Experimental study on SC RF cavities by using China large grain niobium for ILC*

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Abstract Large grain niobium has the potential of simplifying the production sequence and consequently reducing the cost of the superconducting RF cavities for ILC. To investigate the feasibility of fabrication and the possibility to achieve high gradient by large grain cavities, two 1.3 GHz cavities were made of China large grain niobium and a series of vertical tests were carried out following several different surfaces treatment procedures. Two cavities have both reached the high gradient of more than 43 MV/m repeatedly and the maximum accelerating field of 47.9 MV/m has been achieved by China large grain niobium. This paper introduces the features of the fabrication and surface treatments on the large grain cavities and presents the preliminary results of the research.

Key words ILC, superconducting RF cavity, large grain niobium

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1 Introduction

In August 2004, it was announced in Beijing by the International Technology Recommendation Panel (ITRP) that the next generation positron and electron linear collider, named International Linear Collider (ILC)[1], would adopt superconducting accelerator technology. The conceptual design of the two main linear accelerators of ILC, one for electrons and another for positrons, is based on 9-cell 1.3 GHz superconducting radio frequency cavities with an accelerating gradient of $E_{\rm acc} \geqslant 35$ MV/m at a quality factor of $Q_0 \geqslant 5\times10^9$. Each accelerator consists of 8000 superconducting RF cavities made of niobium nestled within a series of cryomodules. The baseline material of SC RF cavities for ILC is the polycrystalline high purity niobium[2]. These material sheets are formed from a multiple electron beam melted ingot by an elaborate process of forging, annealing, rolling and chemical etching. The complex processes increase the cost and also the risk of foreign material inclusions. On the other hand, for many years it has been observed that the performance of SC RF cavities made of fine grain high purity niobium and treated by electro polishing has a rather scatter although the average gradient is increased by electro polishing[3, 4]. The reasons are not well understood and a prime candidate for causing the scatter is the “weak” grain boundaries in a niobium surface since the boundaries can easily be contaminated by segregated impurities and form “weak links” which lead to magnetic field enhancement. Also, till now it has been believed that uniform and fine grain material is needed for deep drawing of cavity half cells, and the standard recipes of the fabrication and surface treatments of the polycrystalline niobium cavities have come into being. Dedicated to investigating the possibility of fabricating the 1.3 GHz superconducting RF cavity

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using large grain niobium and achieving high gradient, two single-cell cavities (Fig. 1) of KEK ICHIRO shape made of China large grain niobium have been fabricated. After some surface treatments a series of vertically cryogenic tests have been carried out in the framework of ILC cooperation between IHEP and KEK. In this paper, our results are presented.

Fig. 1. Two 1.3 GHz cavities by using China large grain niobium.

2 China large grain niobium

The large grain niobium sheets are produced by OTIC, Ningxia, China. Standard procedures[5] convert the ore to niobium ingots after multiple electron beam melting of aluminothermically reduced niobium oxide. Sheets of 2.8 mm thickness are sliced from the ingots by saw machine. RRR, the most important value for SC RF cavities, is more than 300. Tantalum with a concentration of 50 ppm is the maximum metallic impurity. Among the interstitially dissolved impurities, oxygen whose content is 28 ppm in these sheets is dominant due to the high affinity of Nb for oxygen above 200 ℃. The size of the large grains in the sheets is not uniform. In the sheets to fabricate the large grain cavities, the average grain number on the surfaces is about 19. Even two sides of one sheet have different grain numbers. The average maximum grain radius in one sheet is about 85 mm and the minimum is 1.38 mm. The specification of the large grain niobium sheets is listed in Table 1.

3 Cavity fabrication

To investigate the maximum accelerating field that large grain cavities can achieve, KEK ICHIRO shape is adopted, which has a lower $H_{\mathrm{peak}}/E_{\mathrm{acc}}$. From the test results on the existing single-cell cavities, this shape promises higher accelerating gradients[4]. The half-cells were produced by one-step deep drawing. To prepare the deep drawing, subsequent to an annealing, a 10 μm surface layer was removed by chemical etching in the acid mixture of HF (48%), HNO_3 (60%), and H_3PO_4 (85%) in the volume ratio 1:1:1 for one minute. As the grain sizes of the large grain sheets were not uniform, tearing at the iris, and strong earing and grain steps at equator region occurred. After deep drawing, the half-cells were trimmed to the final size for electron-beam welding. Precise 3D geometrical measurements on the inner contour of all the half-cells, height, roundness at equator and iris region, were performed and the deviation values were one order of magnitude larger than those of the half-cells from polycrystalline niobium. To avoid making a hole or non-fusion caking at the region of high surface current, 40% welding from the inside (equator RF-side) was adopted firstly and then from outside with 10% overlap. Finally the beam pipe and flange assembly made from fine grain material was electron beam welded.

Table 1. Specification of China large grain niobium sheets.

impurity	ppm	properties	
Ta	50	RRR	>300
C	5	diameter of sheets/mm	270
O	5	grain number(max.)in one side	21
N	5	grain number(min.)in one side	14
H	2	grain number difference (max.) in one sheet	4
Si	<10	grain number difference (min.) in one sheet	0
Fe	<5	radius of the maximum grain/mm	99.6
W	28	radius of the minimum grain/mm	1.38
Mo	10	yield strength/MPa	50.6
Ti	<5	elongation at break (%)	53.2
Ni	<5		

4 Surface treatments

The cavities were both subjected to the same surface treatments which consisted of centrifugal barrel polishing (CBP), light chemical polishing (CP), annealing, electro polishing (EP), high pressure rinsing (HPR) and baking. The brief introductions of each process are the following.

4.1 Centrifugal barrel polishing (CBP)

After electron beam welding, the roughness and defects are very large especially at the region of equator and not uniform and it is necessary to apply mechanical polishing as pre-treatment before chemical or electro polishing. The centrifugal barrel polishing is proven as a very fast and economical method[6]. The cavity is loaded with special stones and water to revolute and rotate tumbled by a motor. The two large grain cavities were dealt with three 4-hour processes with the rough stones and another three with three other kinds of fine stones respectively. Both were totally removed about 90 μm averagely.

4.2 Light chemical polishing (CP)

By CBP, the inner surface becomes smooth but not enough for EP. Contamination during CBP should also be removed by light chemical polishing. The chemical acid is the same mixture for the niobium sheets. The operators on acid resistant clothes fill the acid into the cavity with one head closed and drain the acid by the end of half the total time needed to remove the thickness required, and rinse the cavity with ultra pure water to stop the reaction. Another head would suffer the same process. The removal thickness of light chemical polishing for our cavities is 10 μm. After acid etching, rinsing with the ultra pure water system for half an hour is immediately carried out to remove any chemical residue from the niobium surface.

4.3 Annealing

Annealing/degassing is performed at 750 ℃ for 3 hours in the vacuum furnace at a pressure of $10^{-3}\sim10^{-4}$ Pa, which serves to relieve mechanical stress of the cavity and remove the hydrogen in the material.

4.4 Electro polishing (EP)

EP is identified as an excellent technology for high gradient SC RF cavities by smooth surface finishing. The horizontally rotating continuous electro polishing method (HRC-EP) in Ref. [7] is used for the large grain cavities. The niobium is the positive electrode and the cathode is made of aluminum. The electrolyte is the mixture of HF (46%) and H_2SO_4(>93%) in a ratio of 1:10 by volume. The solution overflows from open mouths on the rotary sleeves, and retunes by gravity in a reservoir tank with a heat exchanger cooling. The acid pumping speed into cavity depends on the cavity surface area and is 3.8 L/min for the large grain cavities. The voltage is 25.0 volts and the solution temperature in the cavity is between 25 ℃ and 35 ℃. The thickness of removed niobium is estimated from the total current and is cross-checked by the content of Nb in the electrolyte. In the series of the tests, the removal thickness varied from 20 μm to 80 μm according to the programs proposed on the research. The finished roughness depends strongly on the initial roughness, the amount of the material removal and also the concentration of Nb in the electrolyte. 3 μm of thickness is usually removed with fresh electrolyte after a heavy removal more than 20 μm by EP. The additional light EP of 3 μm is effective to improve the cavity performance.

4.5 High-pressure rinsing (HPR)

Immediately after EP process, the cavity is placed in a closed loop of ultra pure water system for 1 hour. The specification of the water includes the resistance of more than 18 MΩ/cm, the total organic carbon (TOC) of 10—20 ppb and the bacterial count of less than 4 per liter. To eliminate the particles larger than 0.3 μm, a filter is equipped before the nozzle from which ultra-pure water spurts to remove any chemical and particulate residues from the niobium surface.

4.6 Assembly and baking

With ultra pure water inside the cavity, the valve attached to the cavity is closed and the cavity is brought into the dust-free clean room (Class 10) where the water is drained. The newly cleaned surface, still wet from ultra pure water, is exposed only to the filtered air in a Class 10 clean room. The laboratory workers in the vicinity need to wear special particulate-free clothing and follow strict protocols to reduce particulate generation. In the clean room, the wet cavity is assembled with an RF input-coupler and pick-up antenna on ends of cavity, respectively, with the indium wire seals used as vacuum joints between the cavity flanges and the auxiliary components. In the vacuum stand, the cavity is dried via heating while pumping by the turbo molecular and mechanical pumping system. The vacuum leaking of flange connections is strictly checked before in-situ low-temperature baking. During baking, the optimum temperature ranges from 115 to 135 ℃ and the vacuum of 10^{-5}—10^{-6} Pa is obtained by ion pump.

5 RF tests

The cavity equipped with input and pickup antennas is transferred to the test cryostat. Mounted in the cryostat, the cavity is first cooled with liquid helium at 4.2 K. Subsequent RF tests consist of measuring the surface resistance from 4.2 K to 2.0 K and the dependence of the Q-value on the accelerating gradient (Q_0 vs $E_{\rm acc}$) at 2 K.

5.1 Temperature dependence of surface resistance

Cooling down to 2 K is achieved via pumping down to the pressure of 3.0×10^3 Pa. During pumping down, the temperature dependence of the surface resistance in the range from 4.2 K down to 2.0 K is measured. Fig. 2 is an example of an experimentally obtained $R(T)$ dependence of a large grain cavity which is the summation of BCS resistance and residual resistance R_0[8]. The curve fitting according to Ref. [9] results in the residual resistance of $3.05\times10^{-9}\Omega$. R_0 can arise from several sources, such as particles, foreign material inclusions and condensed gas. Successive reprocessing of the same cavity may show variations in R_0. Fig. 3 is the distribution of the residual resistance values of 6 tests and in Table 2 which summarizes the tests the values of R_0 are listed. Compared with the cavities made of the polycrystalline niobium, the large grain cavities have a lower residual surface resistance which is attributed to less impurities and smoother surface of the large grain material.

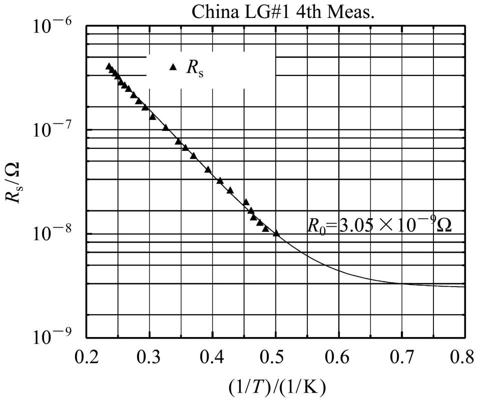

Fig. 2. Measurement of the temperature dependence of the surface resistance.

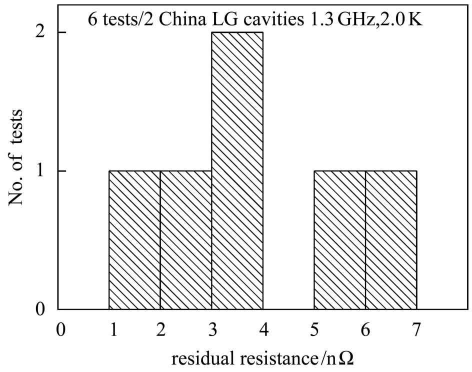

Fig. 3. Statistical distribution of residual surface resistance for 6 tests on two single-cell, 1.3 GHz, large grain cavities prepared by electro polishing.

5.2 Performance of China large grain cavity #1

China LG #1 was polished 30 μm of thickness per time by EP and then was vertically tested to figure out the influence of EP removal thickness on the accelerating gradient.

Figure 4 and 5 are the excitation curves of the Q-value dependence on accelerating gradient (Q_0 vs $E_{\rm acc}$) of the cavity tests. In the test after EP removal thickness was only 30 μm, during the first power rise the initial field emission signal was caught at the field gradient of 14—15 MV/m. Multipacting, another phenomenon related to free electrons, was seen at the field level between 20—24 MV/m and took 4 minutes to progress in the test. After processing out, the starting field of field emission did not change but at the gradient of more than 15 MV/m, the input power increased was almost entirely reflected with a drop of Q-value and a sharp rise of X-signal. X-ray reached to 2.48 mSv/h at the accelerating field of 24.02 MV/m and limited the test. The Q-drop also demonstrated that the baking of 12 hours was presumably insufficient. For the second test the baking of 48 hours was conducted after EP of 30 μm. The Q-value was higher than the previous test and X-ray signal and multipacting started at the same fields. But multipacting could not be surmounted by processing and when raising the RF power to 24 MV/m, X-ray was triggered by a damage on the surface so that lowering the power did not recover the high Q. When the removal thickness by EP reached 90 μm, the performance of the cavity was evidently improved. Between 20 MV/m and 27 MV/m a processing of 9 minutes worked well to overcome the soft barriers of multipacting. The maximum accelerating gradient reached 40.76 MV/m at Q_0 of 9.81×10^9 and was limited by quench with high X-signal. As some particles or defects on the surface were suspected to answer for field

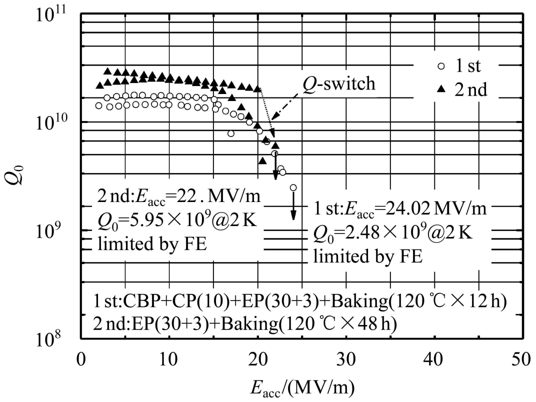

Fig. 4. Excitation curves of the first and second tests of China LG #1.

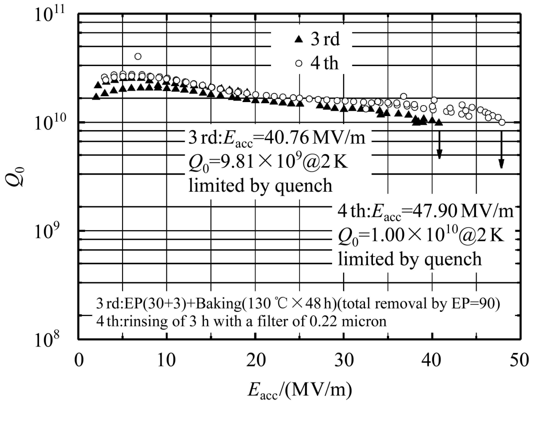

Fig. 5. Excitation curves of the third and fourth tests of China LG #1.

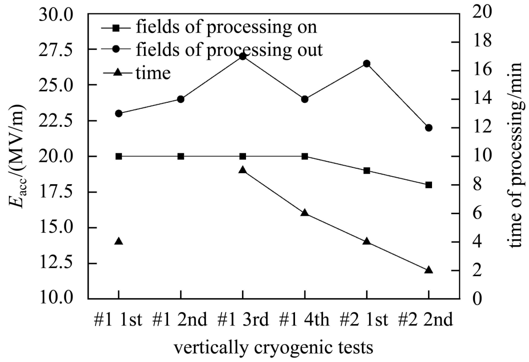

Fig. 6. The field levels and time of multipacting of 6 tests of China LG cavities.

emission, the cavity was rinsed for 3 hours after the replacement of a filter of 0.22 μm. In the fourth test the power could be increased to the gradient of 47.90 MV/m at Q_0 of $1.0{\times}10^{10}$. The field levels and time of multipacting processing are summarized in Fig. 6.

5.3 Performance of China large grain cavity #2

China LG #2 is dedicated to demonstrating the high gradient of China large grain niobium material by a removal thickness of 80 μm once and it is identified to be enough for polycrystalline cavity to reach a high gradient of 40—45 MV/m. After baking at 120 ℃ for a short time of 12 hours, the cavity reached a gradient of 43.8 MV/m, limited by Q-drop with a weak signal of X-ray. The frequency over the accelerating gradient shifted at the high field level so quickly that the resonance was easy to lose. The degradation of the quality factor at high gradient was cured by an additional baking of 12 hours. The second test achieved a high Q-value of $7.2{\times}10^9$ at 43.2 MV/m as shown in Fig. 7. In the two tests the cavity was not severely hampered as multipacting barriers were well processed in a short time as shown in Fig. 6.

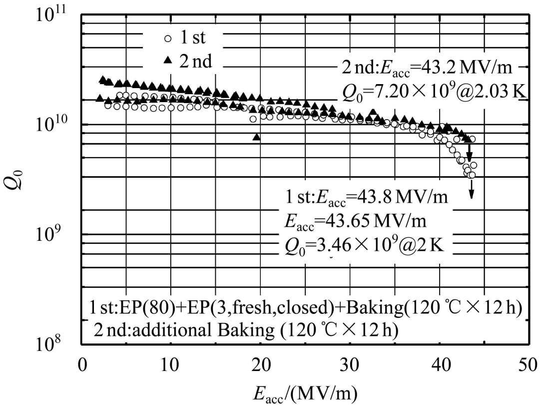

Fig. 7. Excitation curves of the first and second tests of China LG #2.

6 Summary

Large grain niobium is a promising prospect for SC RF cavities of ILC. The material is less expensive because of the elimination of the sheet fabrication and has a higher purity and a smoother surface due to fewer grain boundaries. Although the non-uniform characteristics of mechanics brought some complexities to the fabrication, such as deep-drawing and electron beam welding, the manufacture was completed successfully. The standard surface treatments which are effective for polycrystalline material are still suitable for the large grain material. When 90 μm by CBP, 10 μm by CP and more than 80 μm by EP for the two cavities were removed, the cavities both reached the high gradient of above 43 MV/m and the maximum was 47.9 MV/m with a quality factor of $1.0{\times}10^{10}$, which is the highest accelerating gradient by China large grain niobium so far. Table 2 summarizes the results of the tests of the two large grain cavities.

Table 2. Summary of the tests of China large grain niobium cavites.

V.T.	EP+Baking+HPR	$E_{acc,max}$/(MV/m)	R_{res}/(nΩ)	limitation
#1 1st	EP(30+3)+Baking(120 ℃×12 h)	24.02	6.08	field emission
#1 2nd	EP(30+3)+Baking(120 ℃×48 h)	22	3.15	field emission
#1 3rd	EP(30+3)+Baking(130 ℃×48 h)	40.76	5.79	quench
#1 4th	HPR+Flashing(3 h)	47.9	3.05	quench
#2 1st	EP(80+3)+Baking(120 ℃×12 h)	43.8	1.95	quench
#2 2nd	Additional Baking(120 ℃×12 h)	43.2	2.65	quench

References

1 GAO Jie. HEP & NP, 2006, **30**(Supp.I): 156 (in Chinese)
2 http://www.linearcollider.org/files/WGGG/materials_wg5_peter_kneisel.pdf, ILC report.
3 Saito K. Proc. of the 2003 Particle Accelerator Conference. 2003. 462—466
4 Furuta F, Saito K et al. Proc. of EPAC 2006. 750—752
5 Salles Moura H R. Melting and Purification of Niobium, Proc. Intern. Symposium Niobium 2001, Orlando, FL, Dec. 2001. 147
6 Higuchi et al. http://conference.kek.jp/SRF2001. PR022
7 Saito K. Proc. of LINAC 2002, http://epaper.kek.jp/l02/PAPERS/WE104.PDF
8 Hasan Padamsee et al. RF Superconductivity for Accelerators. New York: A Wiley-Interscience Publication, 1998. 171—178
9 Thomas P. Wangler Principles of RF Linear Accelerators. Los Alamos: A Wiley-Interscience Publication, 1998. 365