

MULTI-WIRE SLICING OF LARGE GRAIN INGOT MATERIAL

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Abstract

The promising cavity performance has been confirmed on the niobium large grain cavity at many laboratories: Jlab, DESY, and IHEP/KEK in last 4 years R&D. The gradient over 35MV/m can be expected. The niobium materials for these cavity fabrications were sliced by EDM or combination of sawing and surface finishing, which are rather expensive and no way for massive production like ILC. We have developed multi-wire slicing method applying silicon wafer technology. This technology could produce 150 circular sheets in 50 hours with very smooth surface. So far we have demonstrated 102 sheets slicing in 50 hours. In this paper, we present the results and some cavity test result on these sliced materials.

LARGE GRAIN CAVITY

Large Grain Cavity

The cavity made of large grain niobium sheets is under development in many laboratories: Jlab, DESY, IHEP, KEK, and so on. Large grain cavity more likely has several advantages compared to polycrystalline cavity:

- 1) High gradient is expected by BCP, not EP,
- 2) High Q performance is expected due to the higher thermal conductivity by phonon peak.
- 3) Material cost should be lower than polycrystalline niobium sheet,

This material might be softer than polycrystalline material; however one could solve it by choosing a little thicker material. Very promising results have been reported on large grain single cell cavity R&D. Fig.1 shows typical results by P. Kneisel at Jlab [1]. This is on

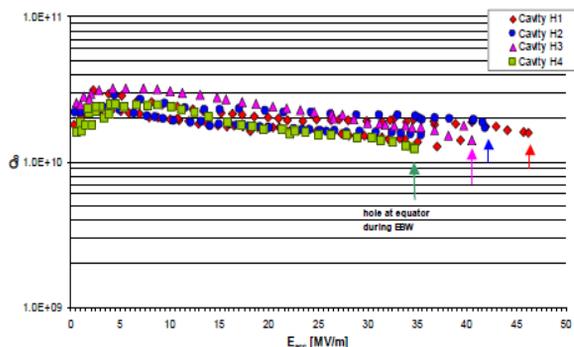


Figure 1: A typical cavity performance by large grain single cell cavity (Courtesy P. Kneisel).

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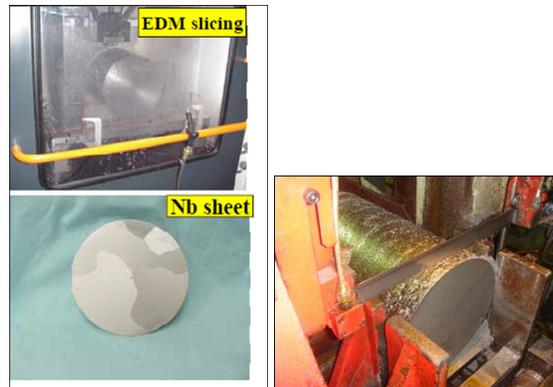


Figure 2: Current large grain sheet production. The left is the slicing technique by EDM slicing at Jlab, the right is mechanical sawing at Tokyo Denkai Co. Ltd.

LL shape single cell cavities treated by BCP. 40MV/m is promising except one result, which is for the cavity made a hole on the equator EBW seam and limited to 35MV/m. Large grain 9-cell cavity activities also are there in DESY, Jlab, IHEP, and KEK. So far the gradient of large grain 9-cell cavity is limited around 30MV/m. More information is gained in this workshop by W. Singer [2]. The strategy by the combination of large grain material, LL shape and BCP seems to be very promising.

Current Large Grain Niobium Sheet Productions

However, current large grain sheet production is problematic, which is time-consuming or very costly due to lots of material wastes. Fig.2 shows a slicing method by the electro-discharge machine (EDM), which Jlab is applying so far. It takes about 48 hours for one slicing. On the other hand, NingXia in China or Tokyo Denkai in Japan uses a more primitive method, which is a mechanical sawing (Fig.2 right) followed by mechanical grinding. In this process, one loses a lot of material, in addition needs surface finishing. To establish the large cavity technology, one issue is to develop a more efficient and inexpensive slicing method.

MULTI-WIRE SLICING

To date, silicon wafer slicing technology is advanced very much. They are routinely slicing 300φ 450L single crystal silicon ingots. This slicing machine is commercially available. The diameter size is just suitable for our niobium ingots, of which diameter is 270-265mm.

To apply this technology for the niobium ingots was proposed in the Single Crystal-Large Grain Niobium Technology Workshop in Brazil 2006 by one of the authors in this paper (K. Saito) [3].

Slicing Principle

The slicing principle is illustrated in Fig.3. A long piano wire of which diameter is 0.16mm is supplied from a wire drum. It is wound on the two rolls with multi-channels on the surface, of which pitch size depends on the requested material thickness. The rolls are fixed firmly on the slicing machine body. The wire is rotated back and forth, going ahead on the whole. An ingot is pressed on the moving multi-wire. Cooled liquid abrasives are sprayed on the wire. The friction between the ingot and the abrasives on the wire slices the material.

If successfully applied this method, one can slice directly a round niobium ingot and make directly many circular sheets at the same time. So far the commercial machine limits the ingot length by 450mm long. Supposed the thickness 2.8mm, one can slice 150 sheets from a 450mm long ingot. About 500 sheets are required every day for three years in ILC mass-production. Only 3-4 machines are enough for the production.

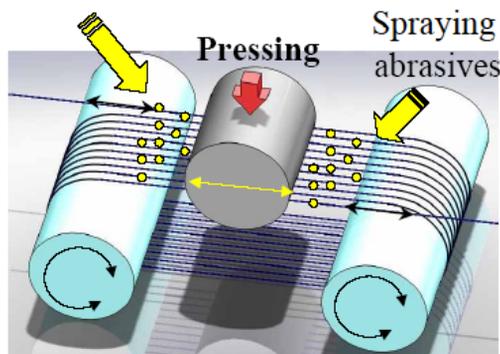


Figure 3: Slicing principle.

Try and Error

When discussed this idea with experts of the silicon wafer, they told us their critical opinions. The typical one was that niobium sheet will warp during slicing and the wire will be blocked. Anyway we started slicing test from small niobium samples. Really a disappointed result happened when we used diamond powder impregnated wire. Its slicing cost was estimated as 10k US dollars for one sheet. K.Nishimura (one of the authors in this paper) proposed us to use his company's slicing machine, where they have a lot of experience on silicon slicing for solar cells. They are routinely slicing 4 million sheets per year. By his help, we succeeded to slice a niobium bar with 150mm diameter. Since his machine is too busy to test 270mm diameter niobium ingot, he suggested us bringing niobium ingots to a slicing machine maker: Toyo Advance Technologies Co. Ltd.

First Slicing Test for 270mm Diameter Nb Ingot

We started slicing used 20mm thick Nb ingot with 270mm diameter at Toyo Advanced Technologies. We repeated twice this test to investigate the optimum slicing condition. Fig.4 shows the machine, which we used. One can see a niobium ingot at the centre of the machine. More zoomed picture is shown in Fig.5. One can see the multi-expanded piano wire at the bottom. The ingot is hung by a support, which presses the ingot on the wire. The cooled liquid abrasives are spray from the both sides. Fig.6 shows the successfully sliced niobium sheets with 2.8 mm thick. Totally 6 sheets were sliced from the 20mm thick ingot. In Fig. 6 one sheet is missing because it was sent to Tokyo Denkai. Slicing duration was 45 hours.



Figure 4: A slicing machine for silicon wafer, which we used for niobium ingot.

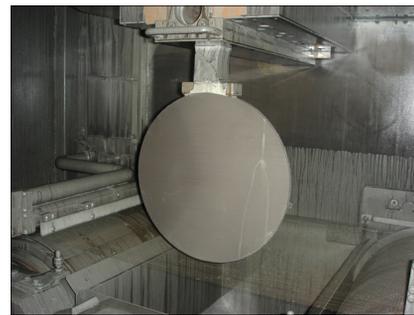


Figure 5: A zoomed picture around the niobium ingot.



Figure 6: Successfully sliced niobium sheets from Tokyo Denkai.

MATERIAL CONFIRMATION BY SINGLE CELL CAVITY

As obtained niobium sheets sliced by the silicon wafer machine, we checked first the material quality by fabricating a single cell cavity and cold testing. The cavity shape is Ichiro End cell shape. No big problem was happened in the deep drawing. The half cups are shown in Fig.7 as deep drawn. The edge around hole was cracked a little but it was not a problem for trimming. Fig.8 shows the fabricated LL end single cell cavity. We treated this cavity by our standard recipe, excepted for electropolishing (EP). EP was replaced by BCP. The deal of the recipe is:

CBP (80µm)+Light BCP (10µm)+Annealing (750°C for 3hours)+BCP (160µm)+HPR (55kg/cm² for 15min) +Baking (120°C for 48hours).

This cavity was tested after the treatment. The result is shown in Fig.9, which is excellent. The gradient higher than 42MV/m was achieved at the first test. Q-slope is observed at Eacc > 40MV/m but high Q₀ ~1x10¹⁰ is obtained at 40MV/m. It should be emphasized that the preparation is BCP, not EP. It was confirmed that this slicing method has no problem on the niobium material quality.

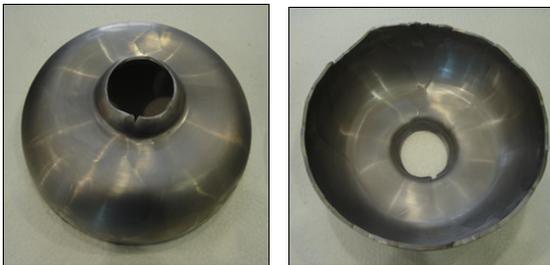


Figure 7: The half cups as deep drawn from the sliced sheets.



Figure 8: A LL end single cell cavity made of the sliced niobium sheets (1300MHz).

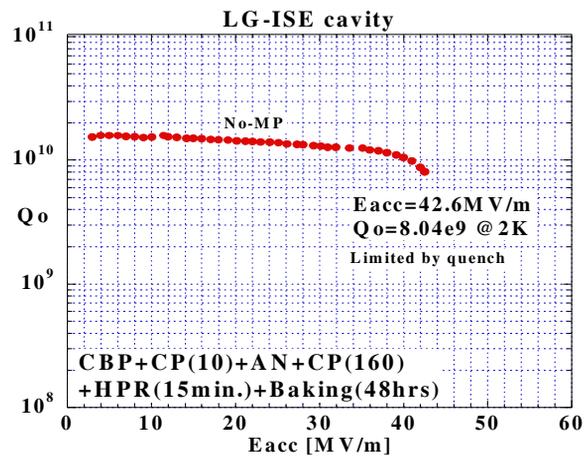


Figure 9: Cavity performance of the single cell cavity made of the sliced niobium sheets.

SLICING TESTS FOR MORE SHEETS PRODUCTION

61mm Long Ingot

As confirmed the niobium material quality by the single cell cavity test, we went ahead to establish more sheets production. At first, Tokyo Denkai prepared a high purity niobium ingot 61mm long and 270mm diameter. We succeeded to slice 19 sheets 2.8mm thick within 48 hours as shown in Fig.10 left. From these sheets we fabricated a LL 9-cell cavity as shown later.

201mm Long Ingot

We informed these results to P.Kneisel. He had interested in this method and ordered 20 sheets 3.2mm thick to Tokyo Denkai. We sliced a 201mm long ingot with 270mm diameter in this chance. We successfully sliced 58 sheets from this ingot within 48 hours. The picture is seen in Fig.10 right.

307mm Long Ingot

More recently KEK ordered a high purity niobium ingot 307 mm long with 265mm diameter to Tokyo Denkai. Using this ingot, we successfully sliced 102 sheets within 50hours as shown in Fig.11. At this stage the machine operators at Toyo Advanced Technologies tried slicing more carefully, which resulted in taking 50 hours for slicing.

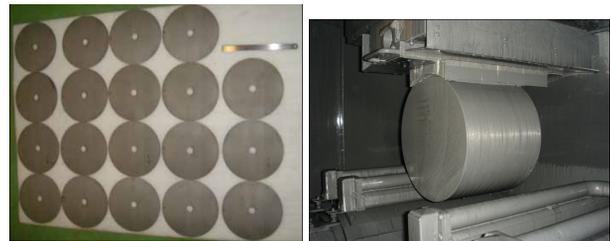


Figure 10: The sliced 19sheet 2.8mm thick from 61mm long niobium ingot (left), Just the finished slicing 58 sheets 3.2mm thick slicing for 201mm long ingot with 270 mm diameter.

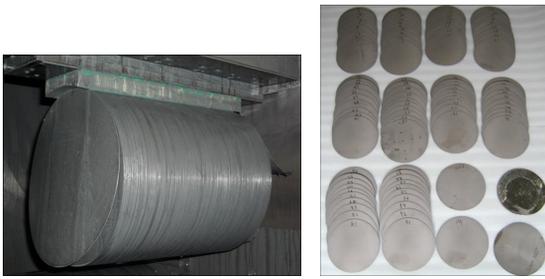


Figure 11: Sliced 102 sheets 2.8mm thick from a 307mm long ingot.

FEATURES OF MULTI-WIRE SLICING

Weight Variation with Individual Sheet

We investigated the detail of feature of these multi-wire sliced sheets on the 61mm long ingot slicing. 19 sheets were measured. Fig. 12 shows the variations with individual sheet. The standard deviation is 1.2g (0.09%) with the average weight 1387g. It shows very uniform between individual sheets.

Uniformity with Thickness in Each Sheet

We measured the thickness using an ultra-sound

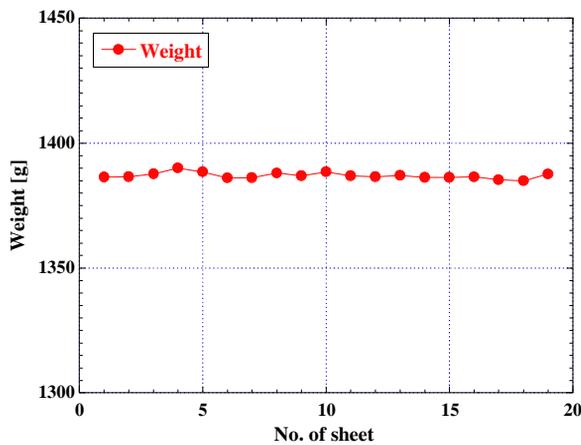


Figure 12: Weight variation with individual 19 sheets.

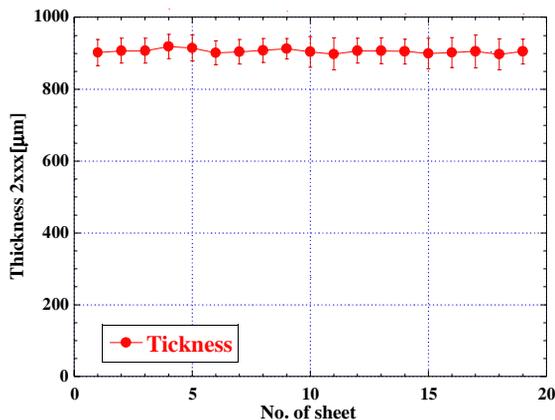


Figure 13: Uniformity with thickness in individual sheet.

thickness meter on 100 points in each sheet. Later we noticed that the sound velocity changes a little depended on the crystal orientation. Fig.13 contains such a measurement error but shows very uniform in the thickness too. The standard deviation was only 38µm on the average thickness 2.906mm.

To measure correctly the thickness uniformity, we used a micrometer on the 102 sheets sliced out from 307mm long ingot. The result is shown in Fig.14. The standard deviation was only 20µm (0.7%) on the average thickness 2.805mm. Very high uniformity has been confirmed on this slicing method.

Surface Roughness

Surface roughness (Rz) was measured on 4 points in each sheet. The result is presented in Fig.15. The average roughness was 4.9µm and the standard deviation was 1.8µm. The surface is smoother than 7µm. The sliced surface is smooth enough to go ahead deep drawing. We do not need any additional surface finishing.

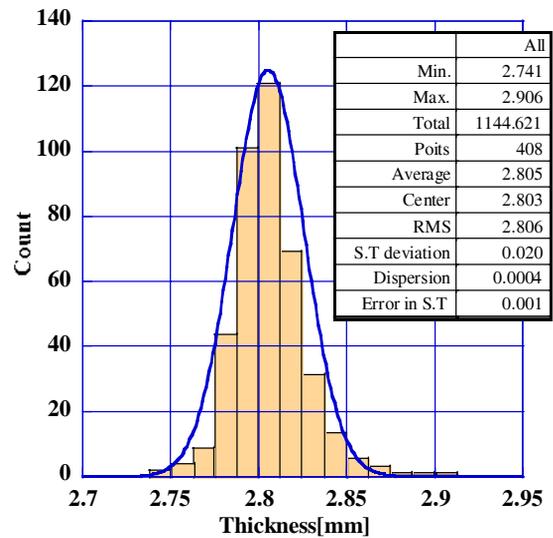


Figure 14: Uniformity with thickness in individual 102 sheets, measured by a micrometer.

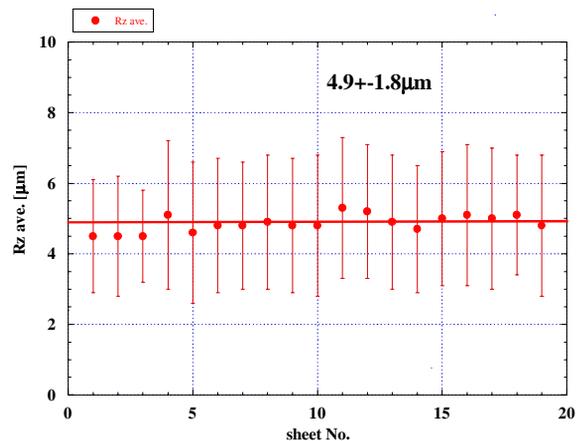


Figure 15: Surface roughness 19 individual sheets.

BENEFITS OF THIS SLICING METHOD

Very Much Simplified Sheet Production on Process

Fig.16 shows the current niobium sheet production for SRF cavities and unnecessary process when this slicing technology is applied, which is presented cross marks. Forging, rolling and on the way mechanical grinding, even annealing all become unnecessary. The process surprisingly becomes simple. Due to such the simplification, if ingot is ready, one could receive 150 sheets in one week. Currently it takes 1-2 months. Sheet production speed is very fast. If one prepares 3-4 machines, it is enough for ILC scale mass-production. So niobium material production is not a bottleneck for the ILC.

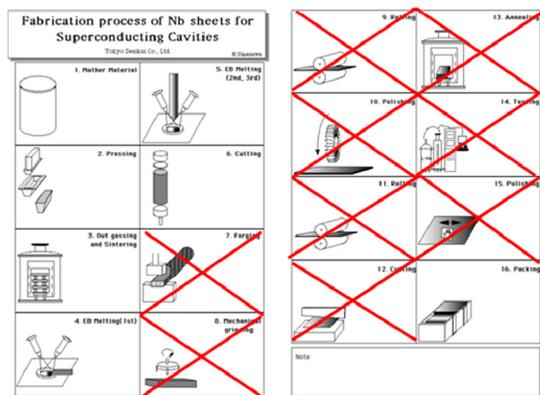


Figure 16: Current niobium sheet production process and the unnecessary processes in the multi-wire slicing.

Less Material Wastes

Fig.17 compares the material wastes during the niobium sheet production for SRF cavities. One can see that the current production wastes the material 45%. The current sheet production based on rolling favours square sheets. We prefer circular sheets for cavity. The four corners of the square are large material wastes, which occupy 27%.

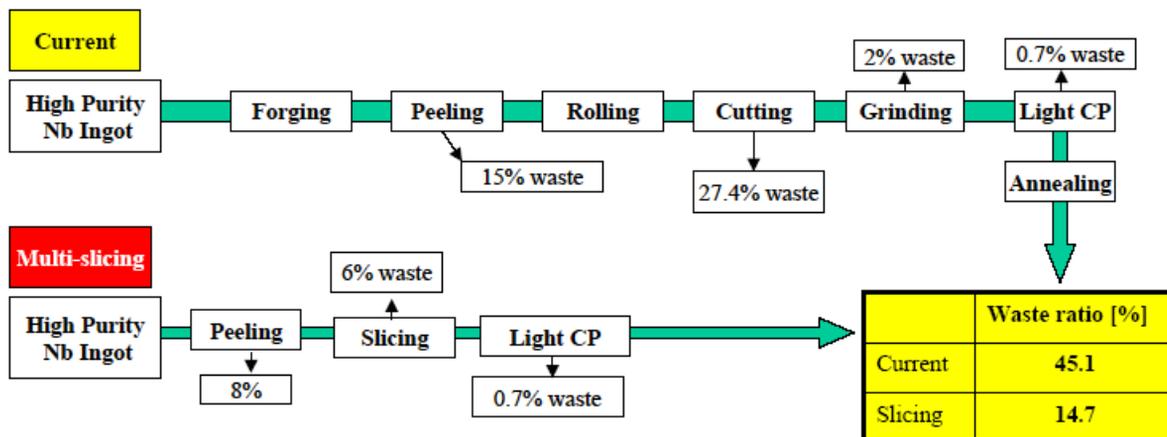


Figure 17: Estimated material wastes during the niobium sheet production.

On the other hand the multi-wire slicing starts circular ingot, therefore the material waste is less and becomes about 1/3 of the current.

Lower Material Cost

So far this slicing cost is not so cheap to be ignored, which is 100\$ or more per sheet. In the mass-production like ILC, we estimate the price could lower to 50-30\$ per sheet. The current sheet price is about 2-3 times of the ingot's price. Amount of the net material is a half of the original ingot because about the half is material wastes, so automatically the material price itself becomes double of the original ingot. The real price has to include the sheet production works. In the multi-wire slicing, the price is dominated by the original ingot. Future price of the sliced sheet, which is an issue to be negotiated with niobium firms, might be a half of the current price.

UNDER GOING LARGE GRAIN 9-CELL CAVITY TEST AT KEK

We fabricated a LL 9-cell cavity using the sliced niobium sheet materials mentioned above. Fig.18 shows the completed the cavity. This cavity is under testing to evaluate the performance at KEK. This cavity was mechanically ground by CBP about 180µm, and made light BCP (20µm). After that, it was annealed at 750°C for 3 hours, mechanically tuned to have uniform field flatness in individual cells (> 96%). Then we made BCP totally about 96µm. The cavity was rinsed by HPR. Slow evacuation and baking (120°C for 48hours) took place. The cavity test result is shown in Fig. 19. The accelerating gradient was limited 27MV/m by field emission.



Figure 18: A completed LL large Grain 9-cell cavity at KEK.

So far we have no BCP system for 9-cell cavities.

Putting ball valves on both end flanges of the cavity, we filled BCP acid about a half of the cavity, then rotated the cavity top and down, and opened top valve to exhaust generated NO₂ gases. The acid crossed much often at the centre, so material removal is double at the centre cell compared to end cell. The end-cells are not removed enough, that we suspect as the reason of the field limitation. We are preparing a better BCP system for 9-cell cavity. We hope it will start to operate in middle of November 2009.

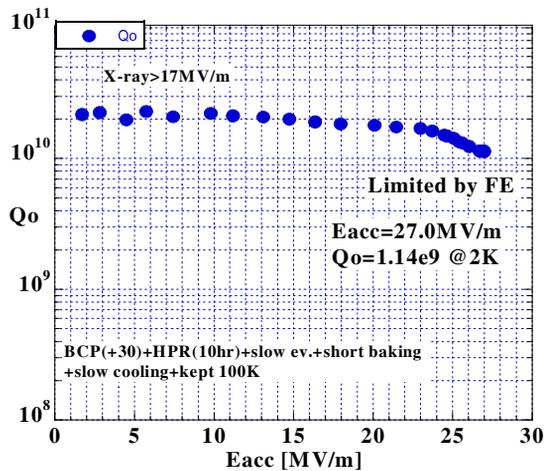


Figure 19: Test result of the LG 9-cell cavity at KEK.

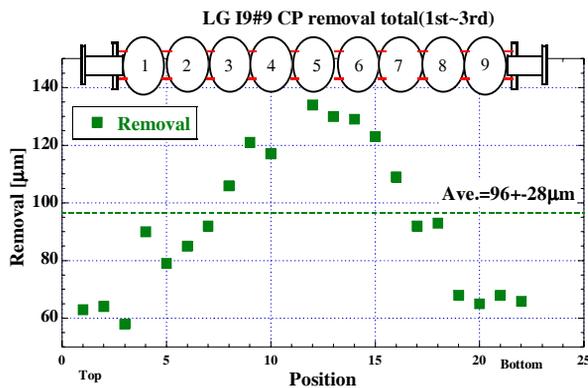


Figure 20: Uninformed material removal by the temporary BCP at KEK.

SUMMARY

In this paper, we reported that we have successfully developed the multi-wire slicing method for niobium ingots with 270mm diameter. The advantages of this method are cleared: unary uniform, very smooth surface. The niobium sheet production becomes very simple resulting in a large cost down in the material. The sliced material is also qualified by the single sell cavity test.

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