Homogeneous Removal of Nb 1-Cell Cavity using Vertical EP

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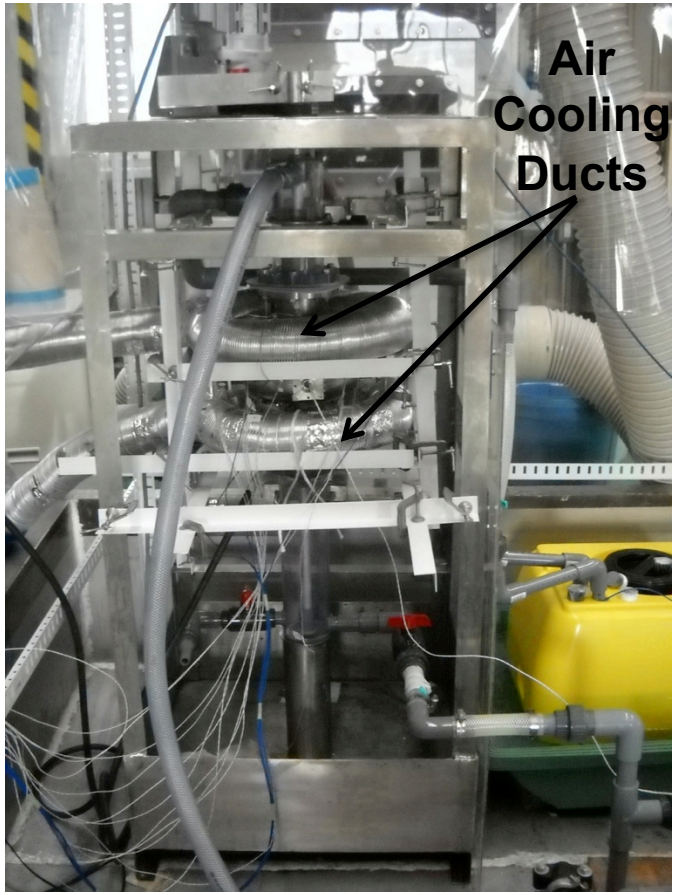
Contents

Introduction

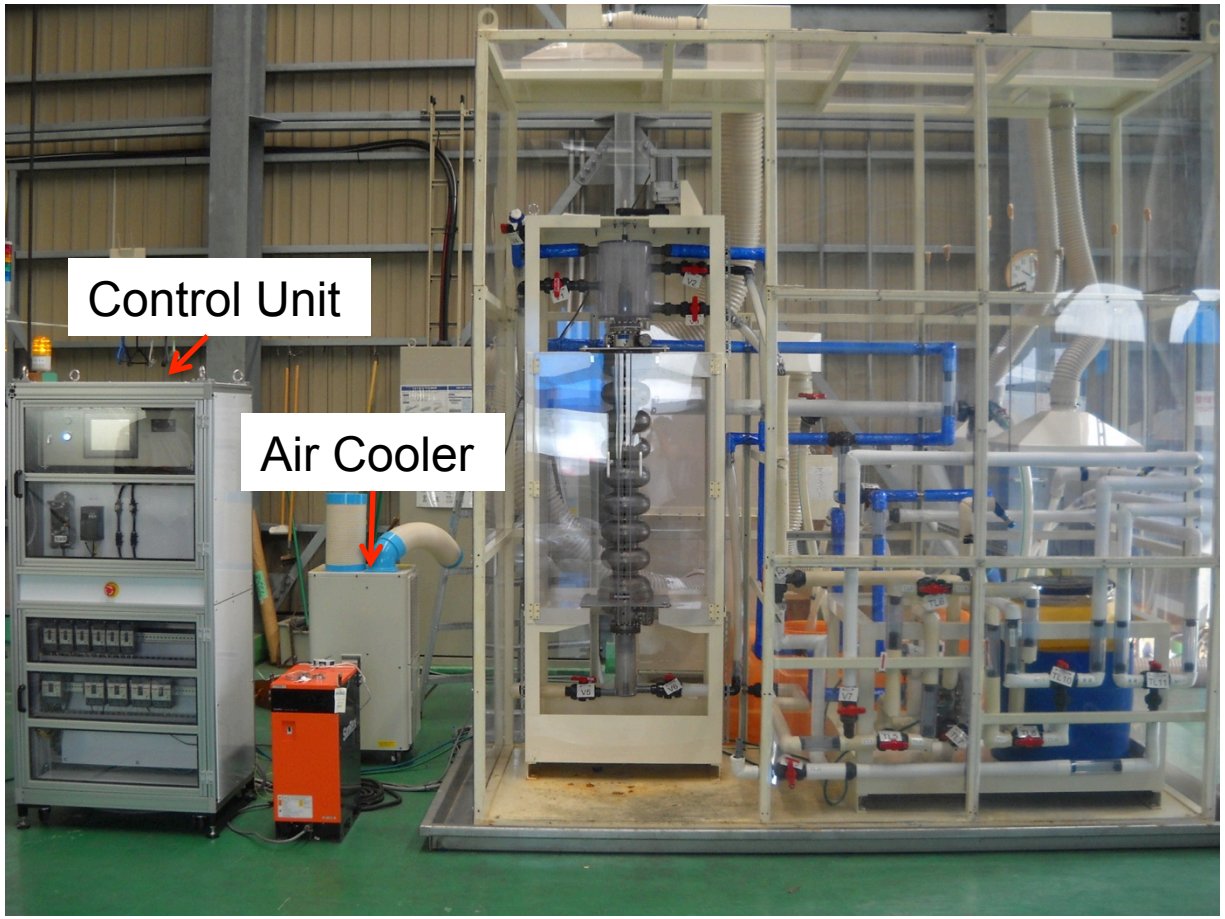
- Marui Galvanizing Co., Ltd (Japan) has been working for vertical electropolishing (VEP) of Nb cavities in collaboration with KEK for the last 3 years.
- VEP setup has advantages over horizontal EP setup especially in respect of simple mechanism and cost reduction.
- However non-uniform removal along the length of a cavity was always observed in VEP.
- In this study we tried to achieve homogeneous removal of Nb.
- For optimization of VEP parameters 1-cell Nb coupon cavity was used.

1 and 9 Cell Cavity VEP Setups

VEP Setup for 1-Cell



VEP Setup for 9-Cell

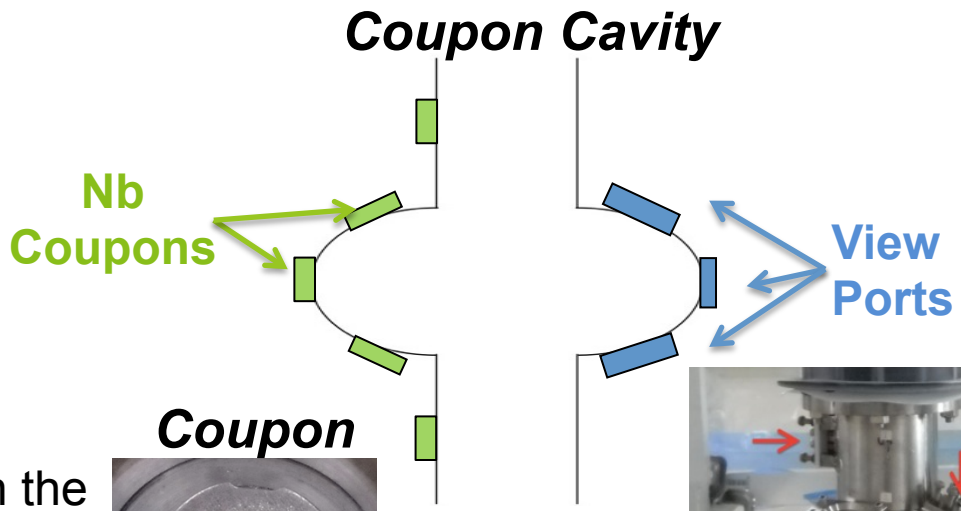


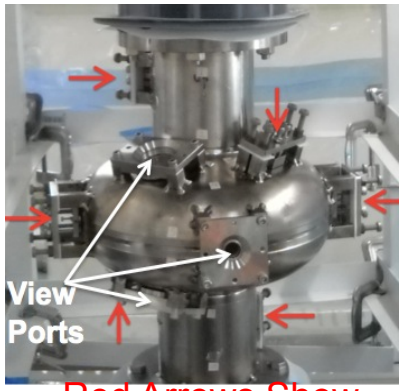
- We challenged to make the setups with PVC material for mass production and cost reduction.
- The 9-cell cavity VEP system can be used for VEP of 1-cell cavity also.
- System contains separate pipe lines and pumps for water and EP solution.
- EP solution and water can be flown from bottom to top and vice versa.

Coupon Cavity and Cathode

Coupon Cavity

- Cavity contains 6 coupons.
- EP current can be measured for individual coupon.
- Coupon surfaces are analyzed with several surface analytical tools.
- The cavity is having 4 view ports on the top iris, bottom iris and equator for light introduction and in-situ observation of cathode wings and H_2 bubbles.

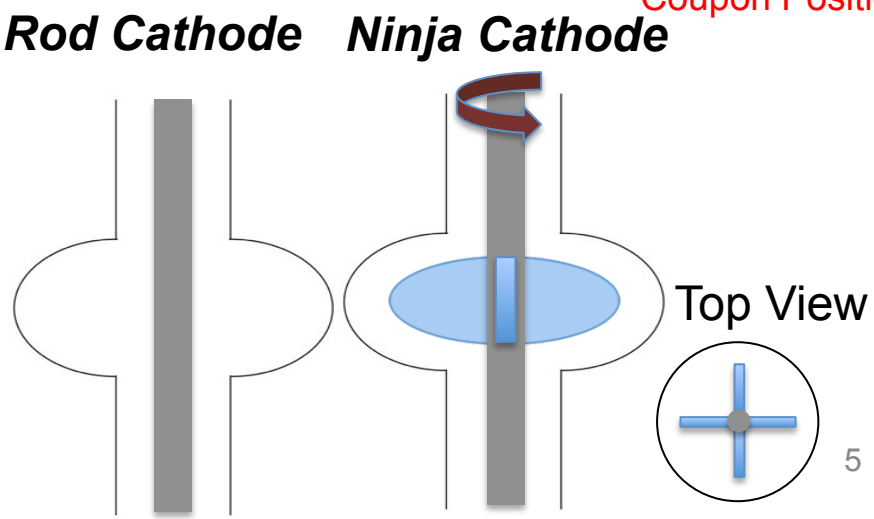




Red Arrows Show Coupon Positions

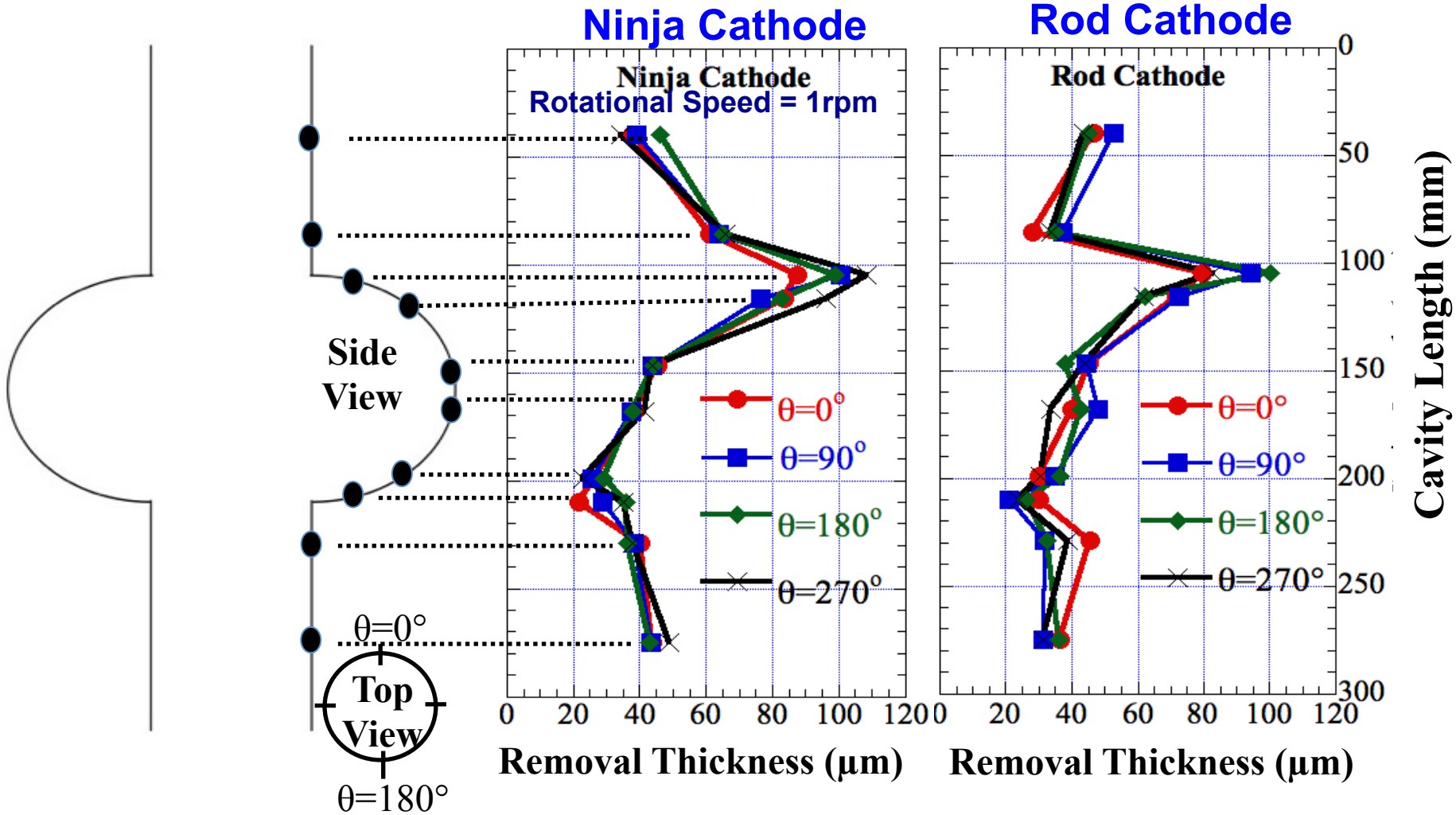
Ninja Cathode

- Marui Galvanizing developed a unique cathode called Ninja cathode.
- The Ninja cathode has 4 retractable Al wings for agitation and uniform EP over the cavity.



Inhomogeneous Removal Thickness with VEP

- Removal thickness was measured with ultrasonic thickness gauge.

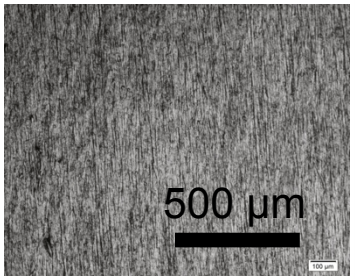


- Removal thickness was the highest at the top iris and the lowest at the bottom iris.
- The higher EP rate on the top iris was due to H_2 bubbles attack and gravity which reduce viscous layer thickness on the top iris.

  (Effect of H_2 bubbles was proved in lab EP experiments presented in TTC meeting 2014)

Roughness and Microscope Images of Coupons

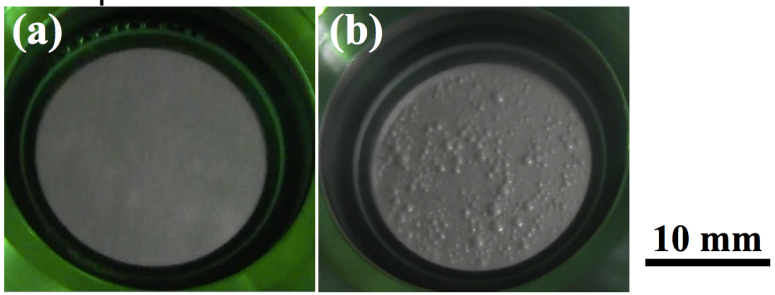
Before VEP

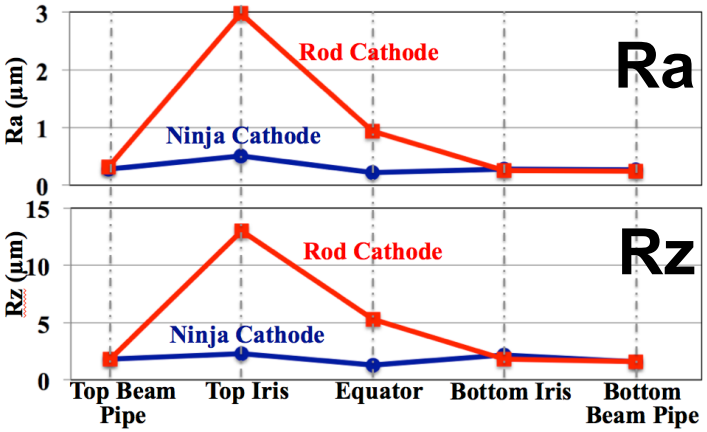


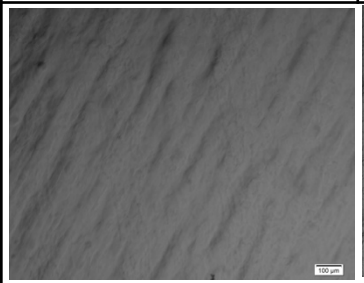
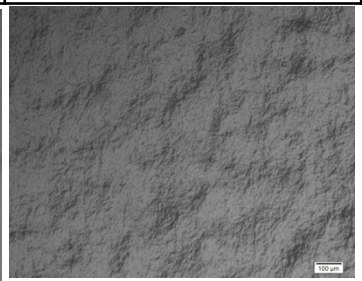
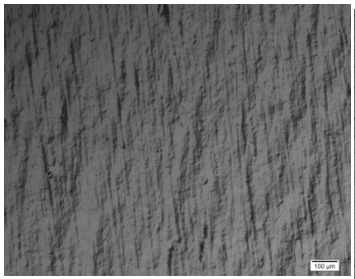
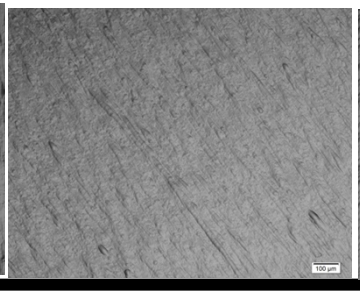
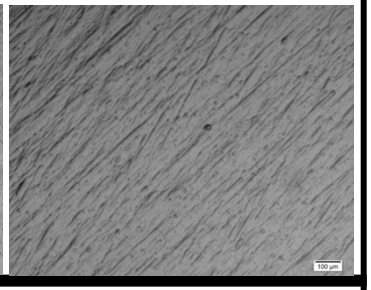
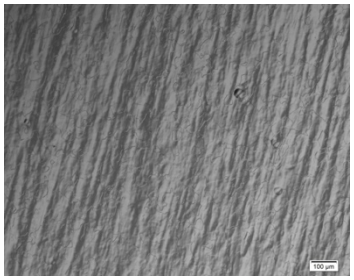
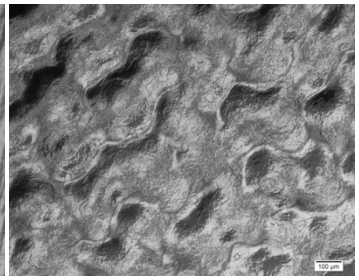
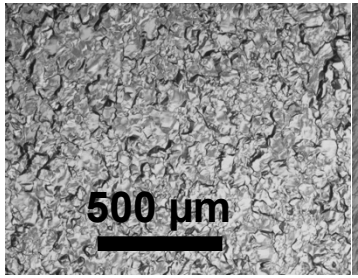
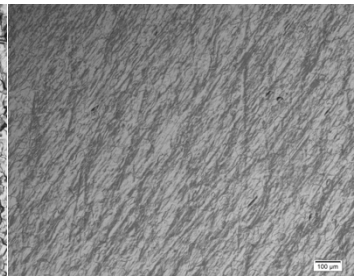
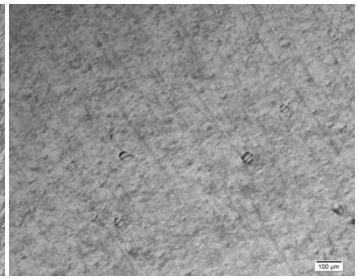
Top Iris View Port

Just after a wing passed

Rod cathode





	Top Beam Pipe	Top Iris	Equator	Bottom Iris	Bottom Beam Pipe
NINJA					
ROD					

- The rod cathode enhanced surface roughness at the top iris and equator.
- The bubbles attack on the top iris surface microscopically and make the surface rough.

Effect of Stirring to Reduce Bubble Attack

❖ Lab EP experiments were performed with and without stirrer for comparison.

Sample Positions

Top

Cathode

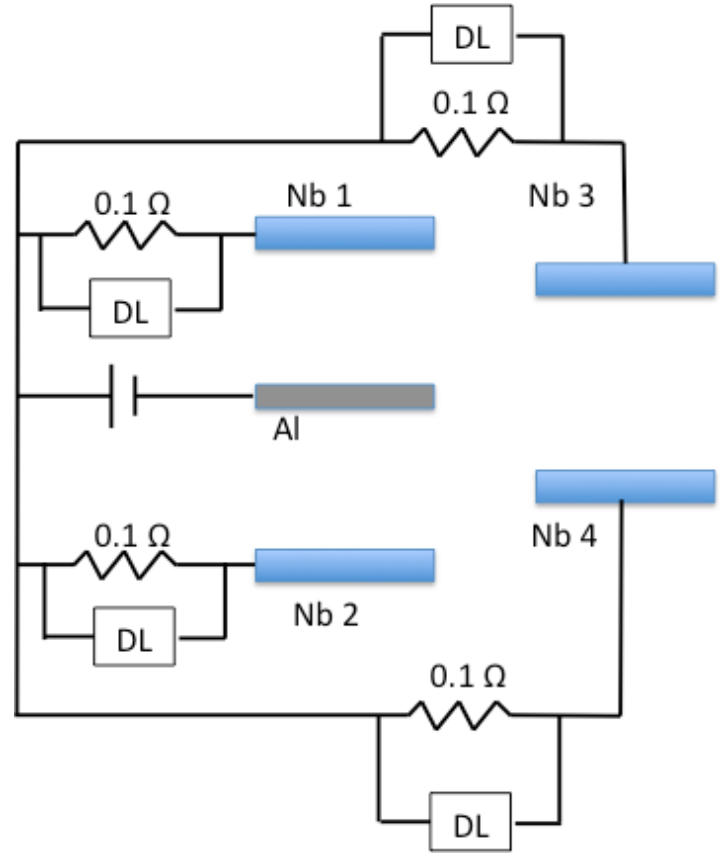
Bottom
(face up)

Side Top

Side Bottom
(face down)

EP Bath

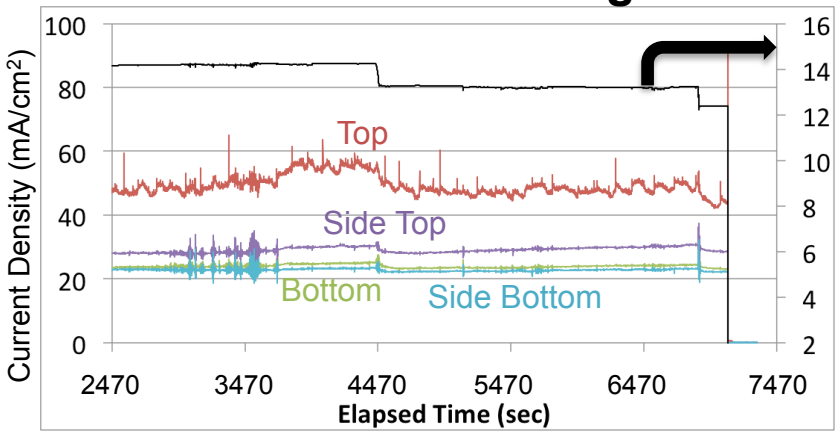
Stirrer

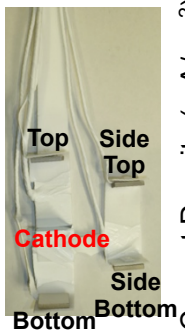


- EP Solution Temperature <22 °C
- Voltage: 11-14 V
- Average Current Density: 30-35 mA/cm^2

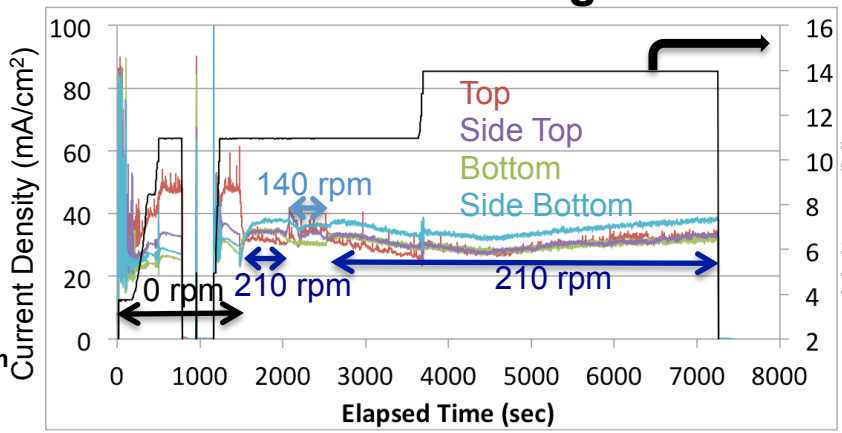
Current Profiles and Surface w/o and w/ Stirring

Without Stirring



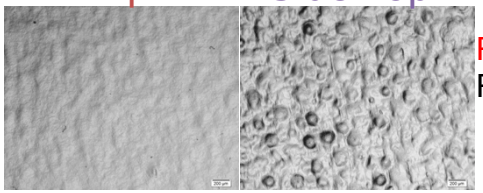


With Stirring



Top | Side Top

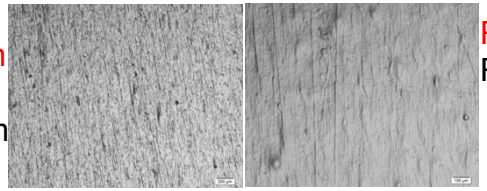
RT = 77.8 μm
Ra/Rz
=0.25/1.3 μm



RT = 47.5 μm
Ra/Rz
=0.80/4.6 μm

Bottom | Side Bottom

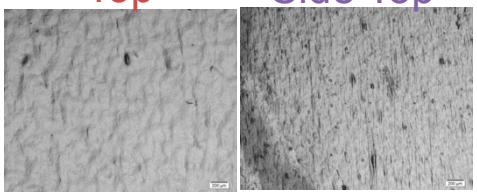
RT = 40.7 μm
Ra/Rz
=0.36/2.5 μm



RT = 39.0 μm
Ra/Rz
=0.29/2.2 μm

Top | Side Top

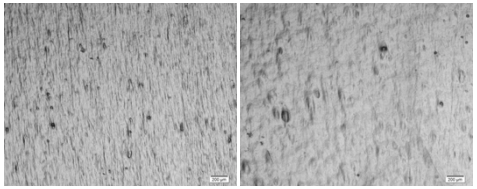
RT = 47.6 μm
Ra/Rz
=0.44/2.2 μm



RT = 44.3 μm
Ra/Rz
=0.39/2.4 μm

Bottom | Side Bottom

RT = 45.0 μm
Ra/Rz
=0.32/2.0 μm



RT = 46.7 μm
Ra/Rz
=0.31/1.8 μm

- EP current for the top sample was the highest because of thin viscous layer occurred by direct hitting of bubbles.
- EP currents for the bottom and the side bottom were almost the same, meaning that gravity effect on viscous layer is not strong.

- Sample currents were found similar when the stirrer was used.
- The removal thickness was ofcourse similar for all the 4 samples.
- The viscous thickness and the bubble hitting of each sample should be equivalent.

VEP Conditions for Homogeneous EP

Based on the lab EP results, to solve the problem of inhomogeneous removal in VEP, cathode rotational speed was optimized.

- Al wings were replaced with non-metal wings.
- Rotation: 50 rpm (Coupon currents were recorded for 1, 10, 20, 30, 40 and 50 rpm)

  At higher rotational speed of cathode

  – Flow of EP solution might be uniform to generate uniform viscous layer on cavity surface

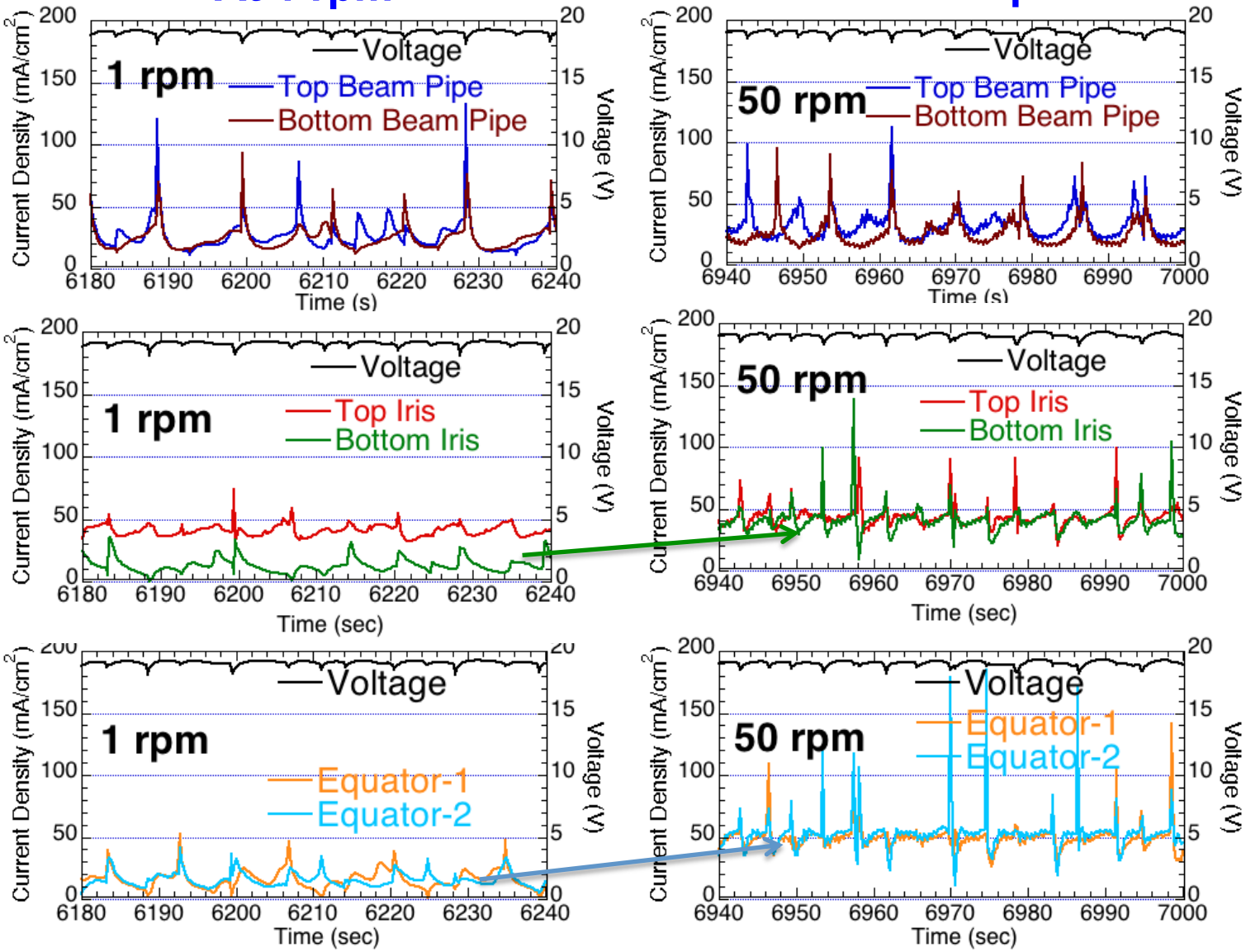
  – H_2 bubbles can be removed from the top iris

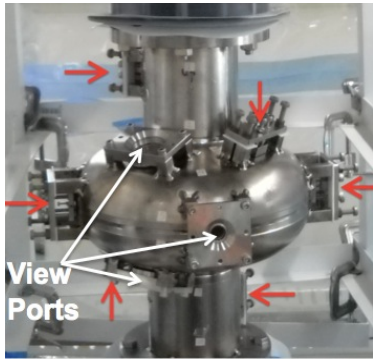
- EP Solution Flow Rate: 5 l/min
- Voltage: 12-20 V
- Current density: ~30 mA/cm^2
- EP tank temperature: 16-27 °C
- EP time: 2 hours
- Target removal thickness=50 μm

Coupon Current Profiles at 1 and 50 rpm

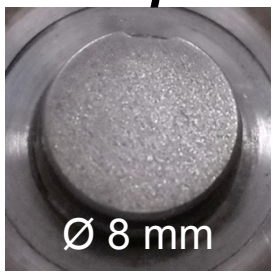
At 1 rpm

At 50 rpm





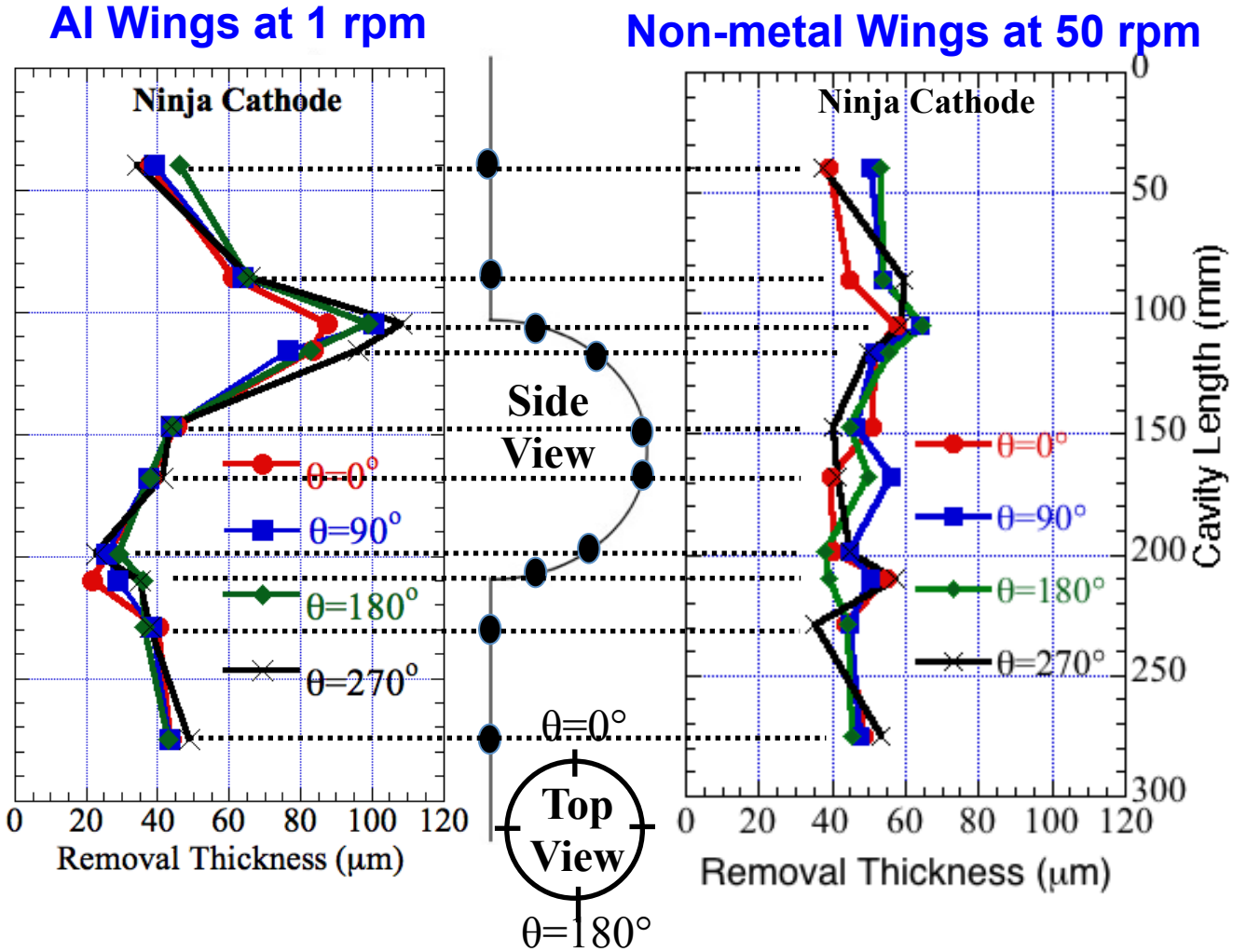
Coupon



- The top iris coupon current was the highest at 1 rpm because of thin viscous layer on the top iris.
- The bottom iris current increased with increase in cathode rotational speed and became similar to the top iris current at 50 rpm.

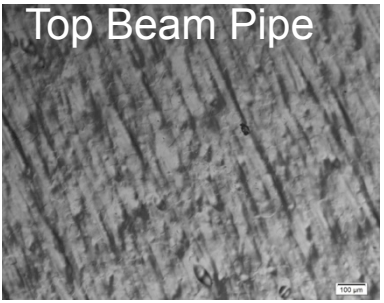
Removal Thickness of the Cavity and Coupons

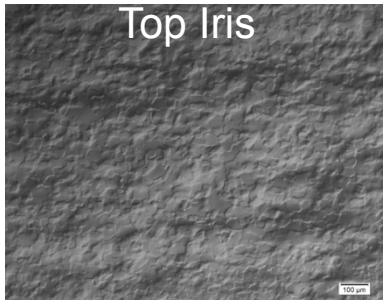
- Average cavity removal thickness (from cavity weight loss) = 55 μm

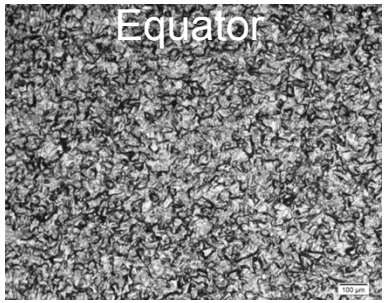


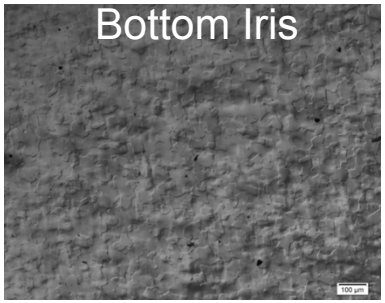
- The removal thickness was almost homogeneous on entire cavity including the top and the bottom irises.
- The high rotation speed might generate uniform viscous layer on the surface of cavity cell since the high flow of EP solution remove H_2 bubbles from the top iris.

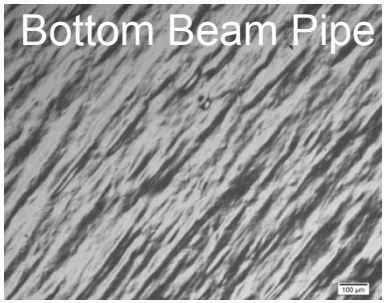
Surface of Coupons (50 rpm)



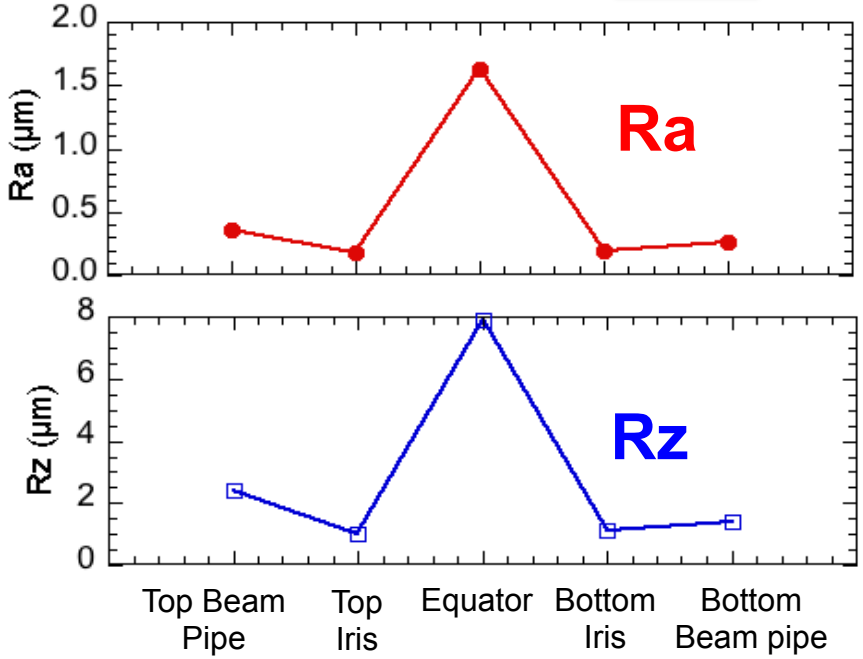




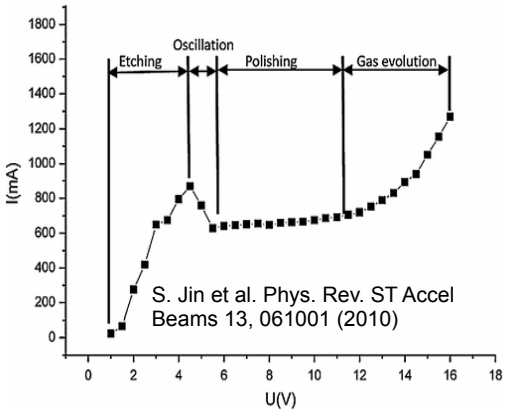




500 µm

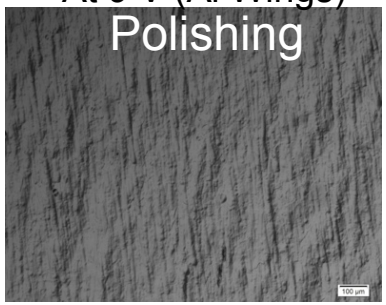


Equator surface with Al wing cathode

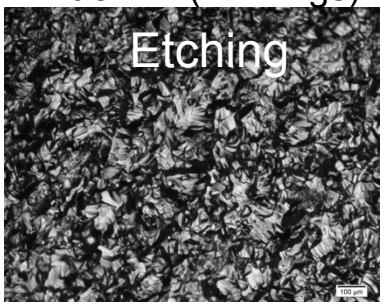


Etching of Nb gives rough surface.

At 9 V (Al Wings)



At 3.1 V (Al Wings)



- All the coupons except equator were found very smooth and shiny after the VEP.
- This rough equator surface might be result of etching instead of polishing.
- Electric field on equator might be lower in absence of Al wings and the lower electric field might be cause of etching.
- High flow of acid on equator surface might disturb passive layer generation and shift polishing region to the higher voltage side.

XPS Analysis

- The coupon cavity after VEP was rinsed with water in an ultrasonic bath.
- The coupons were disassembled and analyzed.
- X-ray photoelectron spectroscopy was used to analyze the top surfaces of the coupons.
- The analysis area was kept large as 700 X 300 μm^2.

Atomic composition on the top surfaces of the coupons:

Element	Top Beam Pipe	Top Iris	Eqator	Bottom iris	Bottom Beam Pipe
Nb	18.4	18.7	23.1	17.3	22.6
O	42.4	45.6	39.8	40.2	45.4
C	37.6	33.7	36.0	41.2	30.7
F	0.1	0.1	<0.2	0.1	<0.1
S	0.7	1.4	0.5	0.8	0.8
Si	0.4	0.4	0.4	<0.4	<0.4

- As contaminants sulfur, fluorine and silicon were found.
- Small amount of sulfur and fluorine are usually remain on surface after EP.
- However sulfur can be rinsed with some detergents like FM-20. (For the above coupons FM-20 rinsing was not performed)
- The source of Si is unknown now.

Summary

- VEP setups were made with PVC material.
- PVC made systems can be used in industries for mass production and cost reduction of the cavity surface treatment.
- In VEP, removal thickness was always found to be inhomogeneous along the cavity length.
- Homogeneous EP rate was obtained with a high rotational speed of the Ninja cathode.
- Coupon surfaces except equator coupons were found to be very smooth and shiny at high rotation speed of the Ninja cathode.
- The rough equator surface might be due to lower electric field and higher flow of acid resulting etching instead of polishing.
- XPS analyses showed very less amount of contaminants including S, F and Si.

Future Work

- Cathode will be improved for further smooth surface especially the equator surface.
- 1-cell and 9-cell cavities will be VEPed using the optimized parameters and vertical test will be performed.

Acknowledgement

We acknowledge H Hayano, T Saeki, M Sawabe, H Monjushiro, K Ishimi and S Arata.

Thank You