# **MEASUREMENT OF PARTICULATES UNDER SLOW PUMPING AFTER HIGH PRESSURE RINSING OF SUPERCONDUCTING CAVITY BY USING MODIFIED SLOW PUMPING SYSTEM**

Hiroshi Sakai† , Kensei Umemori, Eiji Kako, Ryo Katayama High Energy Accelerator Research Organization (KEK), Tsukuba, Japan

#### *Abstract*

Slow pumping system was used for particle free vacuum pumping in Superconducting rf accelerator. In KEK, recently slow pumping system was developed for the cryomodule assembly work with STF 9-cell cavities and worked well to reduce the particulates movements under pumping. However, this slow pumping system also wants to be used for preparation of vertical test. Before assembly work in clean room for vertical test, we normally apply high pressure rinsing (HPR). There are many waters on the cavity surface after HPR. Therefore, we keep one night to dry inside cavity in clean room. Unfortunately, some waters still remain in the cavity even though we keep drying in clean room for one night. This water might make some icing under pumping and stop flow in mass flow meter, which is used for slow pumping to control the flow. Therefore, we modified the slow pumping system to be robust under slow pumping even when water exists in the cavity. In this paper, we present the modified slow pumping system in KEK and the results of the vacuum trend through slow pumping of 9-cell superconducting cavity. We also measured the particulates under slow pumping after HPR by using vacuum particle monitor.

## **INTRODUCTION OF SLOW PUMPING & VENTING SYSTEM AT KEK**

In general, the dusts or particulates in the superconducting (SC) cavity can produce field emission from the inner surface of cavity, and generate large amounts of radiation and cause the degradation of SC cavity performance. For examples, STF-2 cryomodules in KEK, which was constructed to establish the fundamental technology for Superconducting RF cavities with beam operation for the ILC [1], met the heavy field emission after cryomodule assembly work in 2014 [2]. During cavity string assembly in a local clean booth at STF tunnel, the sudden venting was done when the gate valve opened. We did not control the pumping and venting speed and we understood that it is necessary to establish a pumping system that does not allow contamination of dusts. Slow pumping is very effective to suppress the particulates moving under pumping and venting and recently used for cryomodule assembly on big SC accelerators [3]. This situation led us to develop new slow pumping and venting system in KEK at this time.

Figure 1 shows the schematic diagram of slow pumping and venting system of our first prototype [4]. The pumping system is similar to the pumping system which was used

**458**

on EURO-XFEL construction [3]. The vacuum particle monitor [5] was equipped to measure the particulates under pumping and venting in our system. Furthermore, we controlled to slowly move all gate valves. This is necessary not to produce particulate by moving the gate valve [6]. Slow pumping and venting speed were controlled by mass flow meter. Under slow pumping, the mass flow meter controlled the flow via the bypass line with small pipe. When the pressure reached to less than 100 Pa, the pumping line changed to the main pumping line with large diameter of 40 mm to obtain sufficient conductance and finally the cavity vacuum was pumped by Turbo Molecular Pump (TMP). Nitrogen gas was used for slow venting and the diffusers were set on the slow venting line. The pressure was measured by an ion crystal gauge. Dynamic range of the vacuum particle monitor is from  $0.3 \mu m$  to  $3.6 \mu m$  of particle size. The slow pumping speed is typically 0.6 l/min and venting speed is 0.2 l/min. No particulate moving was observed during slow pumping and venting with the above conditions [6]. We could suppress the particle movements by using this prototype.



Figure 1: Schematic diagram of slow pumping & venting system of our first prototype with 9 cell cavity and valve assign (red).

After developing the first prototype slow pumping and venting system, we built slower pumping and venting systems that all valves were controlled automatically to slowly move the gate valve and its configuration was changed as shown in Fig. 2, for STF-2 cryomodule re-assembly work. Thanks to these slow pumping and venting system, we could keep the good cavity performance after STF cryomodule re-assembly work and satisfy the stable beam operation of 33 MV/m in STF-2 cryomodule [7]. Our developed slow pumping and venting system in KEK was also recently applied to other SRF laboratory in Japan [8].

> **SRF Technology Assembly/integration**

<sup>†</sup> hiroshi.sakai.phys@kek.jp

#### **MODIFIED SLOW PUMPING SYSTEM**

Next, this slow pumping system will be used for preparation of vertical test. Before assembly work in clean room for vertical test, we normally apply HPR. There were many waters in the cavity. Flow control was basically conducted by the mass flow meter in our slow pumping system and flow was controlled by very small valves in mass flowmeter of "FCS series" in ref. [9]. However, this flow meter cannot be used under water condition because flow in the mass flow line will be stopped when the water was iced during pumping. In order to escape the icing, we modified the pumping control not by the mass flow meter but by the pressure controlled one, which named as UPCUS [9]. This UPCUS is normally used to control the pressure difference  $(\Delta P)$  between the entrance and the exit of the UPCUS precisely and could use for various gas control.



Figure 2: Detailed block diagram of modified slow pumping & venting system. Valves were assigned as "V plus number".

Figure 2 shows the block diagram of modified slow pumping and venting system for the preparation of vertical test. Slow pumping line shown in orange line in Fig. 2 is controlled by UPCUS. Slow venting line shown in pink line in Fig. 2 is controlled by mass flow meter, which is the same as our previously developed system [1]. When the vacuum is decreased to 100 Pa under slow pumping, pumping line is changed to main pumping line shown in red line in Fig. 2. The vacuum particle monitor is equipped to measure the particulates under pumping and venting.

UPCUS do not control mass flow  $( \Delta S)$  but pressure difference ( $\Delta P$ ).  $\Delta S$  [l/min] is proportional to the  $\Delta P/\Delta t$  [Pa/s] x V [l], where V is the volume of the superconducting cavity and  $\Delta t$  is the elapsed time. In our case,  $\Delta S$  will be indirectly controlled by controlling the constant  $\Delta P/\Delta t$  under slow pumping. It is noted that V is about 30 l for 9-cell cavity and it took 1 hour to reach 100 Pa on the first test with 0.6 l/min slow pumping [6]. It is possible to keep same speed by using UPCUS.

In detail, UPCUS control range is from 100 kPa to 1.5 kPa. For the first test, we set constant pressure difference from 100 kPa to 1.5 kPa for 1 hour to keep almost same flow ratio of 0.6 l/min. In order to continue slow pumping, the UPCUS is kept as full open condition up to

### **SRF Technology Assembly/integration**

100 Pa. After that, we switch slow pumping to main pumping. The capacitance gauge (CG) mainly measures the vacuum pressure under slow pumping. The ion gauge (IG) mainly measures the vacuum pressure after changing main pumping.

### **MESUREMENT OF PARTICULATES AFTER HPR UNDER SLOW PUMING**

We check whether this modified slow pumping system works for 9-cell cavity. We also check whether this system will work after HPR. We tried slow pumping three times as follows:

- 1. 9-cell cavity slow pumping without HPR
- 2. 9-cell cavity slow pumping with HPR
- 3. 9-cell cavity second slow pumping without disassembly of the cavity after the procedure of B)

#### *Slow Pumping without HPR*



Figure 3: (Left) Assembly work of 9-cell cavity to the modified slow pumping system without HPR. (Right) setup of the slow pumping system with 9-cell cavity.

Figure 3 shows the setup of first trial of this modified slow pumping system. A 9-cell cavity, which was already used for the vertical test, was used for the study. The cavity was carefully reassembled in class 10 clean room, as shown in the left figure of Fig. 3, and connected to the modified slow pumping and venting system, as shown in the right figure of Fig. 3.



Figure 4: Vacuum trend measured by CG during slow pumping.

Figure 4 shows the vacuum trend under slow pumping by using UPCUS. We could successfully control the constant flow. This flow ratio was calculated as 0.6 l/min as shown in Fig. 4. After reaching 100 Pa, slow pumping was

changed to the main pumping automatically and reached 3  $x 10^{-4}$  Pa after one day.



Figure 5: (Left) Vacuum trend measured by CG (blue) and IG (red) just after starting slow pumping. Rotation of TMP is shown as green line (right). Trend of measured cumulative numbers of particulates just after starting slow pumping. Each colour shows the range of measured particulate size from  $0.3 \mu m$  to  $3.6 \mu m$ .

Figure 5 shows the results of particulates by measured vacuum particle monitor just after starting slow pumping. No particulate was observed under slow pumping except for  $0.3 \mu m$  to  $0.5 \mu m$  range. We saw the increasing of particulates of 0.3-0.5 µm ranges from  $1 \times 10^5$  Pa to  $8 \times 10^4$  Pa. We doubted that the long bellows between 9-cell cavity and slow pumping system made some particulates because it was not rinsed under replacement. Finally, there were 18 counts only in the range between 0.3  $\mu$ m to 0.5  $\mu$ m for one day. We could suppress the particulates movements by using this system.

*Slow Pumping with HPR* 



After HPR, we will assembly After dry assembly in one night, we 9-cell cavity in class 10 have some water at top flange

Figure 6: (Left) Assembly work of 9-cell cavity after HPR. (Right) The replaced top flange after HPR after keeping one night in class 10 clean room to dry cavity.

After first slow-pumping test, we vented the nitrogen gas with slow venting and disassembled the cavity. After that, we performed HPR to this 9-cell cavity. We noted that there were no particulates during slow venting. First, all ports of 9-cell cavity were opened under HPR. And finally, all ports except for the bottom one were covered by the stainless flanges in order to protect particulates come into the cavity during HPR. The high pressure rinsed 9-cell cavity were moved to class 10 clean room and dried the inner surface of the cavity for one night. After that, the 9-cell cavity was re-assembled and connected to the slow pumping system. We note that we saw many water-drops on the removed flange as shown in the right figure in Fig. 6 even though we kept drying for one night.



Figure 7: Vacuum trend measured by CG with HPR (blue) during slow pumping and without HPR (red). Left (right) figure shows the vacuum trend of the linear (logarithmic) scale, respectively.

Figure 7 shows the vacuum trend under slow pumping with HPR (blue) and without HPR (red). We could perform the same slow pumping speed after HPR as shown in the left figure of Fig.7. Compared with the slow pumping without HPR, it took a long time to reach to 100 Pa. We assume that there were many water mists in the cavity under pumping. The slow pumping changed to main pumping and reached to 7 x 10<sup>-5</sup> Pa for three days.



Figure 8: (Left) Trend of measured cumulative numbers of particulates just after starting slow pumping. (Right) Trend of measured cumulative numbers of particulates for more three days. In both figures, each colour shows the range of measured particulate size from  $0.3 \mu m$  to  $3.6 \mu m$ .

Figure 8 shows the results of particulates measured by vacuum particle monitor after slow pumping. We observed many particulates just after starting slow pumping as shown in the left figure of Fig. 8. This sudden increasing of particulates as shown in the left figure of Fig. 8 was appeared in the pressure range of  $1 \times 10^5$  Pa -  $9.8 \times 10^4$  Pa. The sizes of these particulates were widely distributed from  $0.3 \mu m$  to  $2.0 \mu m$ . The maximum counts of particulates are about 90 counts in the range between  $0.5 \mu m$  to  $1.0 \mu$ m. This distribution seems to be very strange. Normally, the smaller the size of the dust, the greater the amount of dust. For example, we saw that the maximum counts were shown in the range of minimum size like 0.3  $\mu$ m to 0.5  $\mu$ m when some dusts come into the cavity [1, 6]. In our case just after HPR, the maximum counts were not seen in the smallest size. We assume that the observed particulates might be something concerning with water.

#### *Second Slow Pumping Test without Disassembly*

We tried slow pumping again without disassembly of the cavity to see the particulates movements. First, we vented the nitrogen with slow venting. Under slow venting, we did not observe the particulates.



Figure 9: Vacuum trend measured by CG after HPR during first slow pumping (red) and second slow pumping without disassembly of the cavity after HPR (blue). Left (right) figure shows the vacuum trend of the linear (logarithmic) scale, respectively.



Figure 10: Trend of measured cumulative numbers of particulate under second slow pumping. Each colour shows the range of measured particulate size from  $0.3 \mu m$  to 3.6 um.

Figure 9 shows the vacuum trend under slow pumping after HPR (red) and second slow pumping again (red). Compared with the slow pumping just after HPR, it took shorter to reach to100 Pa under slow pumping. This is very similar behaviour with that without HPR as shown in Fig. 4. Finally, we could reach below 1 x 10-4 Pa for one day. We found that the system works well again.

Figure 10 shows the measured particulates trend under second slow pumping. We found no particulates. We did not reproduce the particulates events as shown in Fig. 8. Under second slow pumping, we did not see the gentle slope under slow pumping just after HPR. If this behaviour of gentle slope around a few 1000 Pa come from the high pressure rinsed water mist, this water went out and we could smooth slow pumping like that without HPR shown in Fig. 4.

We assume that many particulates might be associated with some water mists under the comparison between the measured results just after HPR and second slow pumping. If so, we could distinguish the particulates come from mist and the other dusts by measuring the particulate distribution. Unfortunately, it is too early to state this behaviour come from the water mist. We need to take more data after HPR for vertical tests by using vacuum particle monitor.

### **SUMMARY AND FUTURE PROSPECT**

We modified the slow pumping system to escape the icing in the mass flow meter to carry out the slow pumping after assembly work with HPR. First, we smoothly carried out slow pumping with this modified system by using 9 cell cavity without HPR. For the 9-cell cavity applied HPR, we observed some different vacuum trend around 1000 Pa level. At this pressure level, it took long time to pump under slow pumping. But we met no icing in the bellows and cavity. The vacuum finally reached the  $1 \times 10^{-5}$  Pa level. We saw a lot of particulates at the range of  $0.3$ -2.0  $\mu$ m particles sizes just after HPR. After venting this 9-cell cavity and applying slow pumping without disassembly this cavity, we met no particulate and pumping profile under slow pumping is very similar with the slow pumping without HPR.

We found that this modified slow pumping system works well with HPR. This system gave the clean vacuum work. We will take more data after HPR for vertical tests to survey why the measured particle distribution by vacuum particle monitor were strange. For example, we try to compare the measured particulates with and without slow pumping condition.

#### **ACKNOWLEDGEMENTS**

We would like to express our gratitude to M. Asano, H. Yamada of NAT Co. and T. Okada of K-VAC Co. for their efforts about HPR preparation and cavity assembly works in clean room by using slow pumping system. This research was partially supported by the research fund from Ministry of Education, Culture, Sports, Science and Technology (MEXT).

#### **REFERENCES**

- [1] T. Behnke *et al*., "ILC Technical Design Report", Fermilab, Chicago, IL, USA, Jun. 2013. https://www.linearcollider.org/ILC/Publications/Technical-Design-Report/.
- [2] Y. Yamamoto *et al*., "High Gradient Cavity Performance in STF-2 Cryomodule for the ILC at KEK", in *Proc. IPAC'16*, Busan, Korea, May 2016, pp. 2158-2160. doi:10.18429/JACoW-IPAC2016-WEPMB017
- [3] K. Zapfe and J. Wojtkiewicz, "Particle free pump down and venting of UHV vacuum systems", in *Proc. SRF'07*, Beijing, China, Oct. 2007, paper WEP74, pp. 681-684.
- [4] H. Sakai et al, "Improvement of a Clean Assembly Work for Superconducting RF Cryomodule and Its Application to the KEK-STF Cryomodule", in *Proc. SRF'19,* Dresden, Germany, Jun.-Jul. 2019, pp. 721-725. doi:10.18429/JACoW-SRF2019-TUP104
- [5] Wexx Co., Ltd.; http://www.wexx.jp/

**Pure** 

# **SRF Technology**

**Assembly/integration**

- [6] H. Sakai *et al.,* "Development of the Slow Pumping & Venting System", in *Proc. of PASJ2018*, Nagaoka, Japan, 2018, p. 1157. https://www.pasj.jp/web\_pub-
- lish/pasj2018/proceedings/PDF/THP1/THP111.pdf
- [7] Y. Yamamoto *et al*., "Stable Beam Operation at 33 MV/m in STF-2 Cryomodules at KEK", in *Proc. SRF'21*, East Lansing, MI, USA, Jun.-Jul. 2021, pp. 382. doi:10.18429/JACoW-SRF2021-TUPFAV003
- [8] T. Ebisawa *et al.*, "Preparation of the Cryomodule Assembly for the Linear IFMIF Prototype Accelerator (LIPAc) in Rokkasho", in *Proc. SRF'19*, Dresden, Germany, Jun.-Jul. 2019, pp. 726-729. doi:10.18429/JACoW-SRF2019-TUP105
- [9] FCS series, Fujikin Co. Ltd., https://www.fujikin.co.jp/support/pdf/712- 01\_pure\_fcs\_eng.pdf

**SRF Technology**