SINGLE CRYSTAL NIOBIUM DEVELOPMENT

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Abstract

Tokyo Denkai, KEK and TKX have developed a new niobium ingot slicing technique that allows slicing 150 pieces of large grain niobium discs in two days. Tokyo Denkai installed the slicing machine in December 2009. We inspected the sliced discs by the ultrasonic imaging and understood niobium crystal growth mechanism in Electron Beam Melting, which gave us an idea to make a single crystal niobium ingot. This paper describes the new high-speed production process of niobium discs, an idea of single crystal niobium ingot production and its pilot study result.

MULTI-WIRE SAW SLICING

The International Linear Collider (ILC) project will require 16,000 of a 1.3 GHz 9-cell cavity. Because one cavity needs 18 niobium sheets (or discs), a total of 288,000 niobium sheets must be produced in few years. Such a huge amount of high purity niobium disc production within a limited period might be a bottleneck, so we have to develop a much more speedy production process. Multi-wire saw slicing is a well-established technology to slice silicon wafers in semiconductor field. Tokyo Denkai, KEK and TKX applied this technology to slice niobium ingot [1]. This technology can slice 150 niobium discs of 2.8 mm thickness in 48 hours. Tokyo Denkai installed the machine in December 2009 (Fig. 1).

Figure 1: Installation of the multi-wire saw slicing machine.

ULTRASONIC IMAGING

Ultrasonic imaging is normally used to detect flaws in bulk materials. Measuring the intensity of the echo from the backside of a niobium sheet offers a kind of fluoroscope technology: the condition of a fine grain niobium's crystal texture, such as annealing or coarsening can be seen. Figure 2 compares ultrasonic images and optical microstructures on the same sample. In case of the large grain niobium, ultrasonic imaging gives us information of the crystal orientation of several grains. The crystal orientation observation by ultrasonic imaging has already been put to practical use on a single crystal of a superalloy for the blades of a gas turbine engine in a power plant [2].

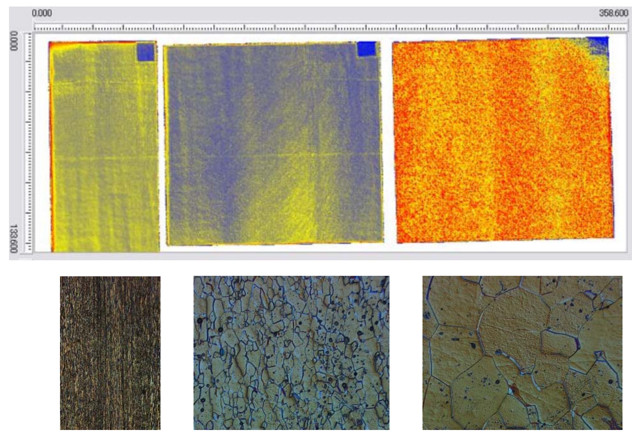

Figure 2: Comparison between ultrasonic images (upper) and optical microscopic image (lower).

ULTRASONIC TOMOGRAPHY

The combination of multi-wire saw slicing and ultrasonic imaging is a powerful tool for observing the crystal growth in a niobium ingot. In an Electron Beam Melting Furnace (EBMF), melted niobium drips into a molten pool and is cast continuously. As the feedstock melts, the ingot grows upper. The ultrasonic image of several discs from the bottom to the top of the ingot reveals the changing shape of crystals. Figure 3 shows the transition of crystals in a niobium ingot. In this figure, No. 1 is near the ingot bottom, No. 48 is near the top.

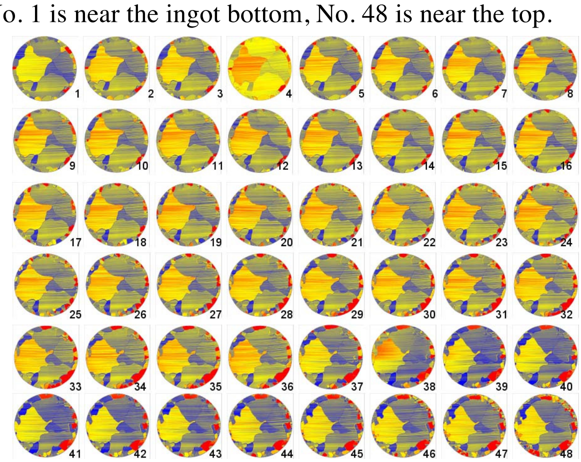

Figure 3: Ultrasonic tomography of a niobium ingot.

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SINGLE CRYSTAL NIOBIUM INGOT PRODUCT EXPERIMENT

As shown in Figure 3, the same crystal distribution appears from bottom to top of the ingot, which means crystals almost grow column-likely. The size and shape of a crystal strongly depend on the base plate. We have an idea that if there is a large single crystal piece or seed at the bottom of the ingot, it might grow column-likely up to the top. Thus, we could create a nearly single crystal ingot. We have pilot tested this idea.

Investigation of Grain Boundary Generation

Before producing a single crystal seed, we investigated the grain boundary generating mechanism. Four single crystal samples were arranged. Each sample was cut by electron discharge machine (EDM), cleaned by buffered chemical polishing (BCP), and welded again by electron beam welder (EBW). Figure 4 shows how to cut and weld together several samples. After welding, the samples were machined both sides by a lathe, and treated by BCP. The grain boundary was inspected by the naked eye. The result is shown in Table 1.

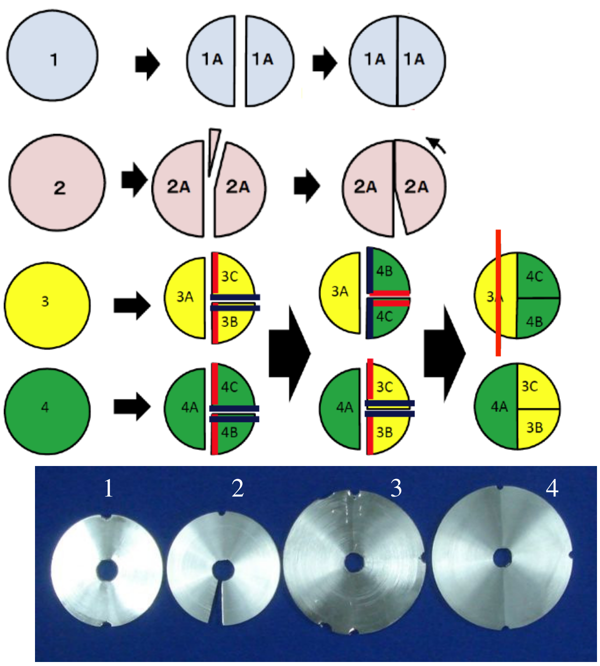


Figure 4: EBW combination of the sample pieces (upper) and machined and BCP'ed coin samples (bottom).

We hit an EBW beam on the 3A sample (red line). After the BCP, there was no grain boundary observed on the beam hit area as well as the jointed surface on 3A sample. A grain boundary appeared on the EBW jointed surface of 2A sample, where the crystal was rotated about 5 degree. In the No. 3 sample, the 4C quarter chip was rotated clockwise, and the 4B quarter chip was rotated counter-clockwise. This means that the welding plane was at 90 degree rotated between 4A and 4B. In this case a grain boundary appeared on every jointed surface. We learned the followings from these experiments:

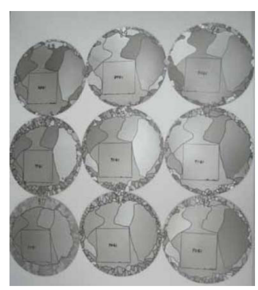
- The same crystal plane does not make a grain boundary by EBW (1A sample case).
- Welding between different crystals makes a grain boundary.
- Even for the same crystal, different angles make a grain boundary.

Table 1: Generation of grain boundary.

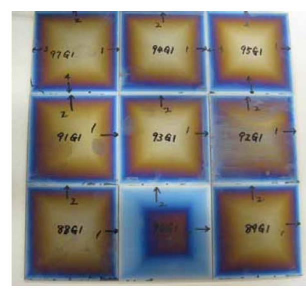
Sample	Welded side	Grain boundary	Bonding plane
No. 1	1A-1A	No	Same as before
No. 2	2A-2A	Yes	Rotated the grain
No. 3	3A-(4C, 4B) 4C-4B	Yes Yes	Different piece 90^{O} rotated the grain
No. 4	4A-(3C, 3B) 3C-3B	Yes No	Different piece Same as before

Seed Crystal

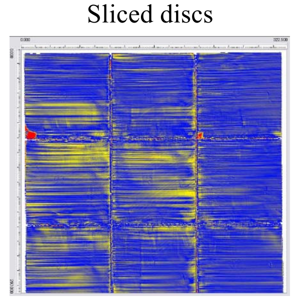
First, we produced a seed single crystal piece, as shown in Fig. 5. We cut a 105 mm × 105 mm × 2.8 mm single crystal piece by EDM from a 2.8 mm thick crystal of a sliced niobium disc. 9 single crystal pieces were cut from nearby 9 discs. These 9 pieces were welded together by EBW in KEK. We inspected the crystal orientation by ultrasonic imaging (see Fig. 5 left below). The welded crystal piece included two other crystals (red dot in Figure 5 left bottom). They came from the next crystal when the crystal was cut. It was cut into a round shape and BCP treated for a seed material of EBM.



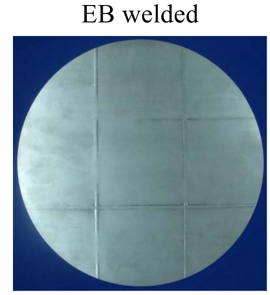
Sliced discs



EB welded



Ultrasonic imaging



Rounded and BCP treated

Figure 5: Preparation of seed crystal.

EB Melting

The ingot making was done by Tokyo Denaki's EBMF. It has a 1.8 MW Electron Beam power, and the diameter of the crucible is 300 mm. We just put the seed crystal piece on a niobium base plate (see Fig. 6 left) and melted the niobium ingot with Tokyo Denkai's normal procedure (see Fig. 6 right). At the first melting, the seed crystal curled just like a potato chip on the base plate. A feedstock of mother material was hung above. Droplets of melted niobium deposited on it.

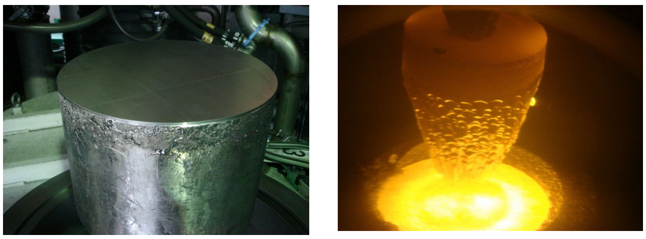


Figure 6: Seed crystal and inside of EBMF.

Ultrasonic Tomography

The niobium ingot was cut out 100 mm length around the seed material. It was adjusted so that the single crystal seed comes to the center. The ingot was peeled off the surface and multi-wire saw sliced. 30 discs, 2.8 mm thick having diameter 290 mm, were sliced.

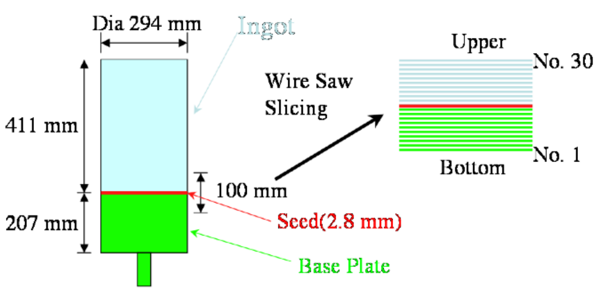


Figure 7: Wire saw slicing.

Figure 8 shows ultrasonic images of the niobium discs. In this figure, No. 16 corresponds to the position of the seed crystal, Nos. 1 to 15 came from the base plate and Nos. 17 to 30 from the new ingot.

Result

A single or large grain from the artificial single crystal piece (No.16) could not be obtained. The crystal shape and orientations vary continuously. In this case the crystal structure in whole ingot was determined by the niobium base plate. The artificial seed crystal did not work because the seed plate is too thin. As the electron beam hit, it melted out quickly, then lost the single crystal memory. Therefore, the thicker seed crystal is essential.

TENSILE TEST

Tensile tests of single crystal parts were done at room and liquid He (L-He) temperatures. The samples were cut from three directions in a large single crystal part by EDM. One was perpendicular to the wire marks by the multi-wire slicing. The others were parallel and diagonal to the marks. Test results are summarized in Table 2 (room temperature) and Table 3 (in L-He).

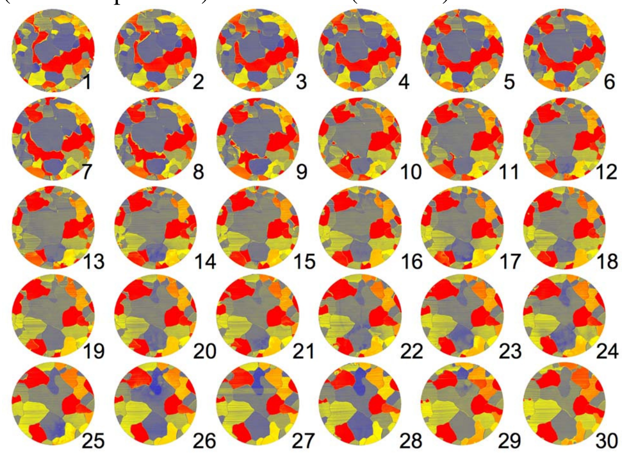


Figure 8: Ultrasonic tomography.

Table 2: Tensile Test Result at Room Temperature (293K)

Sample	0.2%Yield Strength [MPa]	Tensile Strength [MPa]	Young's Modulus [GPa]	Elonga tion [%]
Horizontal	50.0	86.2	46.9	51.6
Vertical	35.6	72.3	40.0	49.8
Diagonal	36.8	78.1	38.9	48.7

Table 3: Tensile Test Result at L-He Temperature (4.2 K)

Sample	Tensile Strength [MPa]	Young's Modulus [GPa]	Elongation [%]
Horizontal	971	90.1	5.0
Vertical	925	96.1	9.8
Diagonal	861	105	8.8

SUMMARY

Application of the multi-wire saw slicing to niobium ingot would be a key technology of mass production of niobium discs. Its short process time helps us to cross a hurdle of the ILC project. The tomography consisted of multi-wire saw slicing and ultrasonic image gave us an inspiration of niobium single crystal ingot. In the next experiment, we make more thick artificial single crystal seed. The details of the tensile test will be presented somewhere in near future.

REFERENCES

[1] K. Saito et al., "Multi-Wire Slicing of Large Grain Ingot Material", Proc. of the SRF Conference, 20-25 September 2009, Berlin, Germany, pp. 467-472.

[2] Toshiba Corp. R. Takaku et al. Crystal Orientation Measurement Device and Crystal Orientation Measurement Method. GO1N 29/00. 2008-03-13