Novel Characterization of the Electropolishing of Niobium with Sulfuric and Hydrofluoric Acid Mixtures

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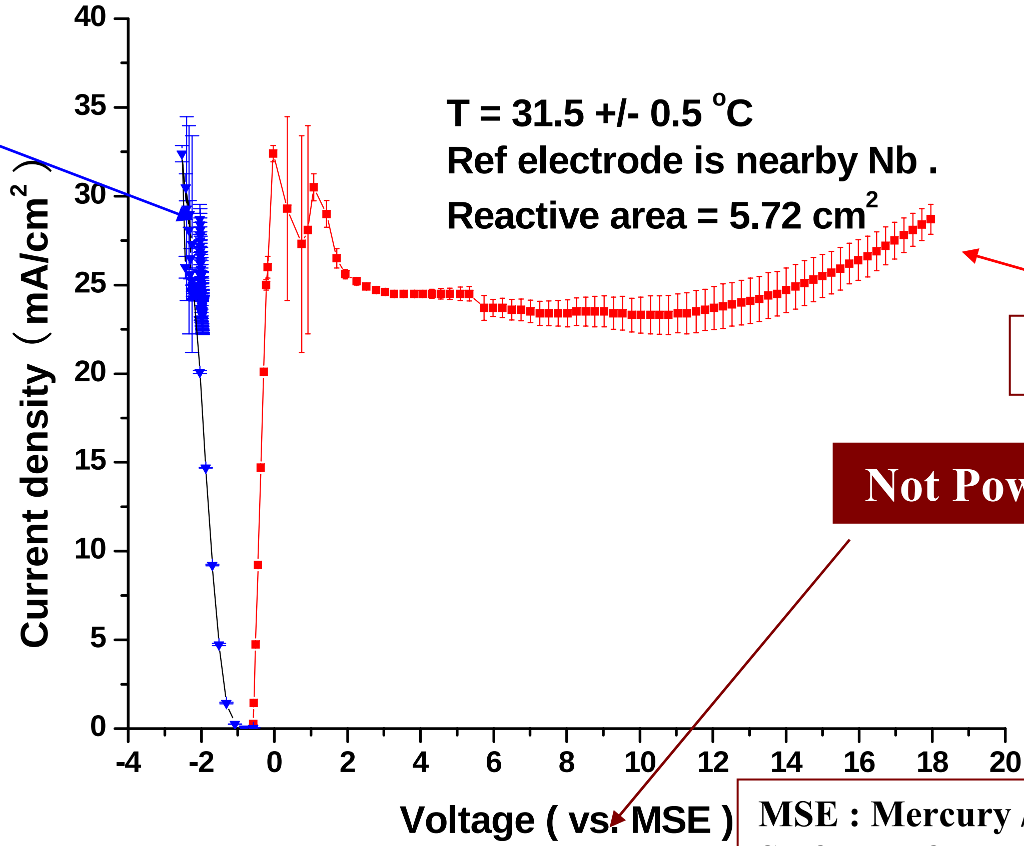
Three-Electrode-Setup Improved Electrochemical Characterization of EP



MSE : Mercury / Mercurous Sulfate Reference Electrode

Example:

V_{PwrSup} = 15 V

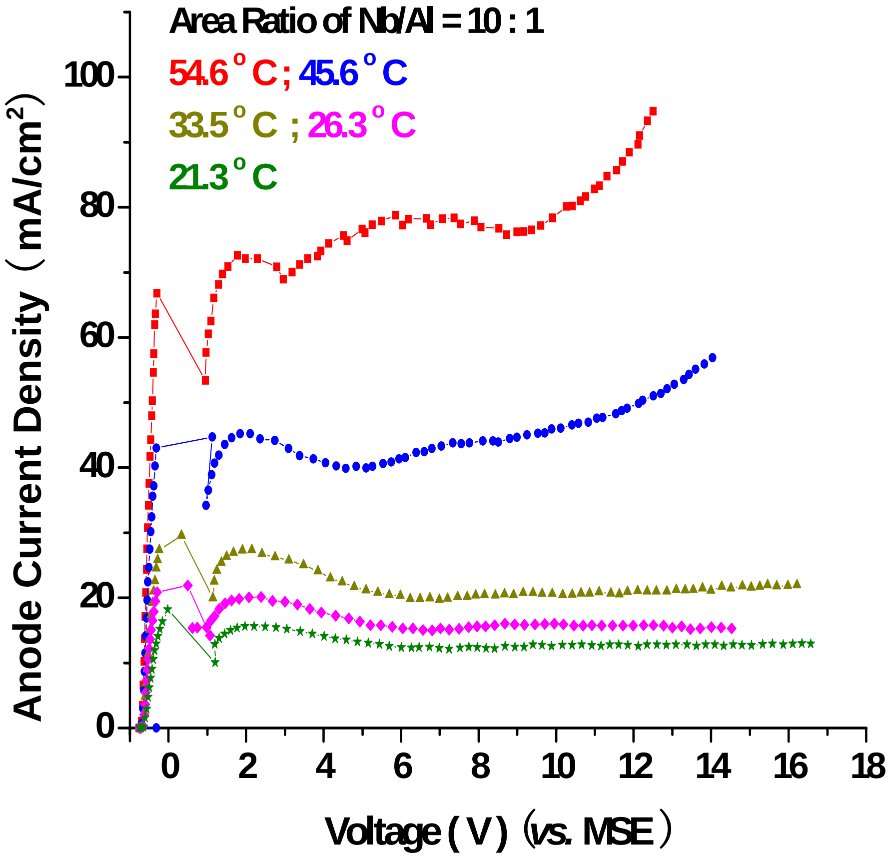
$V_{cathode}$: ~ 4 V

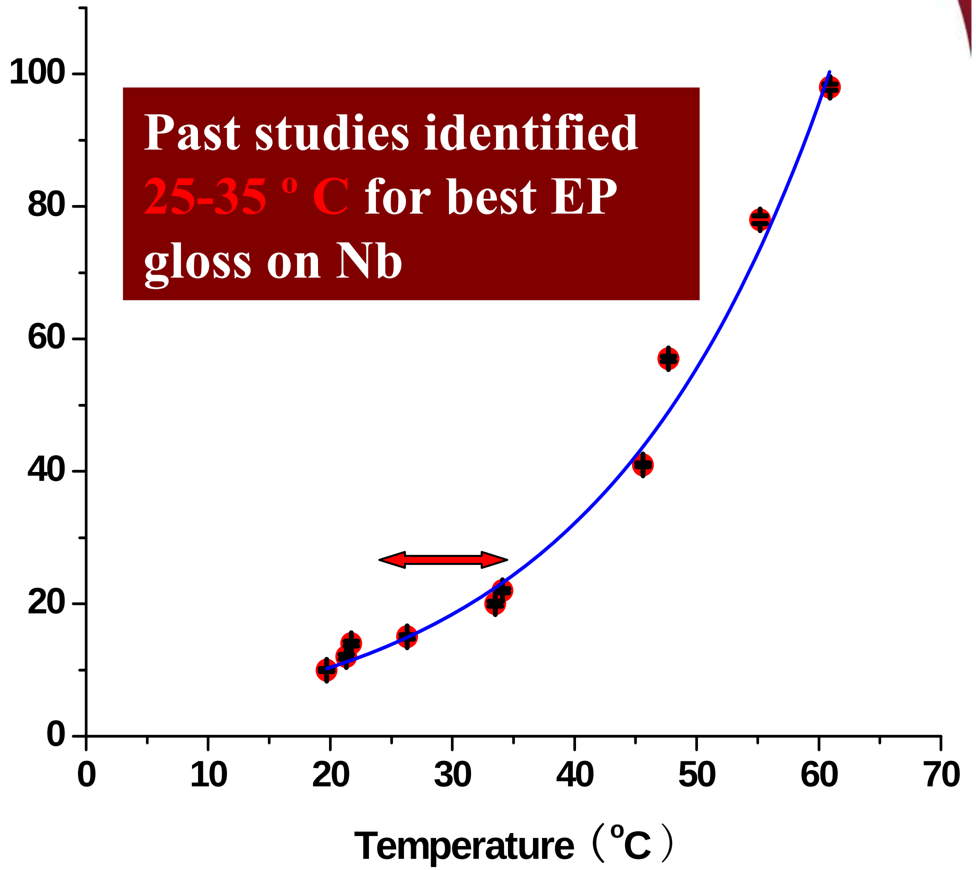
$V_{electrolyte}$: ~ 2 V

V_{anode}:~ 9V

Separating impacts of individual components in EP system. → **Enables enlightening study of temperature, flow, and composition dependent effects (electrolyte) in detail.**

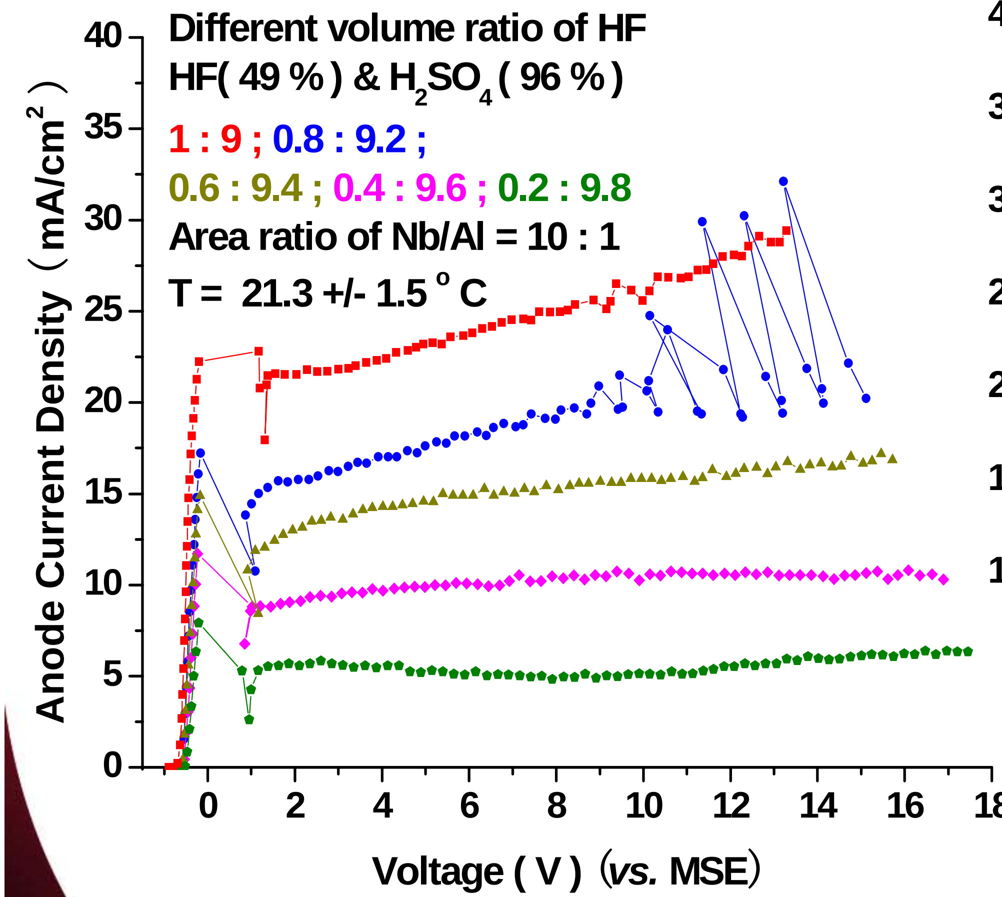
Local Current Density Strongly Depends on Local Temperature

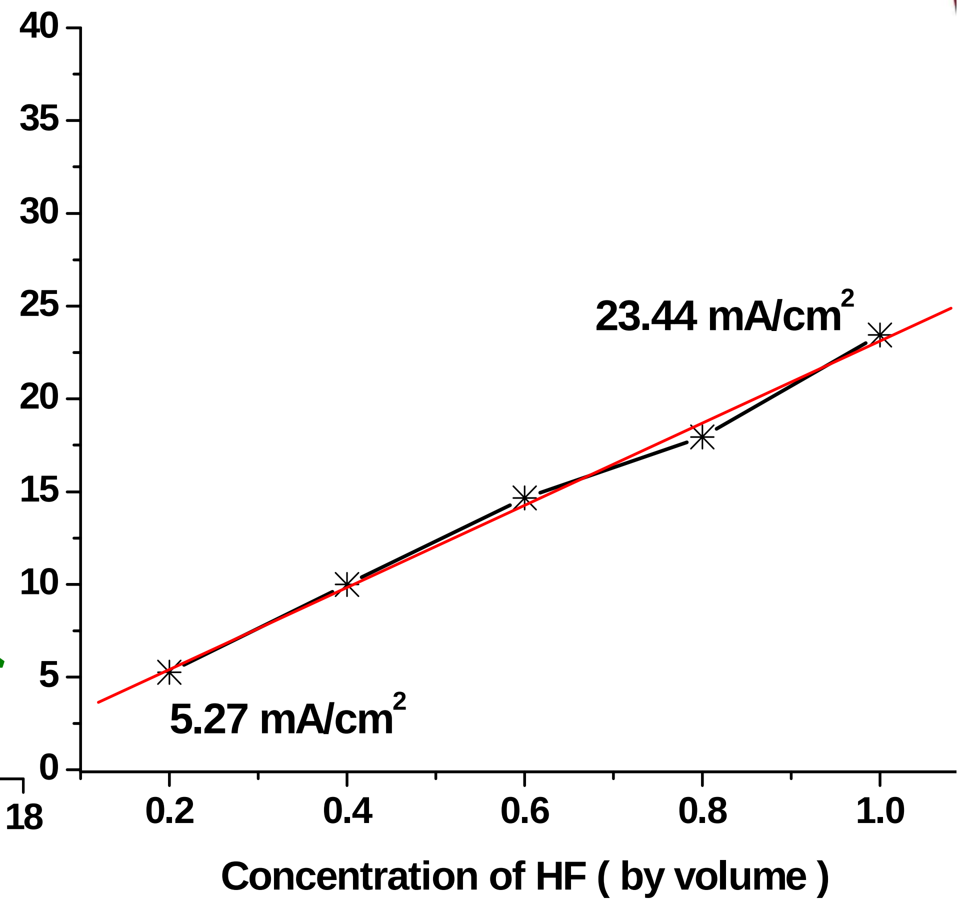




For cavity EP, expect unstable temperatures when the electrolyte also serves as the process coolant, and particularly hot in no-flow condition and higher heat flux where flow rate is high , so non-uniform polishing effect is expected

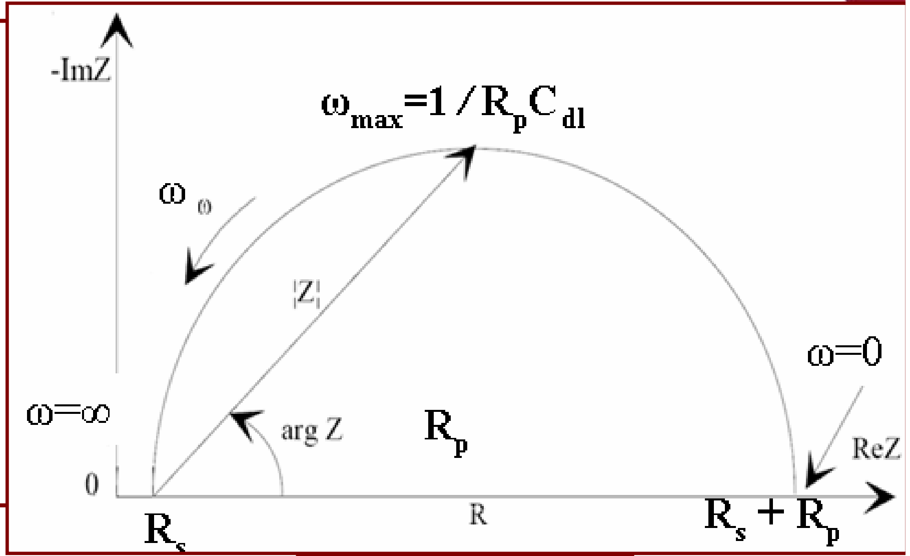
Anode Current Density Varies Linearly with HF Concentration





What is Electrochemical Impedance Spectroscopy?

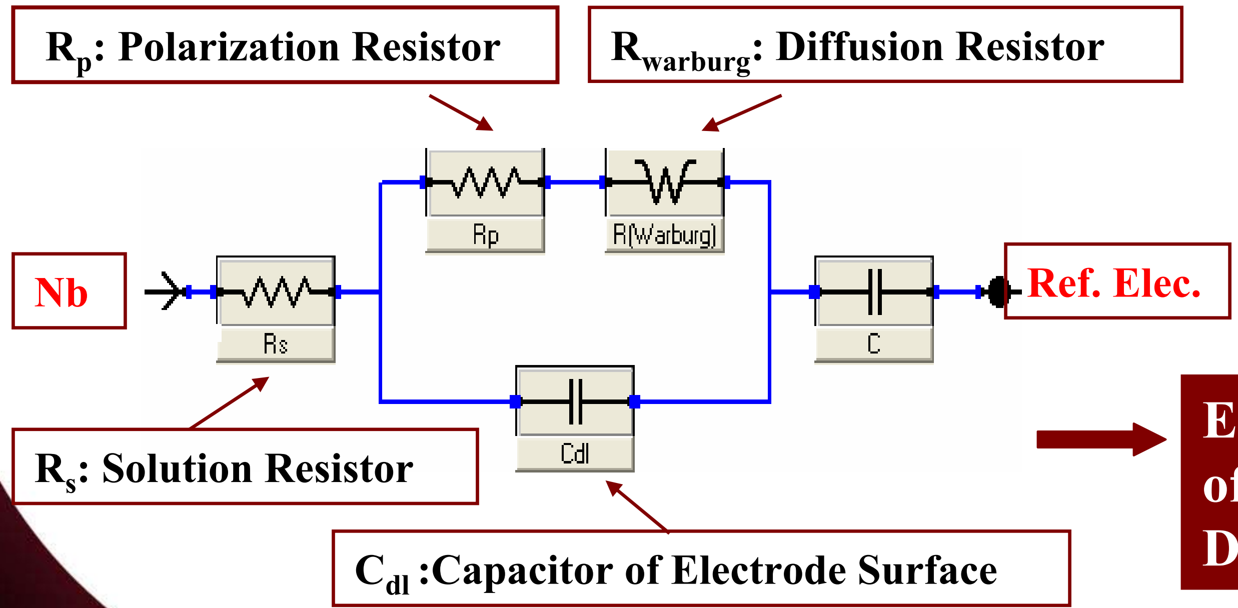
- Investigate the electrical dynamics of niobium-acid interaction during electropolishing.
- EIS : 10 mV variable-frequency ac superimposed on normal dc polarization voltage; record the impedance at the different frequency.



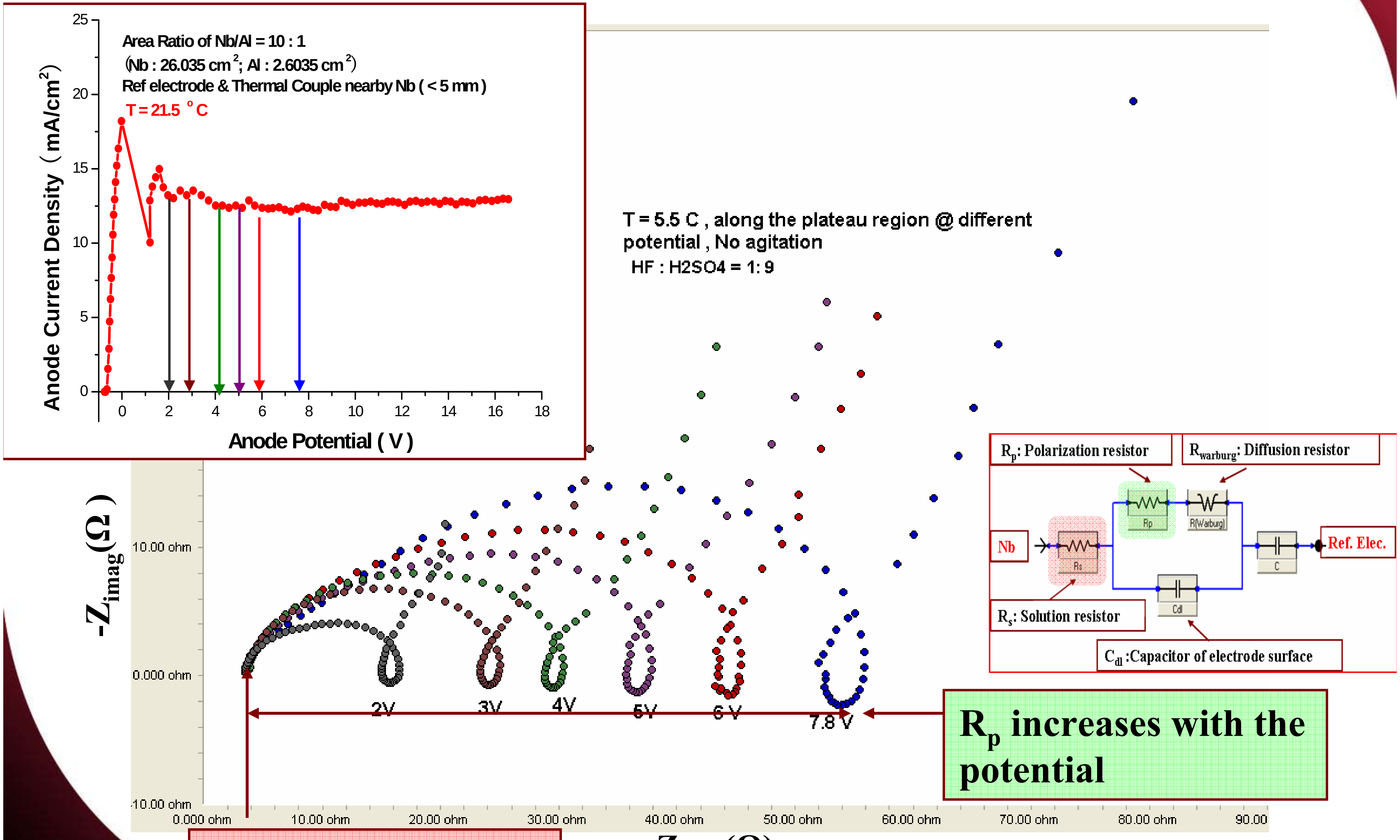
Nyquist Plot

EIS

Equivalent Circuit of Nb-Acid Interface During EP



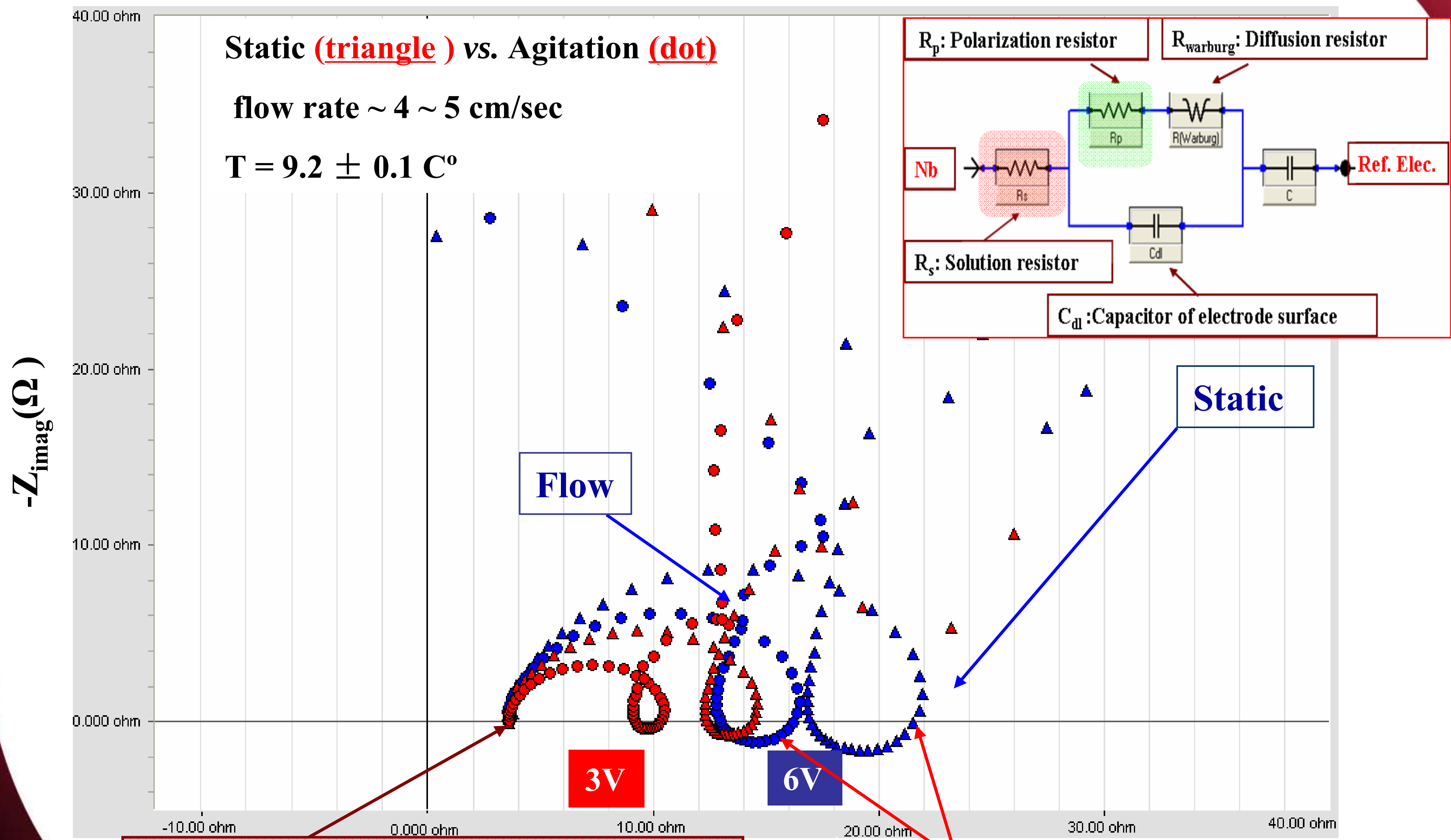
EIS Study of Constant Current Density



R_p increases with the potential

R_s remains constant

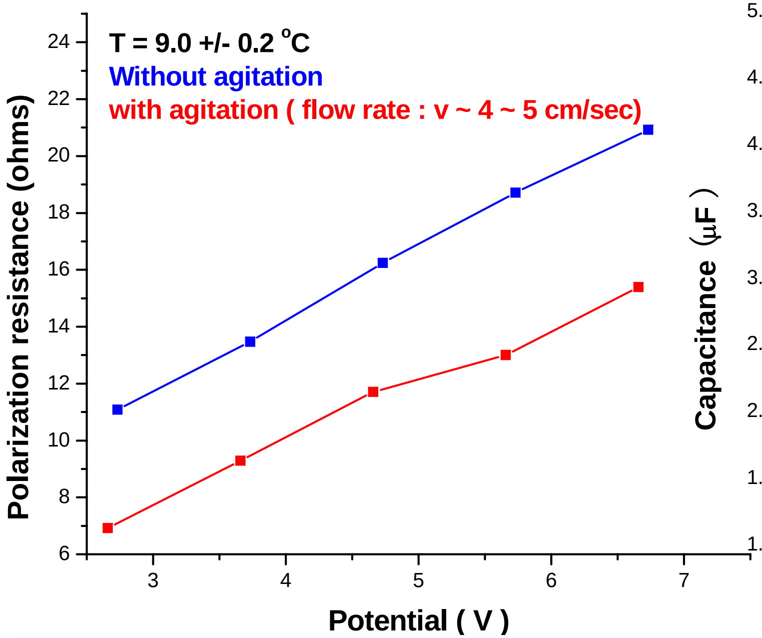
EIS Study of different flow rates

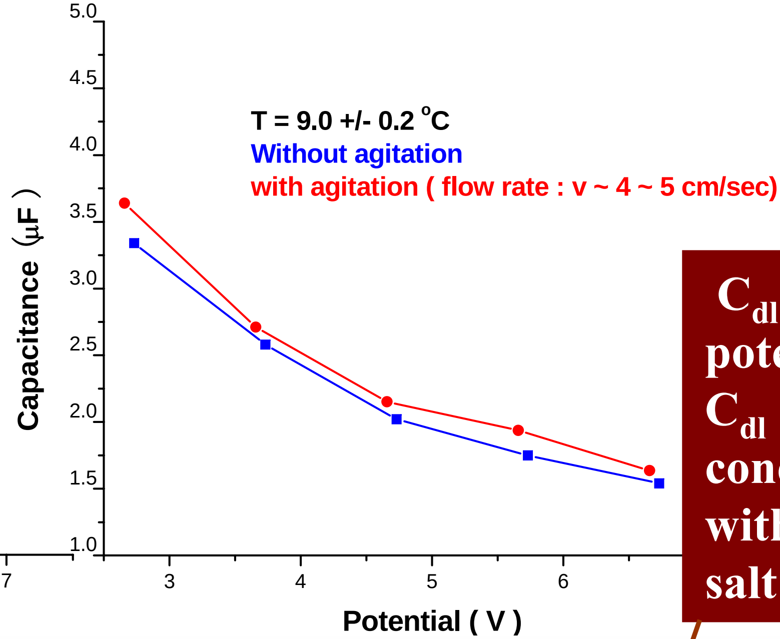


Rs @ at different flow condition remains as constant

Rp decreases with increasing flow

What We have Learned from EIS Studies?





C_{dl} ↓ @ different potential regions & C_{dl} ↑ @ different flow conditions is consistent with the "compact salt film" model

Characteristic feature @ the high frequency (* C = constant)	Salt Film Models						Adsorbates Acceptor Model		
	Porous Film			Compact Film					
	R_s	R_p	C_{dl}	R_s	R_p	C_{dl}	R_s	R_p	C_{dl}
Different Potential (↑)	↑	C	C	C	↑	↓	C	C	C
Different Rotation (↑)	↓	↓	C	C	↓	↑	C	↓	C

Constant R_s @ different potential regions and flow condition rules out the "porous salt film" model

R_p ↑ @ different potential regions is inconsistent with the "adsorbates acceptor" model

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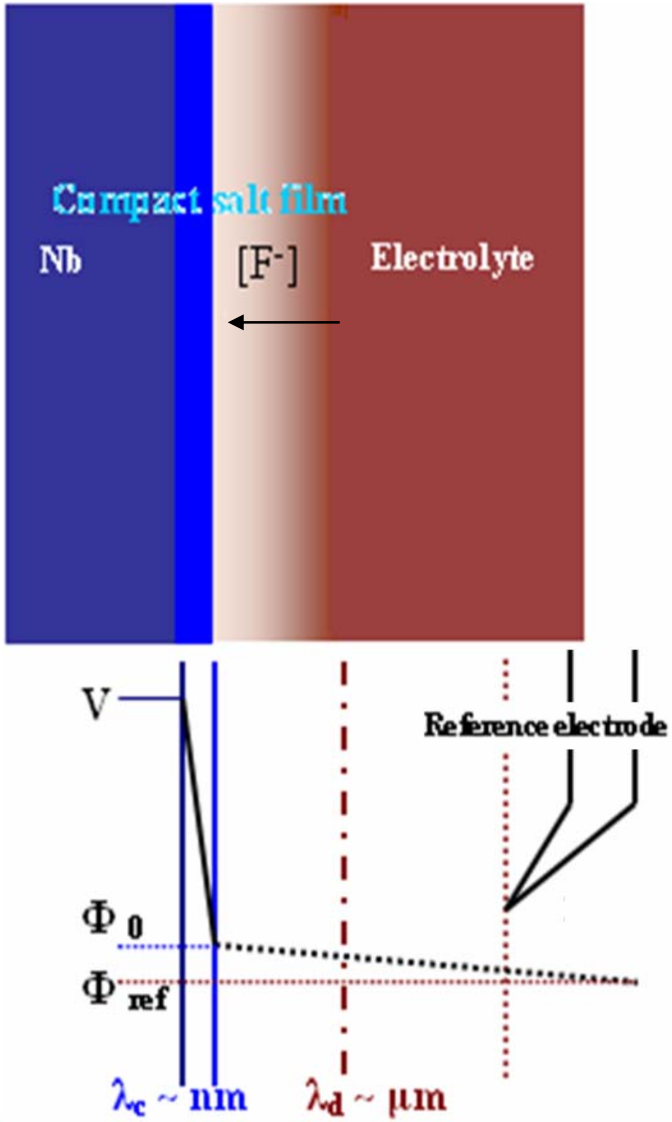
EIS Indicates “Compact Salt Film” Model



➢ Sulfuric tends to anodize the Nb under polarization potential producing the "compact salt film”- “Nb_2O_5”.

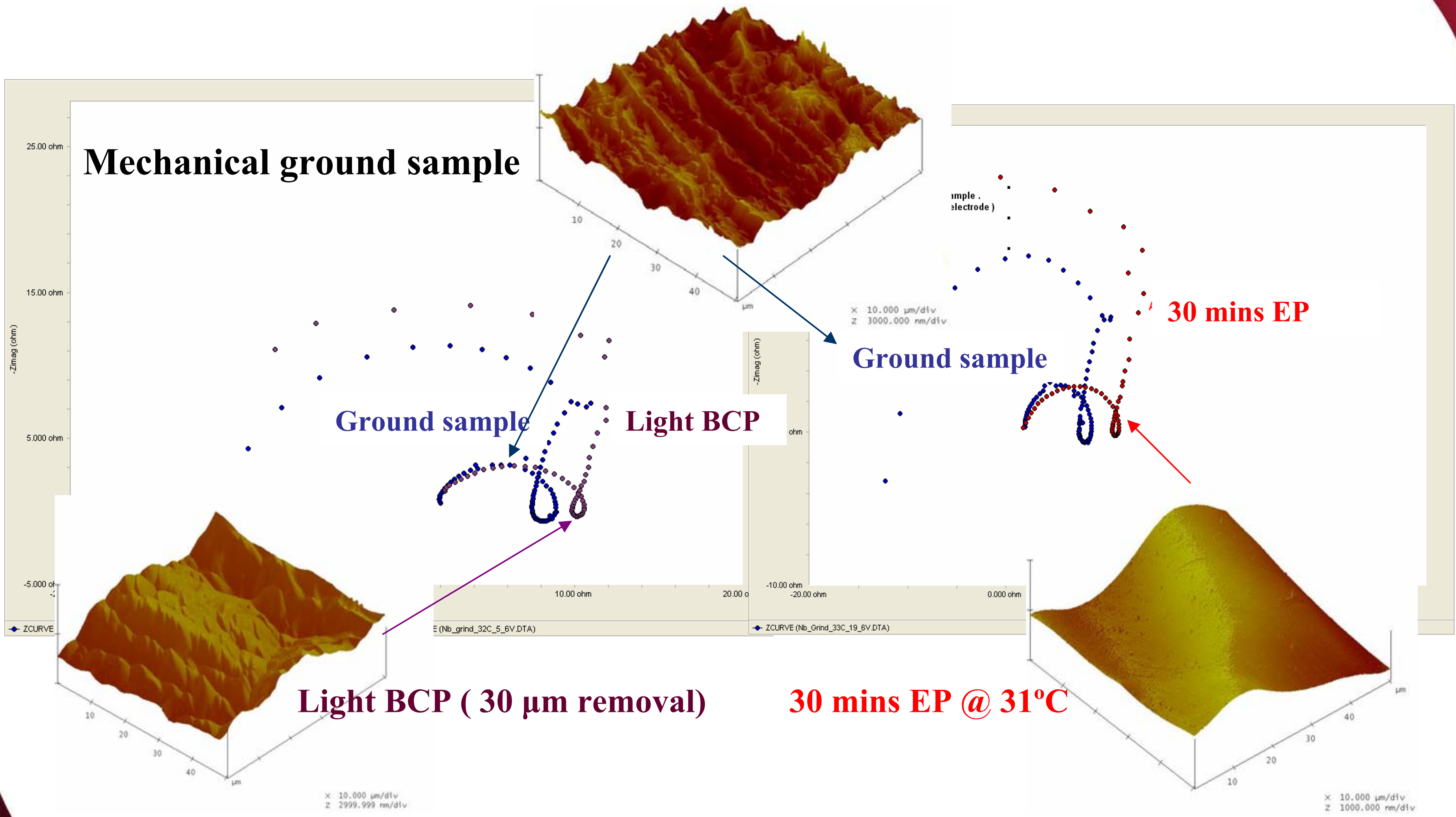
➢ HF tends to dissolve the Nb oxide under kinetic control with the "at the surface" concentration of F^-.

➢F^- concentration “at the surface” is limited by how fast it diffuses through the electrolyte - thus the plateau current.

➢The local gradient in F^- concentration produces the desired polishing action.

Thank You

Preliminary Small Sample EIS Study for Implication



There is a signature difference in EIS response between rough and smooth surfaces.

Potentially useful for on-line process feedback

WEP04 "Surface Roughness Characterization of Niobium Subjected to Incremental BCP and EP Processing Steps"
H. Tian , et. al

Future Work on Small Sample EP

- EIS study with different concentration HF
- Monitor polishing effect with different concentration HF
- Monitor polishing effect with different flow rate
- Monitor polishing effect with different temperature electrolyte
- Simulation