

# Status of Nb sample study

TILC09

Main Linac S0 session

18 April 2009

T. Saeki

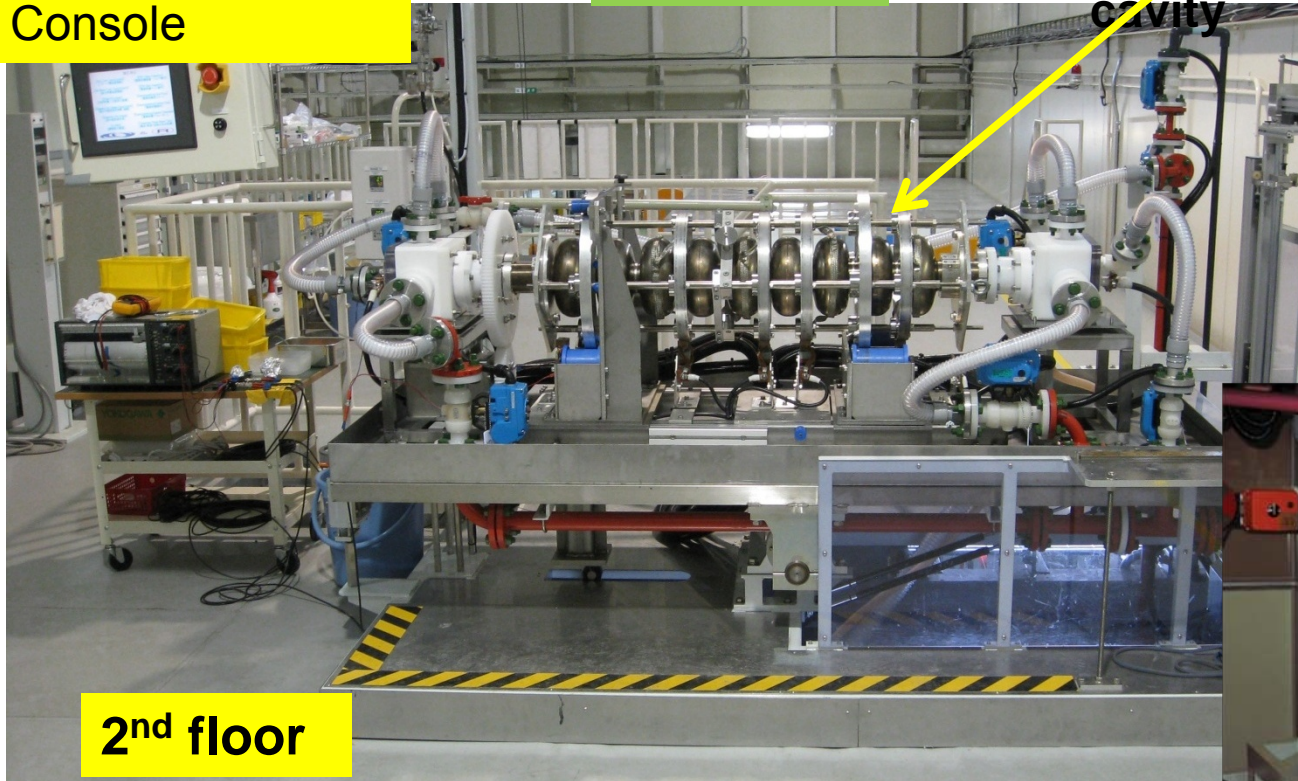
on behalf of SRF surface analysis group at KEK

# EP facilities at STF/KEK

Automatic  
Operation  
Console

EP bed

9-cell  
cavity



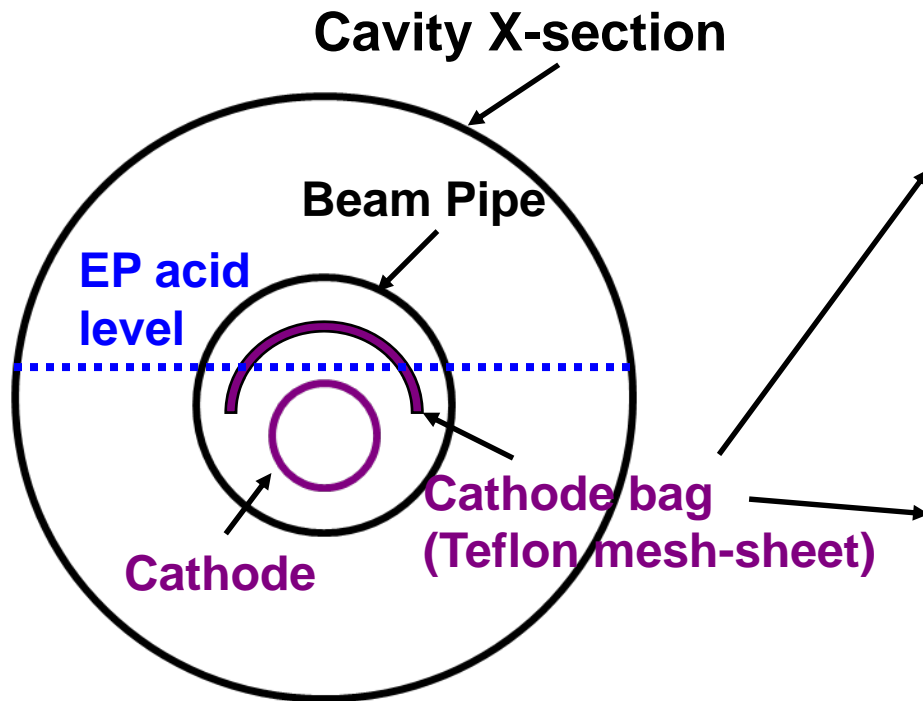
2<sup>nd</sup> floor

1<sup>st</sup> floor



EP acid reservoir tank

# Powder on cathode bag at STF/KEK



Commissioning run of  
STF EP facility  
30 June – 1 July 2008



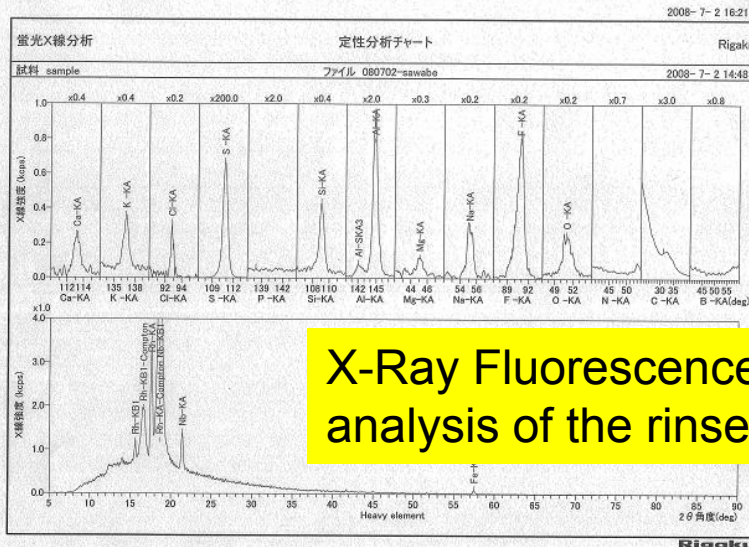
Location:  
Iris Cell#4-cell#5, Iris cell#5-cell#6

# Analysis of powder on the cathode bag

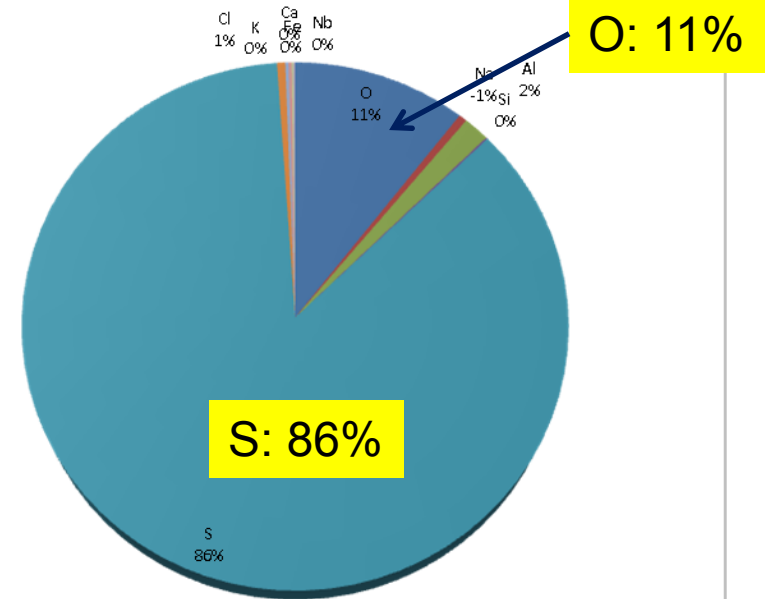
Ethanol rinse of cathode bag



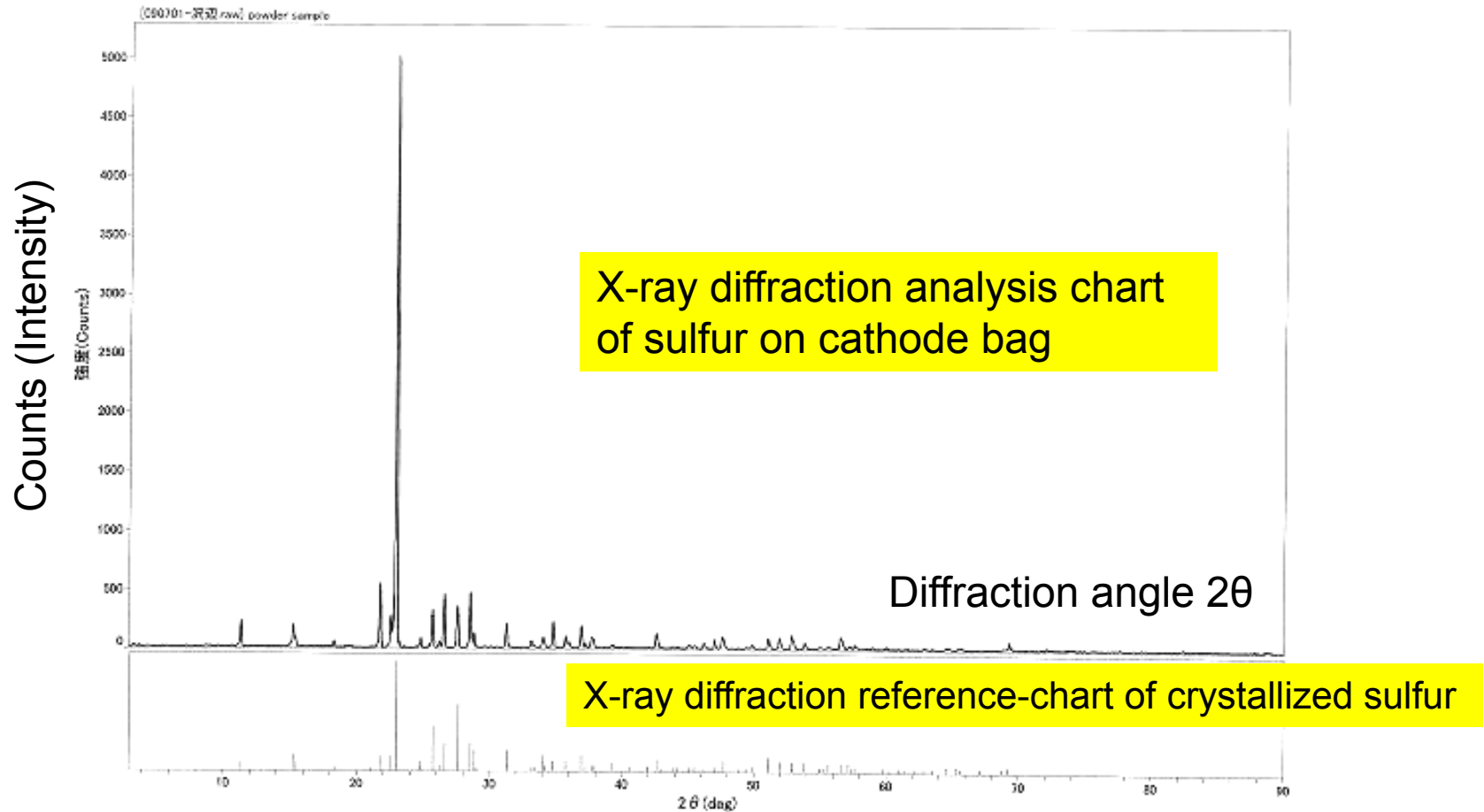
Cell 5



Results of analysis by XRF



# X-ray diffraction analysis



High Energy Accelerator Research Organization

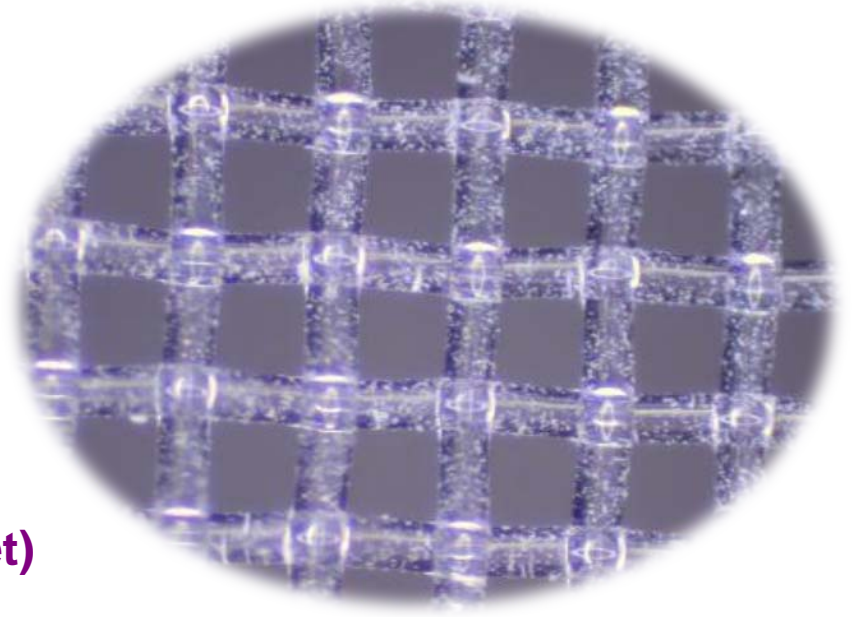
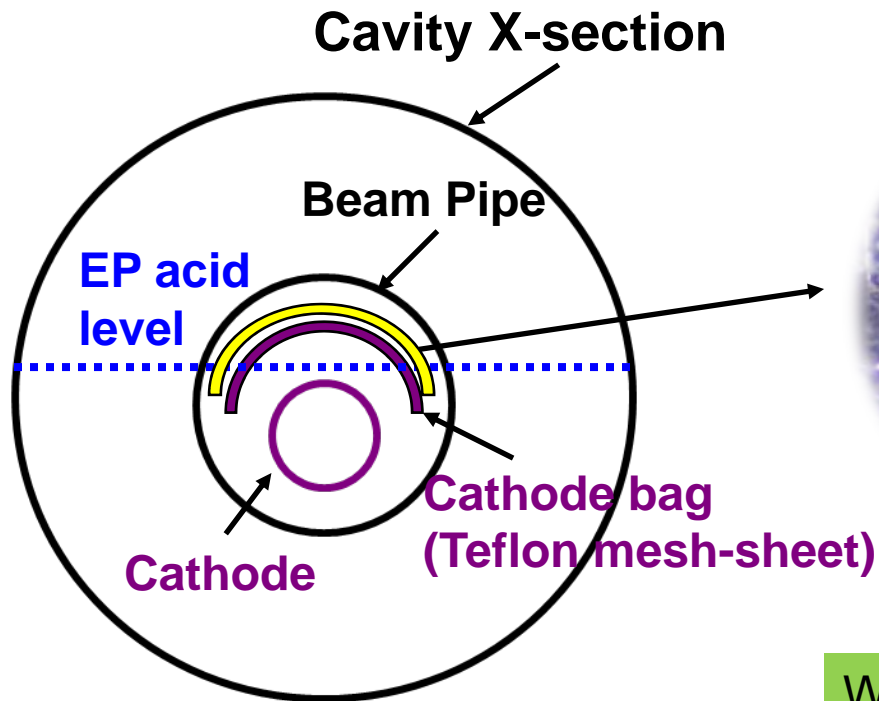
2008年7月1日(火)16:16 (M01-JADE)



The results shows that **sulfur is in a crystallized form**



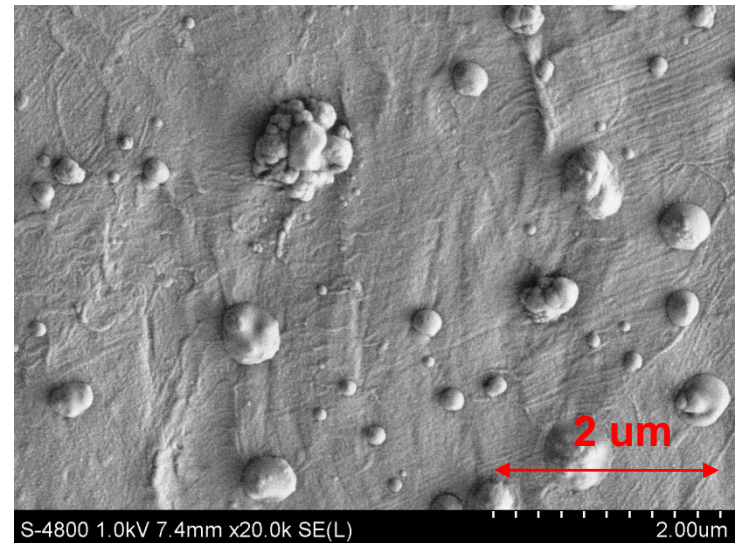
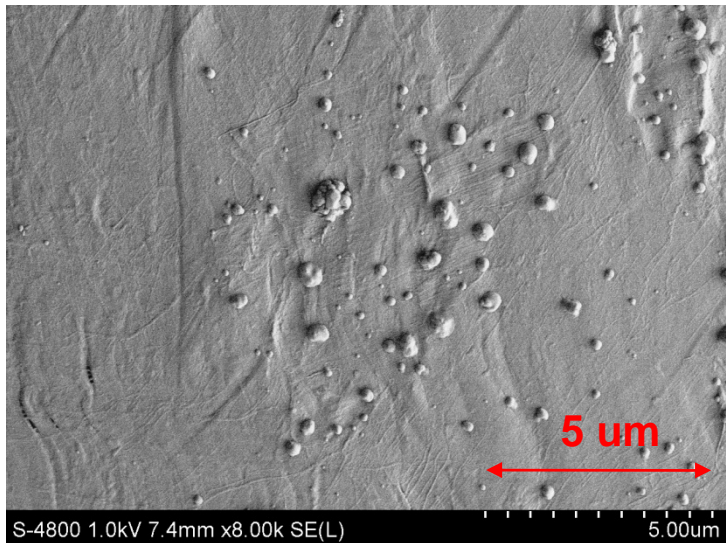
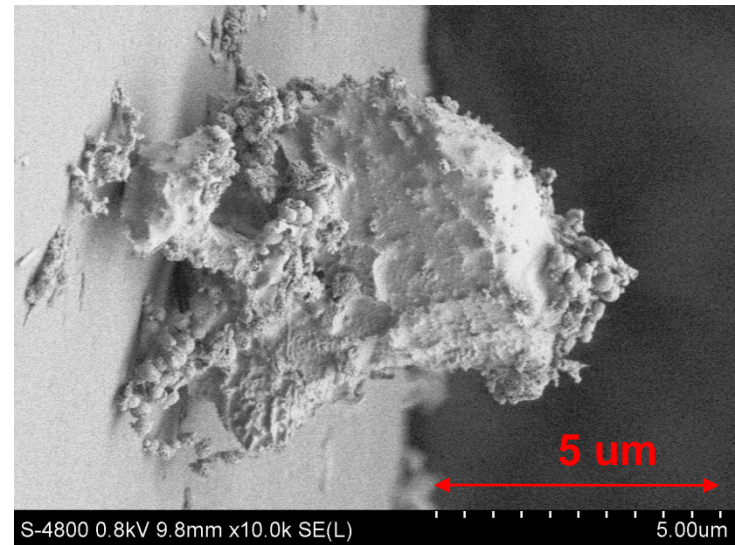
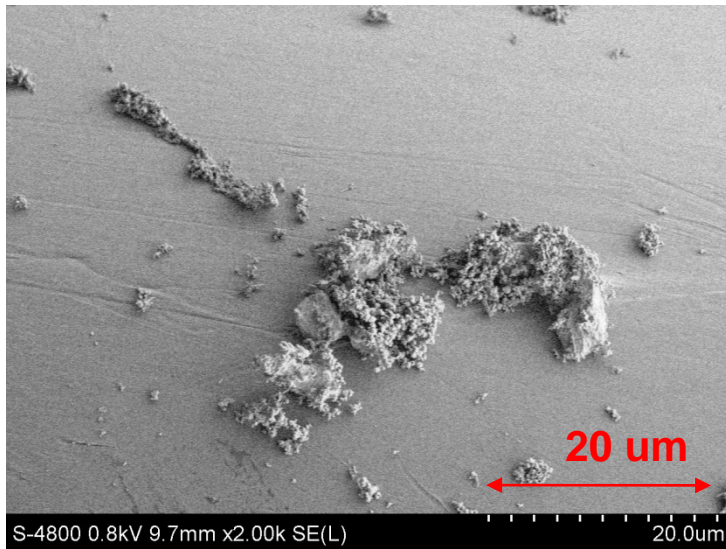
# Collection of sulfur powder on cathode bag



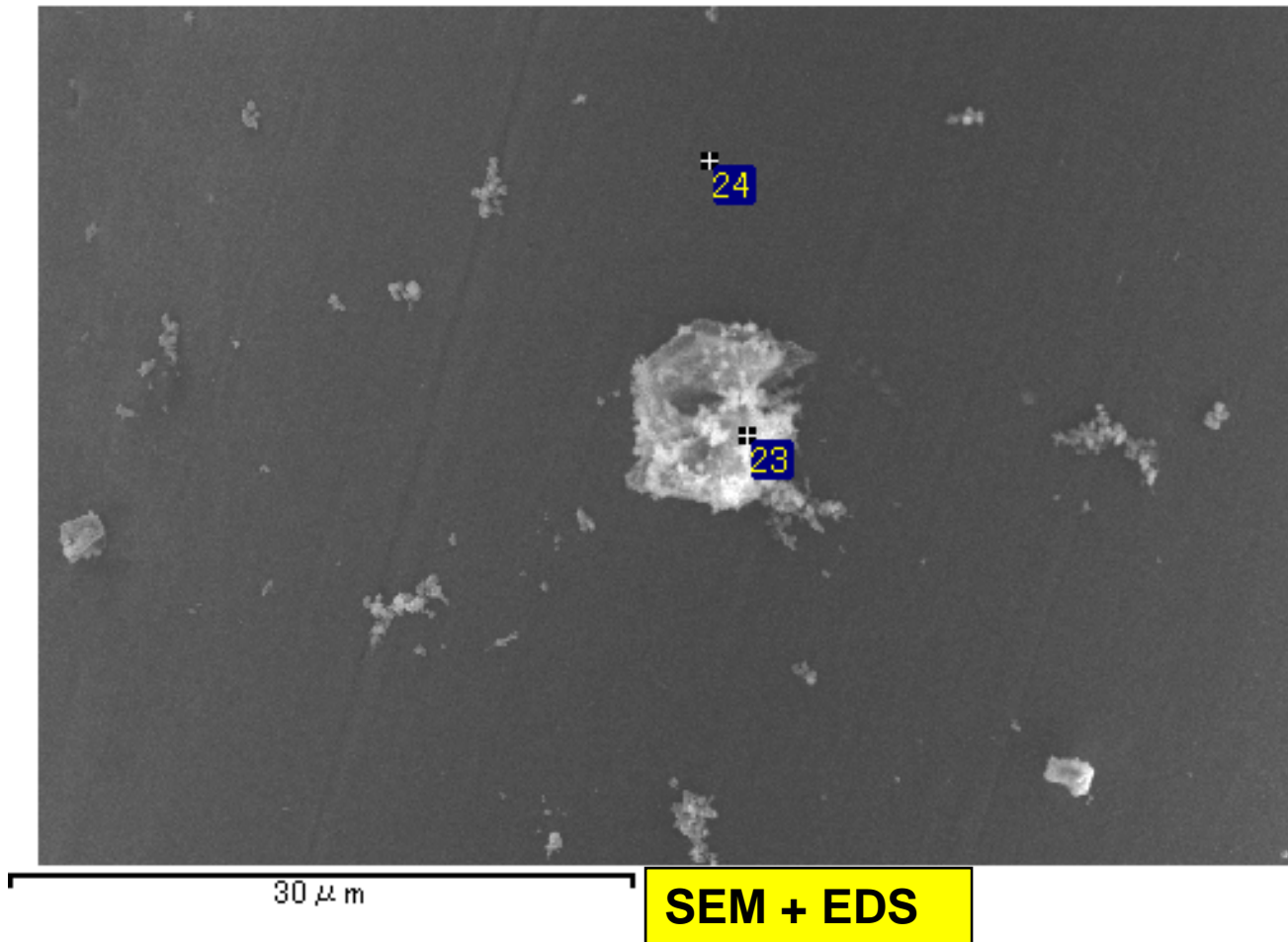
Location:  
Iris Cell#4-cell#5, Iris cell#5-cell#6

We added detachable piece of Teflon mesh-sheet over the cathode bag. The sheet collected sulfur powder in the EP acid during the real EP processes of 9-cell cavities at STF/KEK.

# Observation of Teflon-mesh by SEM



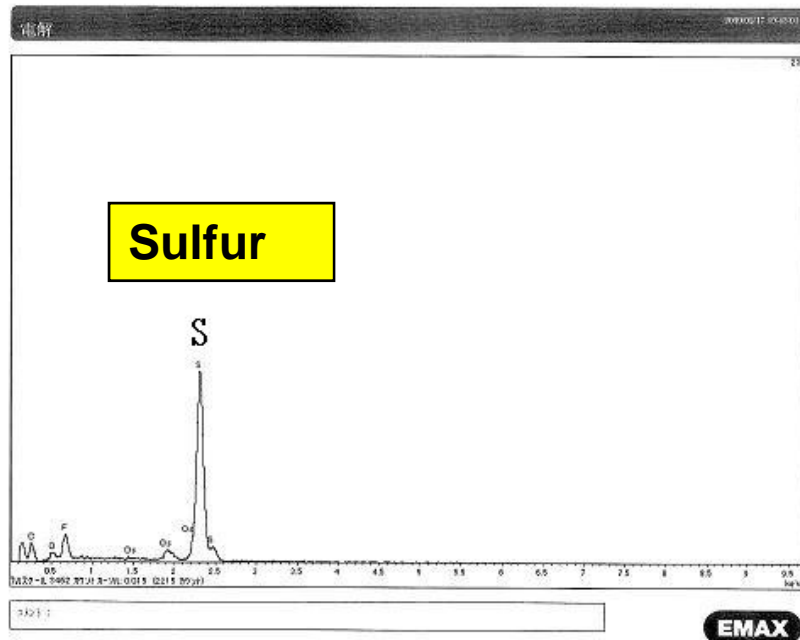
# EDS analysis of Teflon-mesh



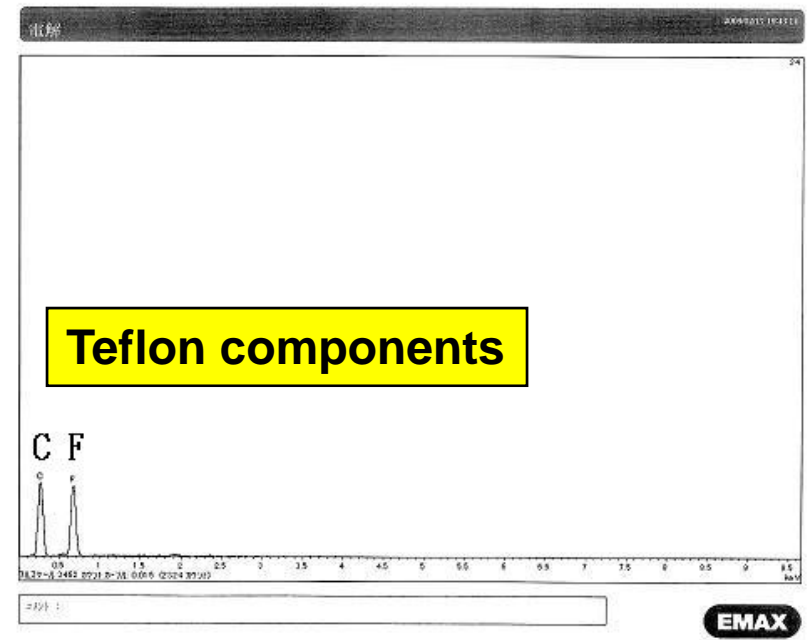


# EDS analysis of Teflon-mesh

23



24

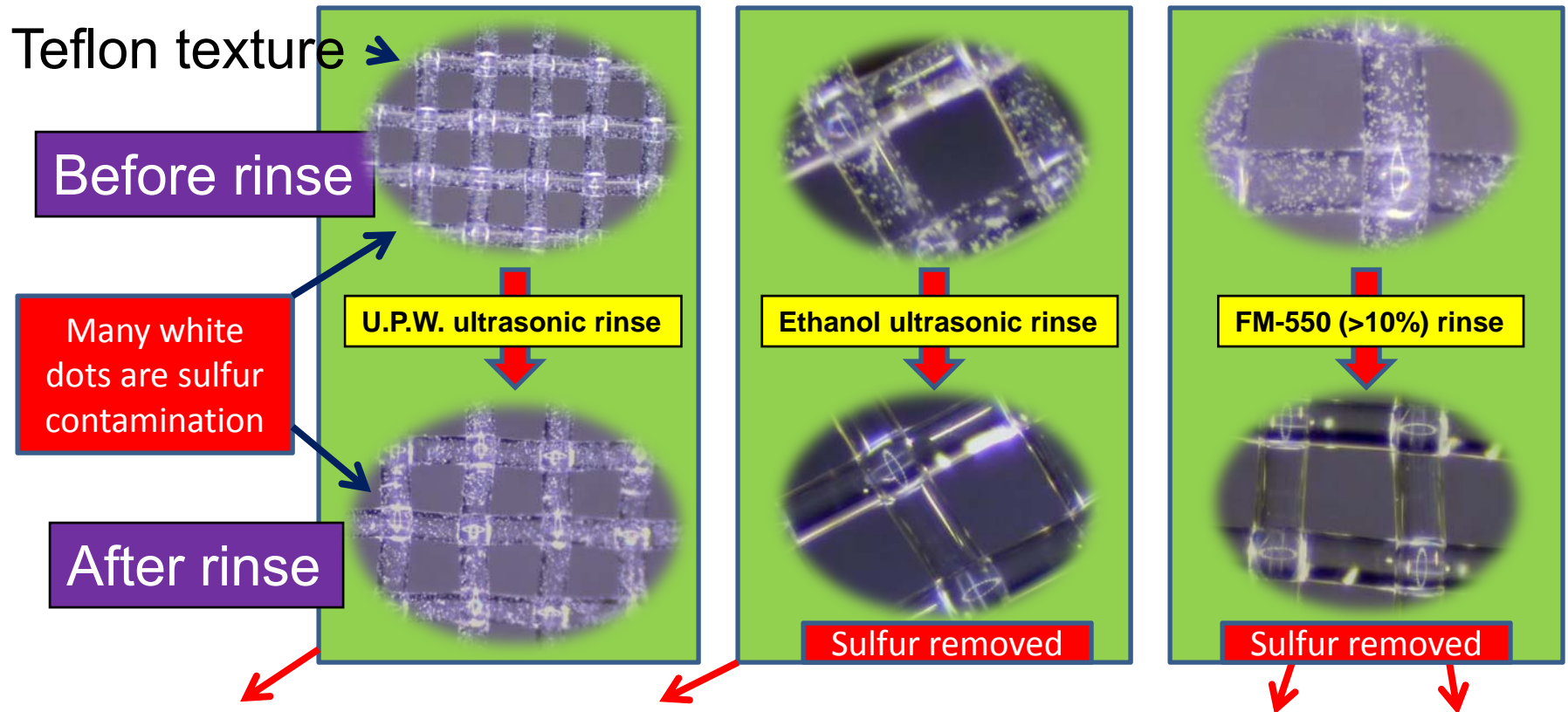


# Tests of rinsing effect for sulfur

- **Degreaser ( LION SUNWASH FM-550, FM-20 )**  
concentration = 0%, 2%, 5%, 10%, 20%  
50ml, T=50°C, 1 hour, Ultrasonic rinse
- **Ethanol**  
10ml, Room temp., 10min., Mechanical vibration (at a few Hz)  
50 ml, Room temp., 1 hour, Ultrasonic rinse
- **H<sub>2</sub>O<sub>2</sub>**  
50 ml, 10% H<sub>2</sub>O<sub>2</sub>, T=50 °C, 1 hour, Ultrasonic rinse



# Rinse Effect to Remove Sulfur precipitation/contamination



	U.P.W. ultrasonic rinse	Ethanol rinse (vibration)	Ethanol ultrasonic rinse	Detergent FM-550 2 %	Detergent FM-550 5 %	Detergent FM-550 10 %	Detergent FM-550 20 %
Cleanin g Result	✕	△	○	△	△	○	○

# Results of rinse effect tests

- Test of FM-20 (LION detergent) and 10% H<sub>2</sub>O<sub>2</sub>

	Detergent FM-20 2 %	Detergent FM-20 5 %	Detergent FM-20 10 %	10% H <sub>2</sub> O <sub>2</sub>
Cleaning Result	○	○	○	✕or△

- Test of Ethanol and FM-550 (LION detergent)

	U.P.W. ultrasonic rinse	Ethanol vibration rinse	Ethanol ultrasonic rinse	Detergent FM-550 2 %	Detergent FM-550 5 %	Detergent FM-550 10 %	Detergent FM-550 20 %
Cleanin g Result	✕	△	○	△	△	○	○



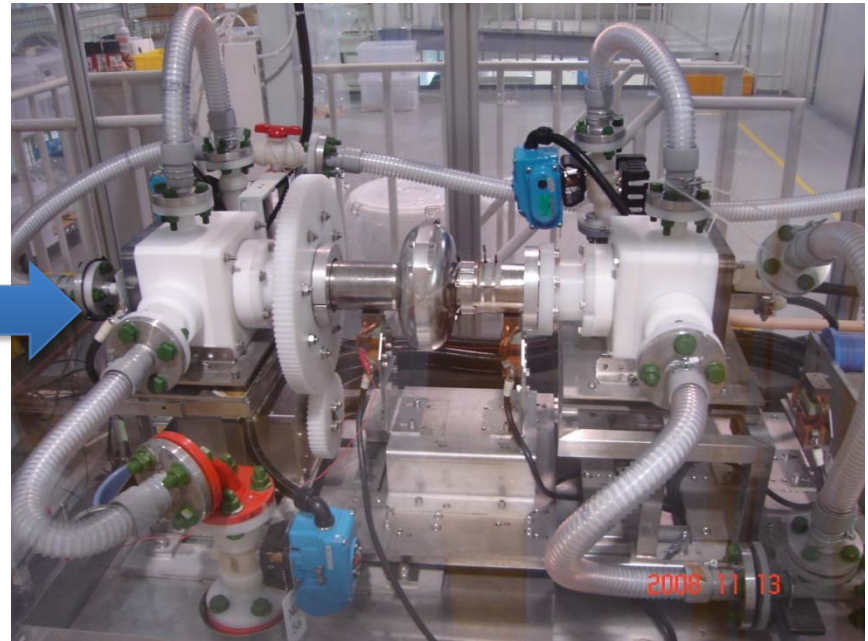
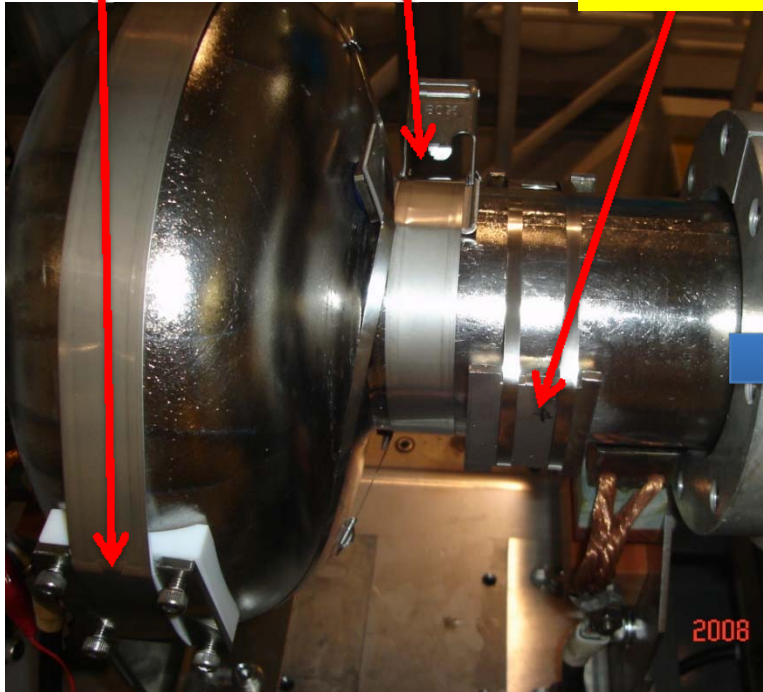
# Special single-cell cavity for sample-study

2 samples  
at equator

2 samples  
at iris

2 samples on  
a beam-pipe

November 2008



6 Nb samples were dressed  
on the special cavity

EP 20  $\mu\text{m}$  + UPW rinse  
(No ultrasonic-rinse)

One of EP'ed sample was analyzed by SIMS.

# Secondary Ion-Microprobe Spectroscopy (SIMS)



We ordered the surface analysis of EP'ed Nb sample by SIMS at a company.

# Specification of Secondary Ion-Microprobe Spectroscopy (SIMS)

Date of measurement : 2009 Feb. 27

Sample: Nb plate

Object : Check if there is S on the surface of the Nb plate by using TOF-SIMS.

Tool: TOF-SIMS (PHI TRIFT IV)

Condition:

Ion =  $\text{Bi}_3^{++}$  (Bismuth ion,  $n=83$ )

Voltage = 30 kV

Ion current = 7 nA (DC)

Measurement area : 600  $\mu\text{m}$  x 600  $\mu\text{m}$

Mass range : 0 – 1850 amu

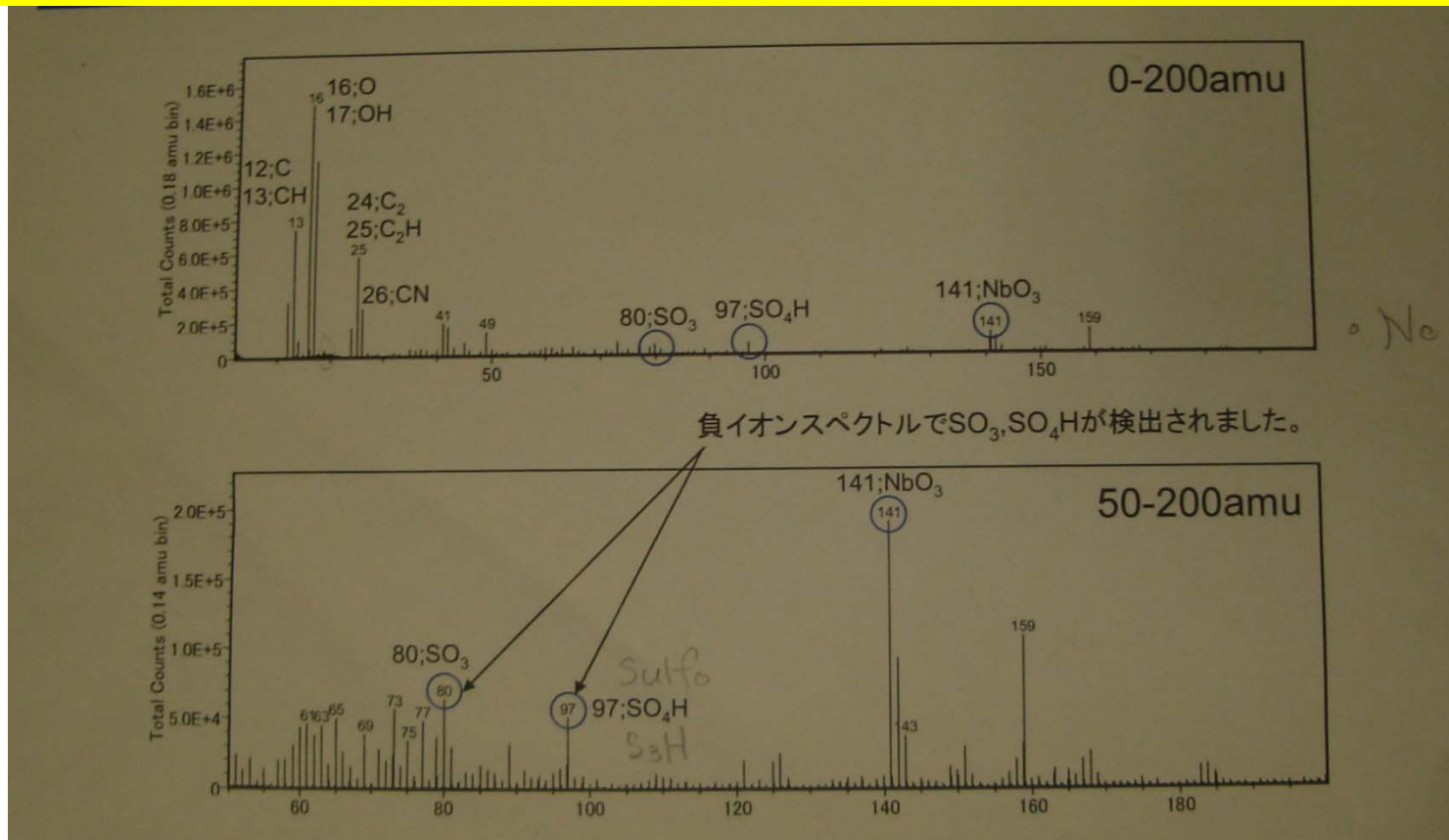
Beam structure: bunch

Secondary ion : minus and plus ions

Measurement time : 10 minutes x 1 time, 30 minutes x 2 times

# Spectrum of Secondary Ion-Microprobe Spectroscopy (SIMS)

There are S signals in the spectrum which is the mean or integral of 600  $\mu\text{m}^2$  area. However, it is very difficult to say how much S is there, because generally the sensitivity coefficient of a component strongly depends on the neighboring components in SIMS analysis. Sometimes, it changes in the order of  $10^2 - 10^3$  depending on neighboring components.

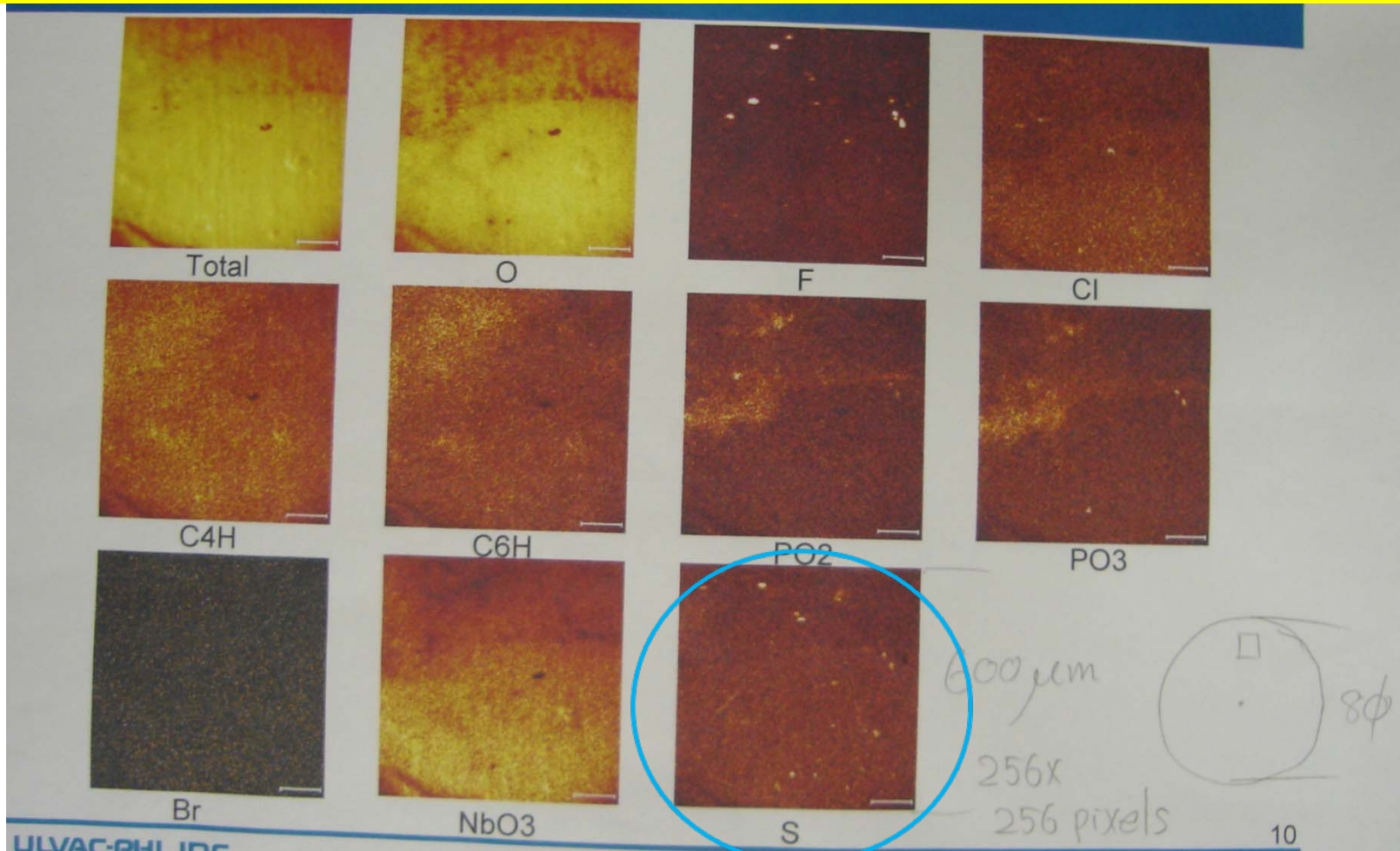


Very preliminary



# Image of Secondary Ion-Microprobe Spectroscopy (SIMS)

Distribution of specific component is seen in an image of 600  $\mu\text{m}$ -square area by 256 x 256 pixels with the pixel size of  $\sim 2.3 \mu\text{m}$ .



There are spots of sulfur in the size of 10 – 20  $\mu\text{m}$ .

# Summary

- In the commissioning run of STF EP facility, we found white powder on the cathode bad.
- The components of white powder is found to be mainly sulfur by X-Ray Fluorescence analysis.
- X-ray diffraction analysis showed that the sulfur powder is in crystallized form.
- We put detachable Teflon-mesh on the cathode bag in the real EP processes of 9-cell cavities to collect sulfur powder.
- Sulfur powder on the Teflon-mesh was observed by SEM and EDX.
- We tried to remove sulfur on the Teflon-mesh by various rinsing methods; detergent, ethanol and  $\text{H}_2\text{O}_2$ .
- We found that ethanol rinse with ultrasonic and detergent (FM-550 and FM-20) rinse with ultrasonic are effective to remove sulfur.
- We fabricated special single-cell cavity with holes in which we can set detachable Nb samples. The special single-cell cavity was EP'ed in the STF EP facility.
- One of the EP'ed detachable Nb sample was analyzed by TOF-SIMS.
- We can say if there is some component there by TOF-SIMS, but quantitative analysis is very difficult.
- We found sulfur on the detachable Nb sample in the measurement area of 600-um square by an imaging with 256 x 256 pixels.