

LONG-PERIOD MONITORING OF ELECTRO-POLISHING ELECTROLYTE IN EP FACILITY AT KEK

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Abstract

We have constructed an Electro-polishing (EP) Facility in the Superconducting RF Test Facility (STF) at KEK at the end of 2007. We have begun to operate the EP facility from 2008 and performed the EP processes 41 times with the same EP-electrolyte. In this article, we report the detailed results of the EP-electrolyte monitoring as well as the observation of changing electronic current oscillation in the EP processes.

INTRODUCTION

In the EP facility, the capacity of the EP-electrolyte reservoir tank is 2,000 L. This size is relatively large if compared with EP facilities in other laboratories. It means that the quality control of EP electrolyte is more difficult because the status of EP-electrolyte changes as the aging of EP-electrolyte proceeds. In the real EP-process operations, we circulated the EP electrolyte of 1,100 L which was firstly delivered into the tank in January 2008 and was disposed in May 2009. During this period, we performed the EP processes 41 times and periodically measured the concentration of Nb, Al, HF in the EP electrolyte. In this article, we report the detailed results of the EP-electrolyte monitoring as well as the observation of changing electronic current oscillation in the EP processes during this period in the EP facility at STF/KEK. [1] [2].

EP PROCESS AT STF-KEK

The schematic of EP-electrolyte flow in the EP process is shown in figure 1. The schematic of water flow in the water-rinse process is shown in figure 2, where the process happens just after EP-electrolyte is drained out. The standard EP process at KEK is shown in figure 3, where the flange of cavity is Chemical Polished (CP'ed), the inner surface of cavity is EP'ed, water-rinsed, detergent-rinsed in the ultrasonic hot-water bath, running water-rinsed in hot-water bath, and High-Pressure-Rinsed.

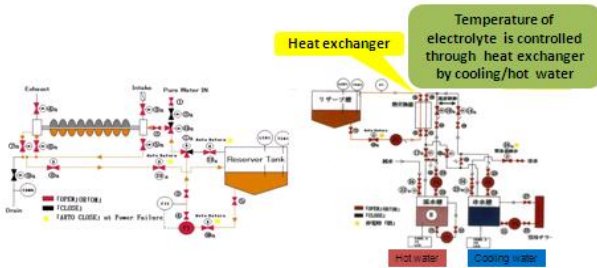


Figure 1: Schematic of EP-electrolyte flow.

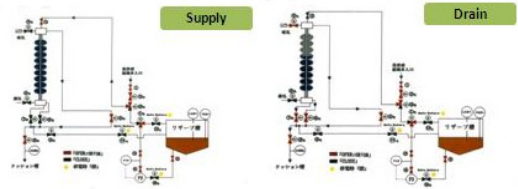


Figure 2: Schematic of water flow in water rinse process.

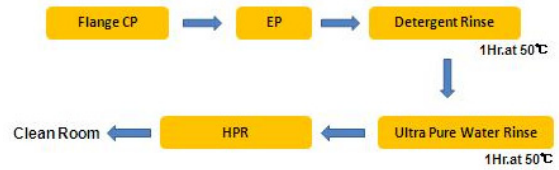


Figure 3: EP process (EP-2)

We performed EP processes 41 times with the first electrolyte from January 2008 to May 2009. The removal weight of Nb in each EP process and summing up of each removal is shown in figure 4.



Figure 4: Removal weight in each EP and summing up of each removal.

MANAGEMENT OF EP-ELECTROLYTE

We measured the concentration of niobium and aluminum in the EP electrolyte by Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP) as shown in figure 5. The niobium concentration calculated by current integration (charge) and that of ICP agreed very well. It was found that the concentration of aluminum increases in the similar way to that of niobium. We also measured the fluorine concentration by steam distillation method and Ion Selective Electrode (ISE). All fluorine was measured as in the form of fluoride ion by this method. The picture of the analytical instruments and

samples of EP-electrolyte are shown in figure 6. We assumed that fluorine in the EP-electrolyte is composed of fluorine in the niobium oxygen complexes (H_2NbFO_5), in hydrogen fluoride (HF) and in fluorosulfuric acid (SO_3F). This assumption was checked by the ion chromatograph measurements of diluted EP-electrolyte and diluted fluorosulfuric acid (SO_3FH) as shown in figure 7.

Because we already measured the niobium concentration in EP-electrolyte by current integration and/or ICP, we can calculate the fluorine quantity in H_2NbFO_5 . On the other hand, the total amount of fluorine is measured by steam distillation method and ISE. Then the sum of fluorine concentration in the form of HF and SO_3F can be also calculated by subtracting fluorine in H_2NbFO_5 from the total fluorine.

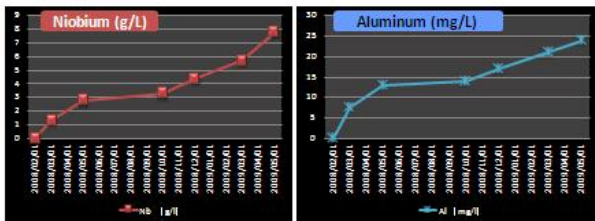


Figure 5: The changes of niobium and aluminum concentration in the function of days.



Figure 6: Analytical instruments and samples of EP-electrolyte.

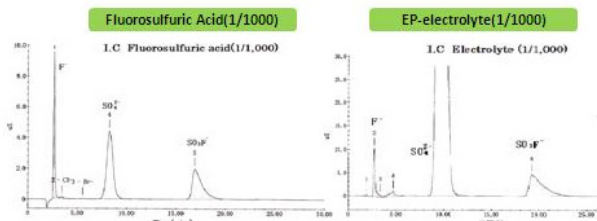


Figure 7: Ion chromatograph of diluted EP-electrolyte and diluted fluorosulfuric acid.

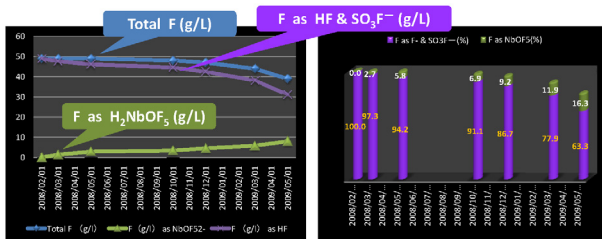


Figure 8: The fluorine concentration in the EP-electrolyte as the function of day.

The fluorine concentration in the EP-electrolyte as the function of day during the period of one year and six months is shown in figure 8. It is found that during the period about 16% of fluorine was used for the EP reaction to remove the niobium, and about 20% fluorine moved out from the EP-electrolyte into exhaust gas and rinsed water.

MANAGEMENT OF EP-CURRENT

We are recording various data in each EP process automatically in the sampling period of one second. We pay special attention to EP current oscillation in them. We compared the shapes of EP current oscillation in last one minute of each EP process. We found that this oscillation pattern changes as the niobium concentration increases as shown in figure 9.

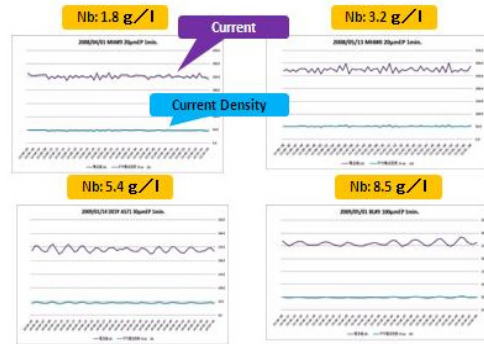


Figure 9: The changes of oscillation pattern with regard to the niobium concentration. The each time-period of all plots is one minute. The EP current is about 250A.

We found that the EP current oscillation pattern becomes smoother and the amplitude becomes smaller as the EP-electrolyte aging proceeds, particularly after the niobium concentration of around 4~5g/L.

THE INCOME AND EXPENDITURE OF FLUORINE / NIOBIUM

Flow of chemicals in the EP system

We performed EP processes 41 times during the period between January 2008 and May 2009. During this period, we monitored the income and expenditure of chemicals in the EP system. The flow of the chemicals in the EP system is shown in figure 10. The brand-new EP-electrolyte is filled in the EP-tank of the system as income of chemicals. As described in the previous section, the chemicals in the EP-electrolyte escape into the exhaust gas and rinsed water. The chemicals in exhaust gas are absorbed in the alkaline water of two absorption towers. The alkaline water in two absorption towers was transferred into the waste-water tanks periodically. In the same waste-water tank, the rinsed water of EP processes is drained in. We measured the concentration of fluorine and niobium in waste-water in the same way as the EP-electrolyte. And finally we transferred the aged EP-

electrolyte and waste-water for disposal out of the EP system as shown in figure 10.

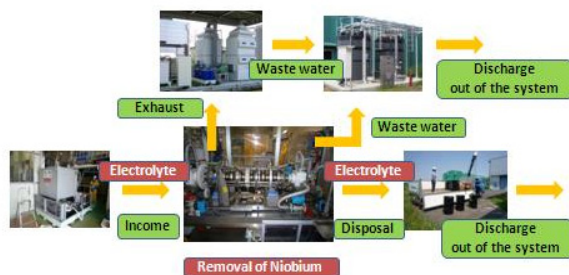


Figure 10: The flow of the chemicals in the EP system. The income is brand-new EP-electrolyte and the expenditure is aged EP-electrolyte and waste-water.

Exhaust gas

The exhaust gas is detoxified with two absorption towers, and the detoxified air is discharged in the atmosphere. The two absorption towers are put in series. And alkaline water circulates in the two towers to scrub the exhaust gas. The fluorine concentration in alkaline water in each tower was measured periodically. The results of the measurements was that no hydrogen fluorine was found in second tower as shown in figure 11. This means that the hydrogen fluoride in exhaust gas is completely removed by the two absorption towers. Alkaline absorption water is sent to the waste-water tank regularly.

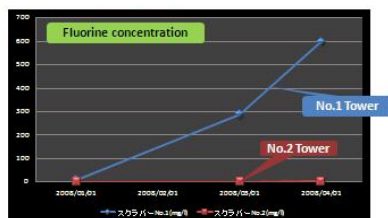


Figure 11: Fluorine concentration in absorption towers. Hydrogen fluoride in exhaust gas is removed completely with No.1 absorption tower.

Monitor of income and expenditure

The income and expenditure of fluorine/niobium are summarized in table 1, where all numbers are based on the measurements of fluorine and niobium concentrations. The income is composed of the total amount of fluorine in the brand-new EP-electrolyte and the total removal of niobium. The expenditure is composed of fluorine and niobium in waste water in eight disposals and aged EP-electrolyte in one disposal. The expenditure of waste water and aged EP-electrolyte can be summed up and compared with the income. The income and expenditure of fluorine/niobium agreed very well. Fluorine of 10,213g in the EP-electrolyte escaped in waste water. This quantity is about 20% of the income. Niobium of 932g moved into the waste water. This quantity is about 10% of all the removal.

Table 1: The income and expenditure of EP-electrolyte.

INCOME (Electrolyte)

Date	Carry Volume (l)	Fluorine (mg/l)	Fluorine (g)		Total Removal Weight(g)	
2007/12/26	1100	49000	53900	-	9433	

EXPENDITURE (Waste Water)

Date	Carry Volume (l)	Fluorine (mg/l)	Fluorine (g)	Niobium (mg/l)	Niobium (g)	Disposal
2008/1/11	900	130	117	-	-	KEK
2008/2/1	600	350	210	-	-	KEK
2008/2/15	900	650	585	-	-	KEK
2008/2/29	1000	340	340	-	-	KEK
2008/6/19	7500	160	1200	8.1	61	KEK
2008/11/18	7500	270	2025	14	105	KEK
2009/4/17	6800	270	1836	17	116	KEK
2009/5/22	10000	390	3900	65	650	Outsid company
Sub-total	35200		10213		932	

EXPENDITURE (aged Electrolyte)

Date	Carry Volume (l)	Fluorine (mg/l)	Fluorine (g)	Niobium (mg/l)	Niobium (g)	Disposal
2009/5/14	1100	39000	42900	7800	8580	Outside
Total			53113		9512	

SUMMARY

We performed EP processes 41 times at KEK-STF EP facilities in the period of one year and six months. The concentrations of niobium and aluminium in the EP-electrolyte were measured. The concentration of aluminum increased in the same way as that of niobium as the function of day. About 16% fluorine was used for the reaction of niobium removal. The EP current oscillation pattern became smoother and smaller as EP-electrolyte aging proceeds. The income and expenditure of fluorine/niobium agreed very well. Fluorine of 10,213 g (20%) in the EP-electrolyte escaped in waste-water. Niobium of 932g (10%) of EP removal moved into the waste water.

REFERENCES

- [1] K.Ueno, et al., the 5th annual meeting of Particle Acc. Society of Japan (PASJ), Aug 2008, "Development of Electro Polishing (EP) facility in KEK" in Japanese.
- [2] K.Ueno, et al., the 6th annual meeting of PASJ, Aug 2009. "Development of Electro Polishing (EP) facility in KEK (2)" in Japanese.