Large Grain Niobium Cavity R&D In Asia and the Future

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Abstract. The status of the large grain niobium cavity R&D in Asia and the future scope are presented. Recently KEK has received CBMM and NingXia large grain niobium sheets through collaborations. KEK has fabricated 1.3 GHz single cell cavities using these materials and measured the cavity performance. Those results are presented in this paper.

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INTRODUCTION

Application of large grain niobium to SRF cavity fabrication was started for two reasons two years ago: to push the current gradient limitation in particular on buffered chemical polished (BCP) polycrystalline niobium cavities, and a programmatic reason of cost effective niobium production for a large scale SRF application like ILC or EURO-XFEL. Currently it is a firm fact that BCP and Electropolishing (EP) both bring a Q-slope around $E_{acc} \sim 25$ MV/m if no baking is carried out during vacuum evacuation. Effect of the baking looks different between BCP and EP. In case of EP, Q-slope is perfectly eliminated and the gradient often reaches the critical RF limitation, typically 40 MV/m on TESLA shape or 50 MV/m on LL shape. In case of BCP, the Q-slope is recovered rather but not perfectly and the gradient is usually limited to a lower value than the critical field, typically 30 MV/m. BCP has a feature to produce sharp steps at grain boundaries due to chemical etching process. On the other hand, EP makes smooth surface even at the grain boundaries. The different baking effect suggests that grain boundary makes a harmful influence, such as magnetic field enhancement on the high gradient performance. Investigation of large grain/single crystal niobium could bring additional information on this subject.

The production process is rather simplified in large grain niobium sheets with a diameter < 300 mm. Niobium sheet (270-260 mm diameter) for 1.3 GHz cavity can be produced by directly slicing a large grain ingot. Forging and rolling processes in current production are eliminated. So far, several methods have been used for the ingot

slicing: electron discharge machining (EDM), mechanical sawing (MS), and wire sawing like silicon wafer production. However, EDM is time consuming and MS has lots of waste material. In the ILC project, we need ~500 sheets per day for three years cavity production. If a fast slicing machine is developed, a large cost reduction can be expected.

So far, JLAB and DESY are going ahead on large grain/single crystal cavity R&D. P.Kneisel and G.Rao have introduced this method and demonstrated very promising results on BCP cavities. KEK has used EP routinely for polycrystalline cavities and reached high gradient. Large grain cavity was not a critical issue to KEK except for lowering the cost. However, we have recently started an R&D program with the help of P.Kneisel at JLAB, USA and J.Gao at IHEP, China, to evaluate the advantages of EP on large grain cavity. We have fabricated and tested four 1.3 GHz single cell cavities. We present these results in this paper. It should be emphasized that KEK is still at very early stage on this R&D.

CURRENT R&D SCHEME OF THE LARGE GRAIN NIOBIUM CAVITY

At first, we make a brief review on large/single crystalline niobium cavity R&D over the world.

USA

As seen in Fig.1, which shows the R&D scheme on large grain/single crystal niobium cavity development, four niobium production firms can produce large grain ingot: CBMM in Brazil, Wah Chang in USA, WC Heraeus in Germany, and NingXia in China. P. Kneisel et al. have investigated CBMM, Wah Chang, and NingXia large grain materials on BCP and showed very promising results [1]. They have demonstrated a very narrow performance scatter between those three different materials. The scatter of maximum gradient is within 5% on 5 single cell cavities statistics. The average maximum gradient was 31.4 ± 1.6 MV/m with TESLA shape. This performance satisfies enough the EURO-XFEL specification.

Europe

W.Singer at DESY has developed large grain/single crystal niobium cavity using WC Heraeus material [2]. The cavities are processed with EP and BCP and they are tested in collaboration with JLAB. A single crystal cavity has been successfully fabricated from a single crystal niobium block, cut out from a very large grain ingot, rolled to a single crystal sheet. The cavity has been tested at JLAB by BCP and reached the critical field 43 MV/m with a flat Q_0 vs. E_{acc} curve. It was demonstrated that BCP can achieve the critical field without any Q-slope on the single crystalline material. DESY has tested large grain cavities processed by EP, and reached the average highest gradient of 34.3 ± 3.5 MV/m with statistics of six measurements. This is enough for the EURO-XFEL project. The gradient is close to the ILC specification but the scatter of 10% is still a problem for the ILC. After this test, they processed the

cavity with BCP and got a degradation of the high gradient performance, down to 28 MV/m. More statistics is needed to make conclusion, however, this result suggests that high gradient performance still could be limited by grain boundaries on BCP.

FIGURE 1. Scheme of Large Grain / Single Crystal Niobium Cavity R&D.

Asia

Figure 1 shows the current activities in Asia on the large grain niobium cavities. As shown above, NingXia large grain material has been recognized to reach high gradient ~ 30 MV/m with BCP. Therefore the China ILC group is eager to push this material as their important contribution to ILC R&D program. Dr. J.Gao at IHEP has set this issue as their ILC R&D program. Ge Minqi at IHEP is fabricating 5 single cell cavities using this material. They will test them in IHEP with BCP first, then they will send some of them to KEK, and test using EP.

As will be described later in detail, KEK has fabricated four large grain single cell cavities: one with CBMM material from P. Kneisel and three with NingXia material from J.Gao. We have compared the cavity performance between polycrystalline and these large grain cavities. KEK has a strong mechanical grinding method (Centrifugal Barrel Polishing: CBP) to remove grain boundary steps. The preparation with combined CBP and EP will give unique information about influence of the grain boundary steps on high gradient performance. Tokyo Denki is also preparing for large grain niobium ingot production. So far they have one suitable EBM furnace for niobium ingot production but the crucible is too small to produce ~ 300 mm dia. ingot. They are building two more EBM furnaces for tantalum production by June 2007, then they will use the spare high power EBM furnace for niobium ingot production, which can produce large ingot with ~ 350 mm diameter. They will start large grain ingot production in July 2007 [3].

KEK ACTIVITY ON LARGE GRAIN NIOBIUM CAVITY

CBMM Large Grain Cavity

CBMM Cavity Fabrication

KEK has received two CBMM large grain niobium sheets (RRR=300) from JLAB. Figure 2a shows one of them. These sheets were sliced from CBMM large grain ingot by EDM at JLAB. After cleaning these sheets by light BCP and ultrasonic water rinsing at KEK, we annealed them at 750 °C for 5 hr in order to release stress, which could be generated during the slicing. We made deep draw them with the same procedure as polycrystalline sheets. The cavity shape is the centre cell shape (I.L shape) of Ichiro 9-cell cavity. We did not repress during the deep drawing process, which is usually done at JLAB to correct inhomogeneous forming due to grain boundary slipping. As a reference cavity, another single cell cavity (IS#8) was fabricated in parallel using Tokyo Denkai (RRR=300) polycrystalline material. Figure 2b shows one half-cup from the CBMM large grain sheet after deep drawing. As seen in Fig. 2c, cracks appeared on the iris circle. The half-cups had locally thinner sections on the iris after trimming but could be barely used by a measure in electron beam welding (EBW) as seen later. The equator was wavy due to grain slipping (Fig. 2b). On the other hand, polycrystalline material has very homogeneous forming. After trimming, we measured the shape with a 3D machine. Table 1 shows the results. Large fluctuations from the design value, which is larger by nearly a factor 10 compared to the polycrystalline one, were observed in the large grain half-cups both at the iris and equator. We made EBW of the CBMM large grain cavity in KEK machinery centre. The equator was welded first from the inside with non-full penetration beam, then from the outside. After that a niobium ring 20 mm long was welded first on the iris by the same EBW method as at the equator (Fig. 2a). This is a measure to avoid EBW failure at the locally thinner locations on the iris (Fig. 2c). Figure 3b shows a picture of the equator EBW seam inside the CBMM cavity. At last, straight beam pipes with titanium flange 60 mm diameter and 130 mm long were welded on the cell. Figure 3c shows the completed CBMM large grain cavity.

TABLE 1. Comparison of circular variation between large grain and polycrystalline.

Equator Inside	+ Max (mm)	- Max (mm)
US No. 1	0.4730	0.4167
US No. 2	0.3866	0.2780
Fine 1	0.04044	0.02941
Fine 2	0.0707	0.05366
Iris Inside	+ Max (mm)	- Max (mm)
US No. 1	0.1801	0.08088
US No. 2	0.1801	0.1250
Fine 1	0.02805	0.02317
Fine 2	0.01563	0.01443

FIGURE 2. CBMM large grain Nb sheet (a) and after deep drawing (b). Cracks were present on the iris section.

FIGURE 3. Iris EBW (a), inside view of the equatorial EBW seam (b) and completed cavity (c).

CBMM Cavity Preparation

The cavity was processed with centrifugal barrel polishing (CBP) to remove defects on the cavity inner surface, especially on the equator EBW seam. We have to remove about 200 μm by CBP due to the deeper EBW under cut on the equator seam (see Fig. 3b). Figure 4a shows the equator EBW surface after CBP. Figure 4b shows the same surface cleaned by light BCP ($\sim 10 \mu\text{m}$) after CBP. The cavity was annealed at 750 $^{\circ}\text{C}$ for 3 hr to degas hydrogen picked up during CBP.

FIGURE 4. Equator EBW seam of CBMM cavity after CBP (a) and after light BCP (b).

After that, it was electropolished for 40 μm first, then successively removed 3 μm using fresh EP acid closed in the cavity: this procedure was developed recently to have high reliability of high cavity performance. Following this EP process, the cavity was

rinsed with high-pressure water (HPR) for 1 hr with ultra-pure water (TOC = 5 ppb, Bacteria = 2 pieces/cc), and dried for one night in the KEK class 10 cleanroom. After assembling, the cavity was vacuum evacuated and baked at 120 °C for 12 hr. The EP material removal and the baking period are different from the KEK standard recipe: EP (80 μm), baking (120 °C, 48 hr). The 12 hr baking period is an input from JLAB on large grain cavity on BCP large grain cavity. Large grain material might have much less surface defects than polycrystalline cavity. We have tried to reduce EP material removal in this study.

CBMM Cavity RF Test Results

The cavity RF parameters are summarized in Table 2. The temperature dependence of surface resistance is presented in Fig.5 for both cavities. The residual resistance is 1.49 nΩ on CBMM (USLG) cavity, which is very small as compared to that of the polycrystalline cavity (IS#8): 7.66 nΩ. As shown in Fig. 6, the gradient of CBMM (USLG) cavity was limited 36.5 MV/m by quench. On the other hand, it has reached 50 MV/m with Q-slope on the polycrystalline cavity (IS#8). We made additional baking on both cavities. The Q-slope in the polycrystalline cavity was eliminated after a total of 48 hr baking, but no effect happened on the CBMM cavity.

TABLE 2. RF Parameters of the CBMM cavity

RF Parameters	
Frequency (MHz)	1289.519
R/Q (Ω)	138.6
E_p/E_{acc}	2.08
H_p/E_{acc} [Oe/(MV/m)]	35.6
Γ (Ω)	281.8
$E_{acc}/(P_{loss}Q_0)^{1/2}$	100.3

FIGURE 5. T-dependence of surface resistance for the CBMM (USLG) cavity and polycrystalline cavity (IS#8).

FIGURE 6. RF performance of the CBMM (USLG) cavity and polycrystalline cavity (IS#8).

Ningxia Large Grain Cavity

Ningxia Cavity Fabrication

KEK has received six NingXia large grain niobium sheets with RRR~300 from J. Gao, IHEP in China. A representative sheet is shown in Fig.7a. The number of grain is 17 in the NingXia sheet and 10 in the CBMM sheet. The average grain size of the NingXia material (~ 6 cm) is 25% smaller than that of CBMM material (7.6 cm). We fabricated three Ichiro single cell cavities using these sheets. In the deep drawing process, we met the same cracking problem on the iris circle and wavy shape on the equator section. The fabrication error after the trimming is summarized in Table 3. The fabrication error (maximum fluctuation for inside and outside from design value) of half-cups is similar for both NingXia and CBMM materials. These numbers are quite large compared to the referenced polycrystalline material. Reduction of the big fabrication error would be a future R&D issue on the large grain cavity. EBW of these cavities is done by the same method as CBMM cavity. A typical EBW seam on equator section is shown in Fig.7b. After completion of the cavities, we removed about 200 μm by CBP. Such a heavy material removal is due to the deep EBW under cut on the equator seam or deep "triple tree" grain boundary slipping (see Fig.7b). The CBP final finished surface is shown in Fig.7c.

FIGURE 7. NingXia large grain Nb sheet (a), inside view of the equatorial EBW before (b) and after (c) CBP.

TABLE 3. Fabrication Error in NingXia half-cell cups.

Items	Cell No.	NX2	NX5	NX6	NX7	NX8	NX9	Average
3D arc inside	max	0	+0.25	+0.3	+0.3	+0.25	+0.3	+0.23
	min	-0.8	-0.6	-0.6	-0.5	-0.5	-0.5	-0.58
Equator Inside	max	+0.37	+0.25	+0.25	+0.25	+0.20	+0.20	+0.25
	min	-0.25	-0.18	-0.13	-0.13	-0.18	-0.20	-0.18
Equator Height	max	58.2	57.9	57.9	57.8	57.8	57.8	57.9
	min	57.9	57.5	57.7	57.5	57.6	57.6	57.6
Equator Surface	max	+0.3	+0.38	+0.23	+0.19	+0.15	+0.25	+0.25
	min	0	0	0	0	0	0	0
Equator Outside	max	+0.25	+0.35	+0.28	+0.15	+0.25	+0.28	+0.26
	min	-0.36	-0.45	-0.38	-0.25	-0.27	-0.38	-0.35
Iris Inside	max	+0.5	+0.45	+0.38	+0.43	+0.33	+0.30	+0.40
	min	-0.1	-0.13	-0.15	-0.13	-0.13	-0.15	-0.13
Iris Surface	max	+0.05	+0.06	+0.05	+0.07	+0.06	+0.06	+0.06
	min	0	0	0	0	0	0	0
Iris Outside	max	+0.08	+0.07	+0.09	+0.07	+0.1	+0.1	+0.09
	min	-0.34	-0.38	-0.35	-0.22	-0.43	-0.40	-0.35

Ningxia Cavity RF Test Results

The three NingXia cavities named as CLG#1, #2, and #3 were tested for the following issues: 1) use KEK standard recipe directly and see the high gradient cavity performance, investigate the shorter baking period for example 12 hr at 120 °C, 2) investigate material removal effect on cavity performance every 30 μm EP after CBP, 3) investigate EP material removal effect on every 30 μm without CBP.

The above issue 1) was investigated using CLG#1 and #2. These cavities were treated by the KEK current best recipe: CBP, light BCP (10 μm), annealing (750 °C, 3 hr), EP (80 μm) + EP (3 μm) with fresh acid, HPR (7 MPa, 1 hr), and bake (120 °C, 48 hr). In the first preparation for CLG#2, the 12 hr bake period was tested. The result is shown in Figure 8. The maximum gradient reached 43.8 MV/m but Q-slope appeared over than 40 MV/m, so we made an additional 12 hr bake subsequently to

the test. The maximum gradient did not change by this baking but the Q-slope was rather improved (Fig. 9, triangle symbols). We need further study but we judged that the 12 hr bake period is not enough for electropolished large grain cavity. As shown in Fig. 8, we baked directly for 48 hr the cavity CLG#1 and the maximum gradient of 47.9 MV/m was achieved with rather small Q-degradation, which was due to field emission. Considering those results, we have concluded that the bake period cannot be shortened with electropolished large grain cavity.

The gradients of all NingXia cavities are presented in Fig.8. The highest gradient CLG#1 has reached is 47.9 MV/m, after a total EP material removal of 99 μm in three steps. CLG#2 has reached 43.2 MV/m with one EP (80 μm +3 μm fresh acid, closed). CLG#3 has reached 13 MV/m with the first 33 μm EP, without CBP. The residual resistance of the NingXia large grain cavities was between 2.5 $\text{n}\Omega$ and 7.5 $\text{n}\Omega$. The cavity performance is very similar to the polycrystalline cavities. By recent study, the average gradient of polycrystalline cavities is 46.7 ± 1.9 MV/m and residual surface resistance of ~ 4 $\text{n}\Omega$ on the statistics of six cavities [4].

FIGURE 8. High gradient cavity performance using KEK standard recipe.

FIGURE 9. RF performance of cavity CLG#2 after baking for 12 hr and 24 h.

The EP material removal effect after CBP was investigated. CLG#1 was processed with CBP, which removed the surface about 200 μm at the equator region. After that, it was tested after each 33 μm EP removal step. In this study, we included the additional 3 μm EP using fresh EP acid after normal 30 μm EP. This process could avoid the risk of contamination after EP. Figure 10 shows the EP material removal effect on residual surface resistance. The residual surface resistance seems to saturate around 3 $\text{n}\Omega$ after 66 μm EP. Including other results on NingXia large grain materials, the residual resistance is between 2.5 $\text{n}\Omega$ and 7 $\text{n}\Omega$. Small residual surface resistance as for the CBMM cavity (1.46 $\text{n}\Omega$) was not observed for NingXia large grain cavities.

Figure 11 shows the EP material removal effect on the gradient. The gradient has been reached 47.9 MV/m after 99 μm EP. Field emission was the limit in every test. We need further study on this issue, however, a trend is observed that large grain material needs more material removal compared to polycrystalline cavity, if we remind polycrystalline cavity has reached 50 MV/m after 40 μm EP.

FIGURE 10. Improvement of surface resistance by additional EP.

FIGURE 11. Improvement of high gradient performance by additional EP.

FURTHER PROGRAM ON LARGE GRAIN MATERIAL IN JAPAN

Large Grain Ingot Production in Tokyo Denkai

As mentioned above, Tokyo Denkai will start large grain niobium ingot production from July 2007 [3]. The crucible of their current electron beam melting furnace for niobium has a 230mm diameter and no way to produce 300 mm diameter ingot.

Currently they are installing three more electron beam melting furnaces by July 2007 for tantalum production, which is their main business. When they will finish the installation, one furnace with 300 mm diameter crucible can be used for niobium ingot production.

Development of Wire Sawing Machine

The large grain niobium material is very promising as seen above. However, the current large grain niobium sheet is not so cheap due to ingot slicing method by EDM (time consuming) or mechanical sawing (lots of waste material). To make realistic the large grain niobium application in cavity production, the development of a high efficiency ingot-slicing method is necessary. In the ILC project, 500 sheets are required every day for three years production. We need a faster ingot slicing machine with less material waste. KEK has a program to develop a niobium ingot-slicing machine by collaboration with a silicon wafer company.

SUMMARY

KEK was behind from US or Europe on the large grain cavity R&D and recently started the R&D by the collaborations with JLAB and IHEP. We fabricated single cell cavities from CBMM and NingXia large grain niobium sheets and obtained very promising results. On the cavity fabrication, inhomogeneous forming was a problem and has to be solved. Cavity performance of the large grain cavity is similar to the polycrystalline cavity. If material cost is really reduced by this method, it is a good way for mass production such as ILC.

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