of the specimen. The manufacturer of the niobium ingot is CBMM, Brazil. It has a relatively high tantalum content and the RRR range is 200 to 300, which is called a medium RRR [7, 8].

Table 1: Chemical Compositions and RRR of Specimen

C	H	N	O	Ta	S	RRR
<30	<2	6	5	1191	<10	242

Unit of chemical compositions: wt ppm

TENSILE TESTING RESULTS

Room Temperature Test Results

The tensile testing was performed by attaching an extensometer to the gauge point. The tensile speed is constant at 2 mm/min for both room temperature (RT) and liquid helium temperature (LHeT) tests. The test results are shown in Table 2. The results of FG niobium are also shown for comparison [5]. The tensile strength of LG niobium at room temperature is about half that of FG niobium. On the other hand, the elongation is large, about twice that of FG niobium. It is known that the tensile strength differs depending on the crystal orientation, and it was expected that the variation would be large. The standard deviation ∇ is 3.2 (measured number $n = 6$), and the scatter is smaller than expected. A small test piece with a length of 40 mm was cut out so that it could fit in each crystal grain of LG niobium, and tensile testing at room temperature was performed as a single crystal specimen. As a result, the strength varied for each crystal grain, and the maximum value became 2.3 times the minimum value [9]. In this experiment, the maximum value is 1.1 times the minimum value. Although the specimen straddles multiple crystals, the strength depends on the weakest crystal orientation and is thought to vary as in the case of a single crystal. At this time, it is unclear why the variability is small. We would like to perform experiments with an increased number of tests and ingots for further study. Moreover, when the specimen after fracture was observed, none was broken at the grain boundary.

Table 2: Result of Tensile Testing for LG and FG Niobium Specimen

	LG RRR=242	**FG**[5] RRR=291	
RT	84 65 75 ∇ = 3.2 n = 6	157 44 37	Note Upper: Tensile strength [MPa] Middle: 0.2% proof strength [MPa] Lower: Elongation [%] ∇: Standard deviation n: Number of specimens
LHeT	611 – 6 ∇ = 132.4 n = 15	832 516 7	

Liquid Helium Temperature Test Results

The procedure for tensile testing in liquid helium conforms to JIS Z 2277. The extensometer cannot be used in liquid helium so that a strain gauge for low temperature was attached to the specimen to perform the tensile testing. An example of the test results is shown in Fig. 4 with a stress-strain curve. The strain was calculated from the movement of the crosshead of the testing machine and was shown until a breakage. A serration in which stress

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fluctuates finely is observed similar to FG niobium. The 0.2% proof strength is obtained by linearly approximating the elastic region, offsetting the straight line to the position of 0.2% strain, and calculating from the intersection with the stress curve. It is approximately 400 MPa from the figure. To obtain this accurately, it is calculated from the strain value obtained from the strain gauge (output value of the strain gauge amplifier) and the output value of the load cell. In the figure, the 0.2% proof strength value exists around 2.5% of the strain. The strain gauge for the low temperature used does not guarantee the strain limit at the liquid helium temperature, but in this experiment, the output became unstable from around 0.5% and the wire was broken at about 1%. Therefore, it is difficult to derive the 0.2% proof strength value from the strain gauge value. In addition, the serrations occur from around 2% of the strain, so the intersection with the stress curve also fluctuates greatly. Therefore, 0.2% proof stress is not calculated in this experiment. In other measurement results, the 0.2% proof stress was approximately half the tensile strength. Table 3 shows the results of all 15 tests. The average value is shown in Table 2.

The tensile strength of LG niobium at liquid helium temperature was about 70% of that of FG niobium, which was 7.3 times higher than that at room temperature. In the case of FG niobium, it is 5.3 times higher, so the LG niobium has more strength. The elongation is less than 1/10 of that at room temperature, which is almost equal to that of FG niobium. The tensile strength varies widely, and the standard deviation ▽ is 132.4 (measured number $n = 15$). It was much larger than at room temperature. As shown in

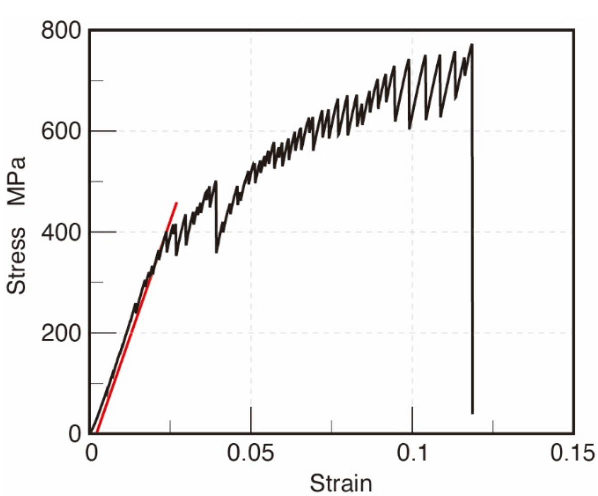

Figure 4: Example of measured stress-strain curve (position: E).

Table 3: Tensile Strength of LG Niobium Specimen at a Glance (Unit: MPa)

Position \ Disk	Disk 1	Disk 2	Disk 3
A	379	671	418
B	620	653	667
C	601	654	724
D	501	435	491
E	808	790	753

Table 3, the strength varies depending on the position in one disk. Comparing the values of different disks with the same specimen position, B, D, and E show similar values. A and C are scattered. The maximum value is 2.1 times the minimum value, which is close to the result of Bieler [9]. It is considered that the difference in tensile strength depends on the crystal orientation and appears remarkably at the liquid helium temperature.

Figure 5 shows an example of the specimens after a fracture. No specimens broke at the grain boundary as at room temperature. The figure shows the specimen at position B, all of them are broken from the same crystal grain. A twin deformation is observed near the fracture surface. This is a phenomenon seen in the low-temperature deformation of bcc-structured metals such as niobium.

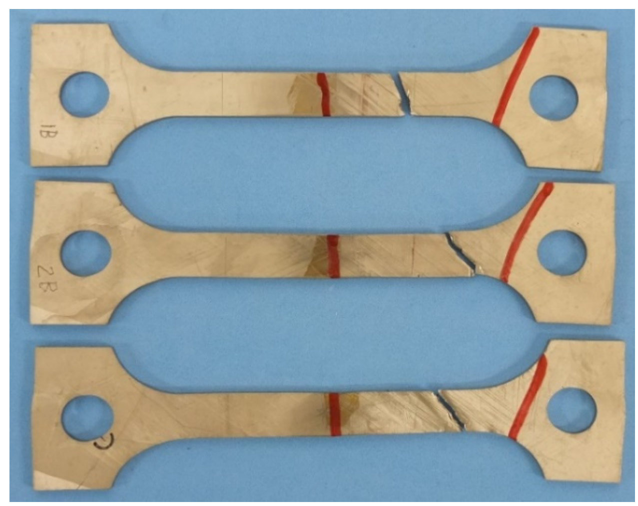

Figure 5: Example of LG niobium specimen after fracture in liquid helium (position: B) (Grain boundary is traced by red felt pen).

SUMMARY

The method for tensile testing of LG niobium in liquid helium was introduced. The tensile strength of LG niobium is 611 MPa, which is about 70% of that of FG niobium. The experimental results varied widely. The reason is that each crystal grain in the disk is large, the strength differs depending on the orientation, and the strength differs due to the crystal layout of the specimen. We would like to consider deeply from the viewpoint of material strength and examine the application of the LG cavity to the High-Pressure Gas Safety Act [10].

ACKNOWLEDGMENT

The niobium material used in this experiment was provided by CBMM, Brazil. Niobium slices have collaborated with Ganapati Myneni of Jefferson Lab, USA. Liquid helium supply/recovery and tensile tests were conducted at Cryogenics Science Center, KEK. The specimens were manufactured at the Mechanical Engineering Center, KEK. We would like to express our gratitude for these assistances and cooperation.

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