

Surface Studies of Niobium Chemically Polished Under Conditions for SRF Cavity Production

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- Reproducibility Studies about Surface Roughness & Composition
- Effect of BCP Solution Flow Rate
- Polycrystal *vs.* Single Crystal



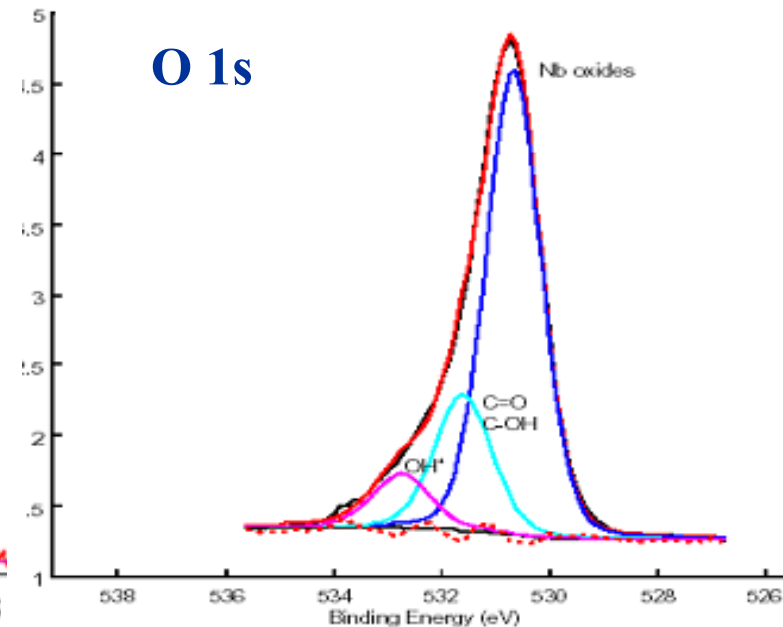
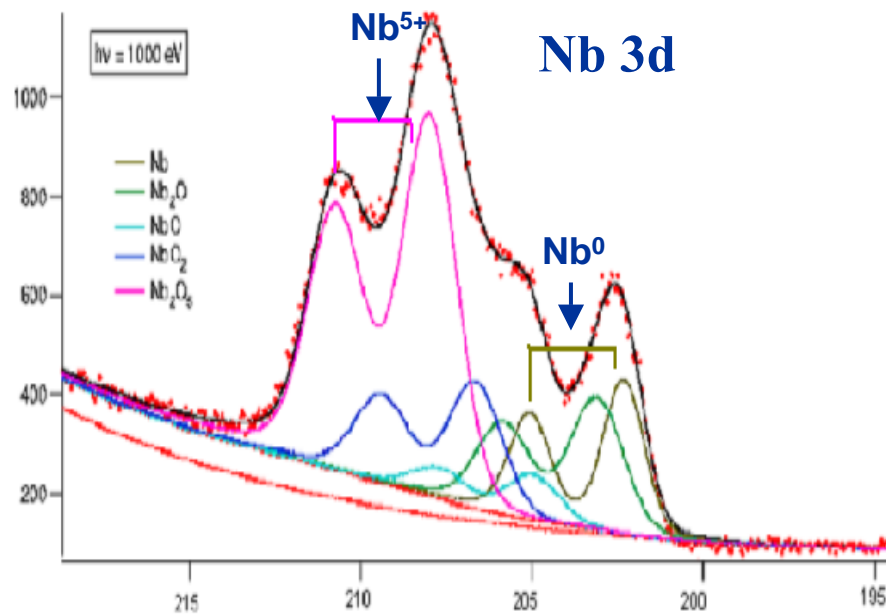
Experimental Work

- ❑ Fresh BCP (1:1:2) etch , 6 samples/batch, room temperature.
- ❑ Varied surface speed to represent flow rate difference in cavity processing: static \longrightarrow 2.0 inches/sec.
- ❑ Lab XPS analysis, 0° take off angle, 6 samples/run.
- ❑ Stylus profilometry (“Dektak”).
- ❑ Synchrotron based XPS analysis \longrightarrow NSLS , X1B.
 - $h\nu = 300 \text{ eV}, 550 \text{ eV}, 930 \text{ eV}, 1150 \text{ eV}.$
 - Take off angle = $0^\circ, 41^\circ, 60^\circ$.
 - Spot size $< 250 \mu\text{m}$ with enough intensity



Experimental Work

□ XPS analysis : Nb 3d & O 1s spectra



Reproducibility Studies

What differences are due to experimental scattering?

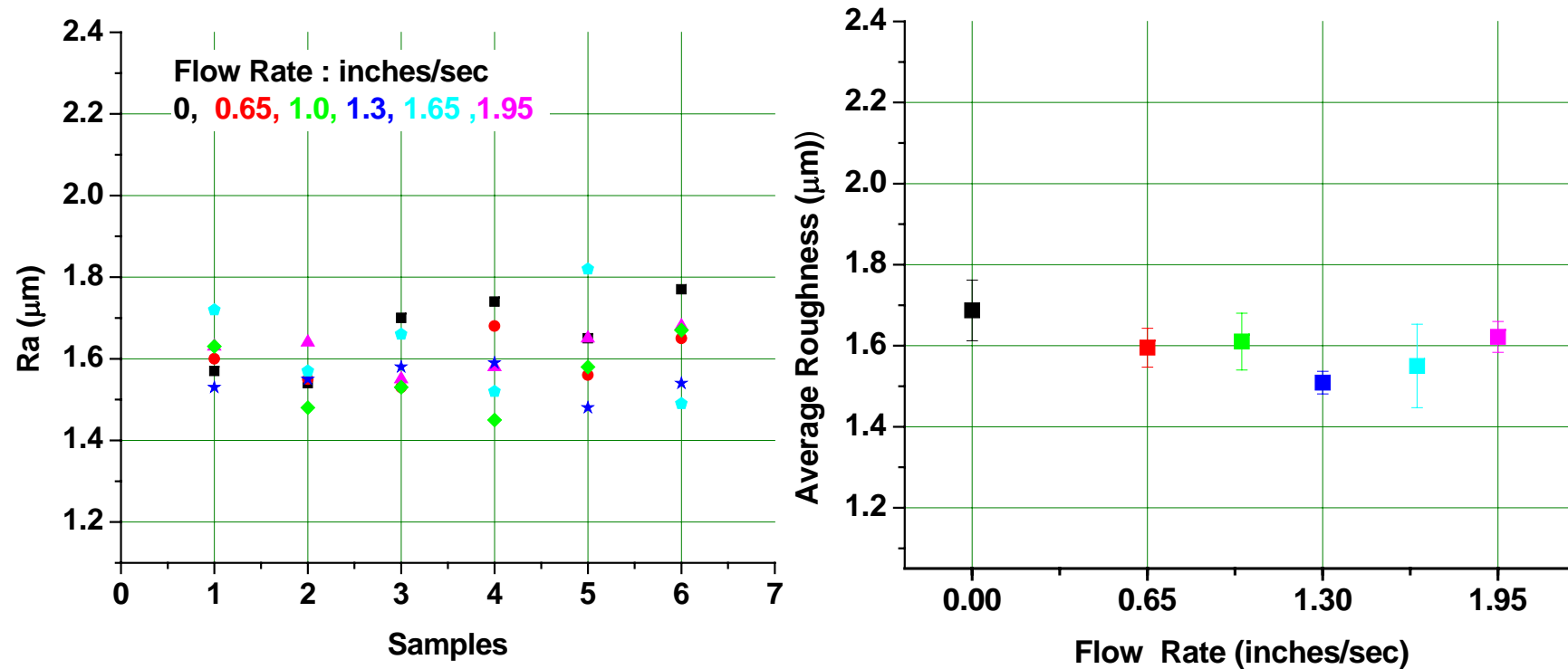
□ **Sample-to-Sample variation**

	No. (samples)	Roughness		Chemistry			
		Ra(μm)	σ (μm)	Nb ₅₊ /Nb _{total}	σ	O _{total} /Nb _{total}	σ
Sheet-7	30	1.66	0.55	0.64	0.03	1.93	0.08
Sheet-11	10	1.61	0.48	0.61	0.02	2.09	0.05

- **Batch-to-batch variation is comparable to sample-to-sample. Position-to-position variation within samples and sheet-to-sheet variation are less.**
- **Roughness values and variation all exceed the few-nm escape depth of photoelectrons.**
- **The average intensity ratios of $O_{\text{total}}/Nb_{\text{total}}$ for each sample are much smaller than 2.5 (Nb_2O_5).**



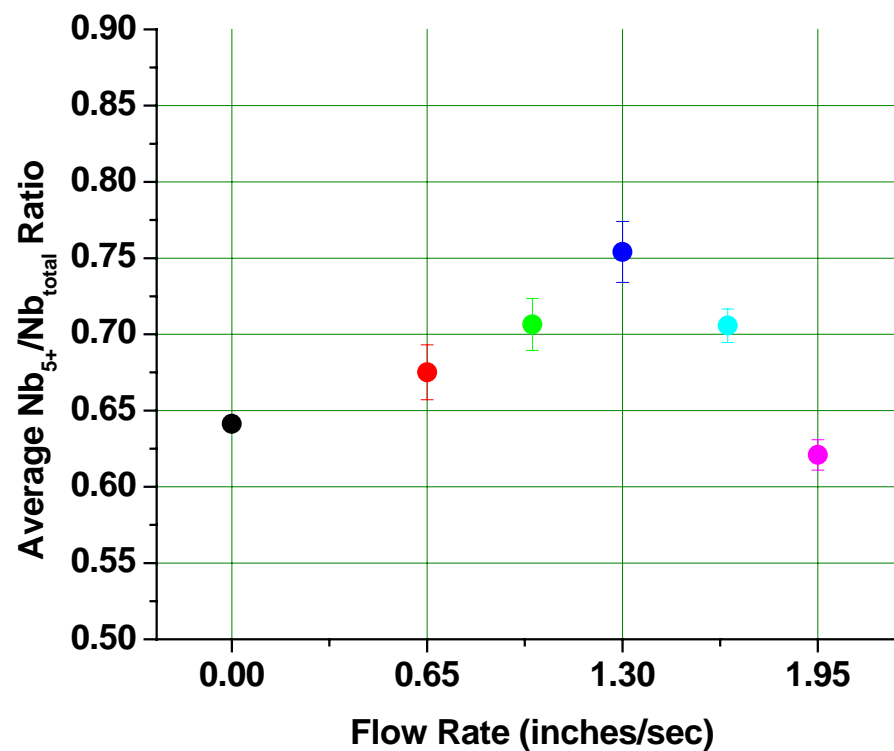
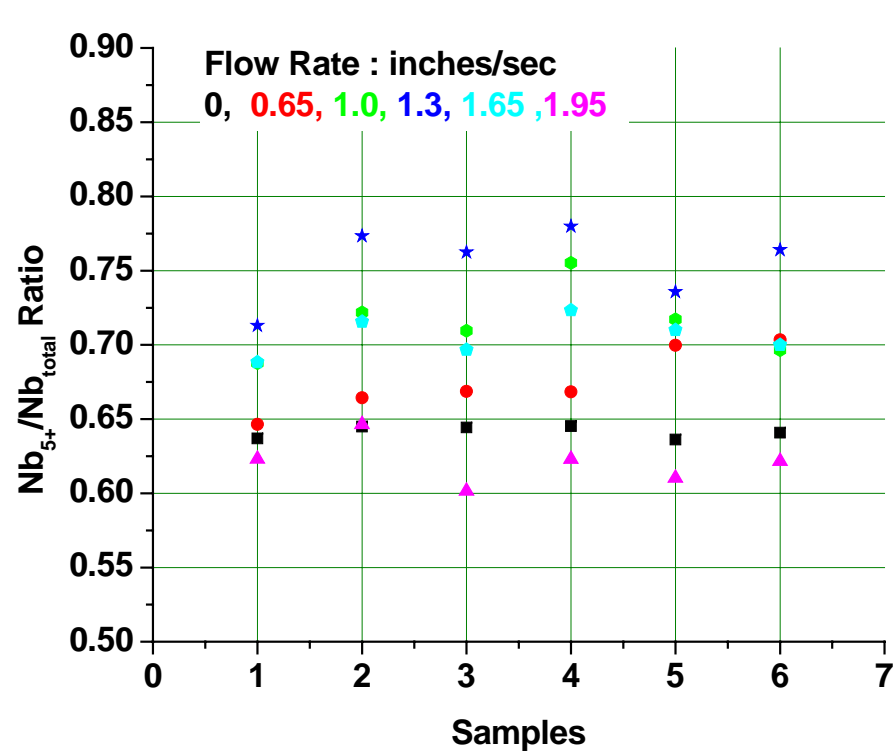
Flow Rate Dependence of Surface Topography



No significant effect of BCP flow rate on surface roughness.



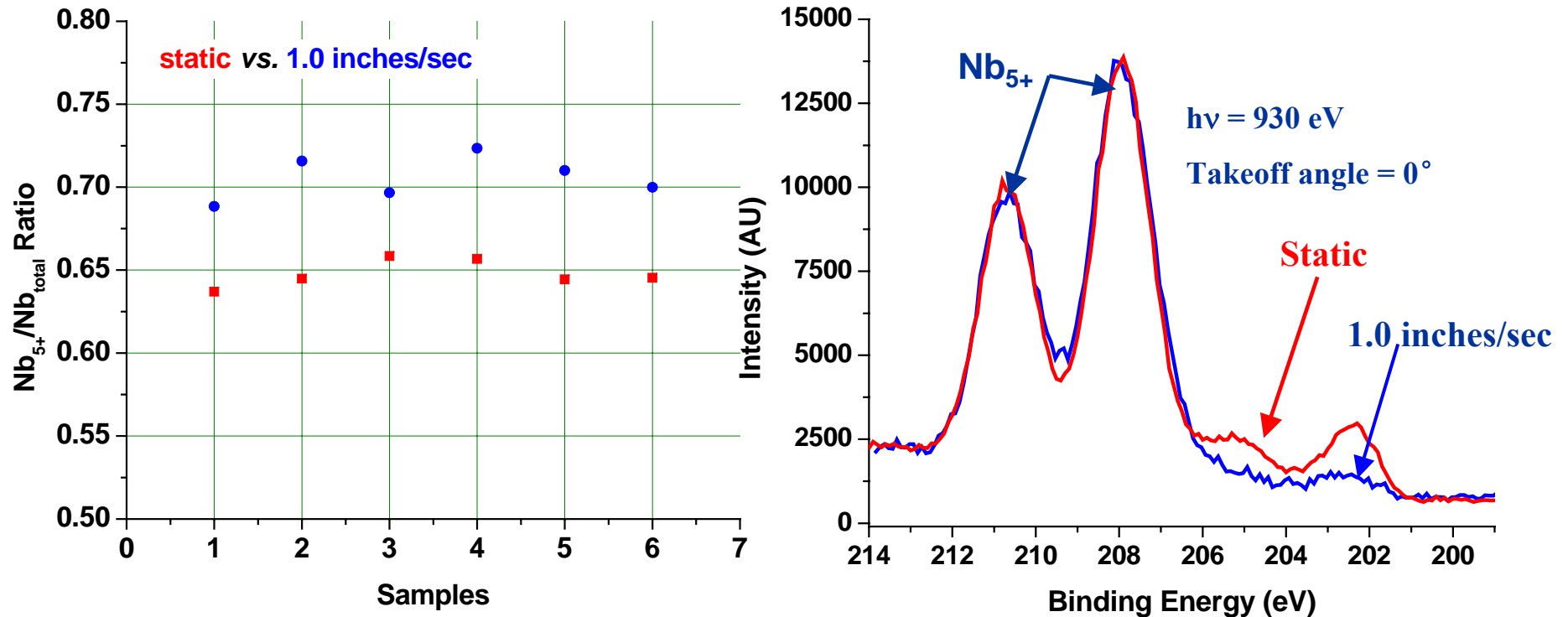
Flow Rate Dependence of Surface Chemistry



Effect of flow rate on niobium speciation is significant



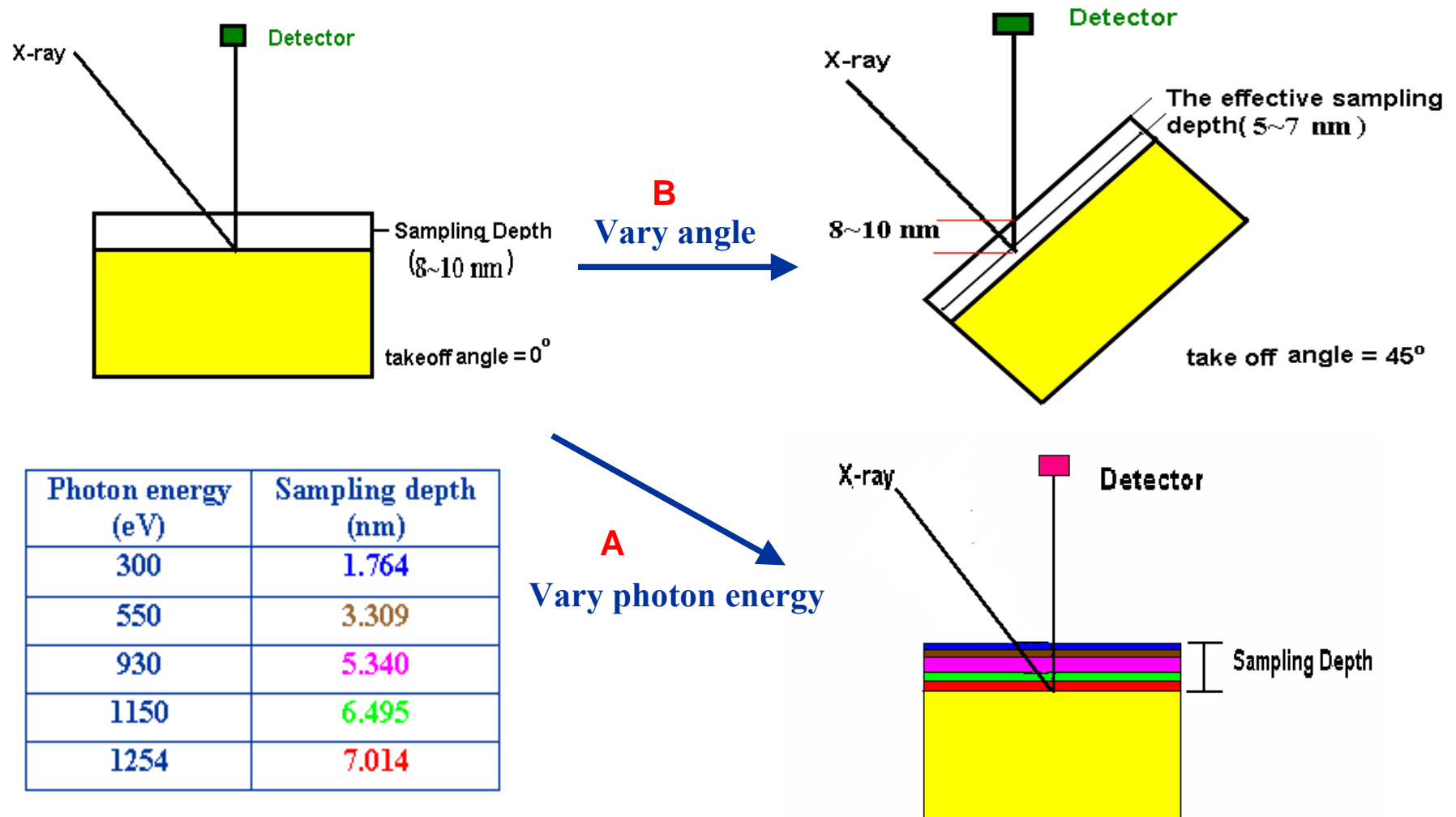
Flow Rate Dependence of Surface Chemistry-II



- ❑ Different solution flow rate causes different surface chemistry, the surface roughness shows no significant change.
- ❑ Nb_2O_5 is thicker on the high-flow sample.
- ❑ Effect of solution flow rate is being studied.

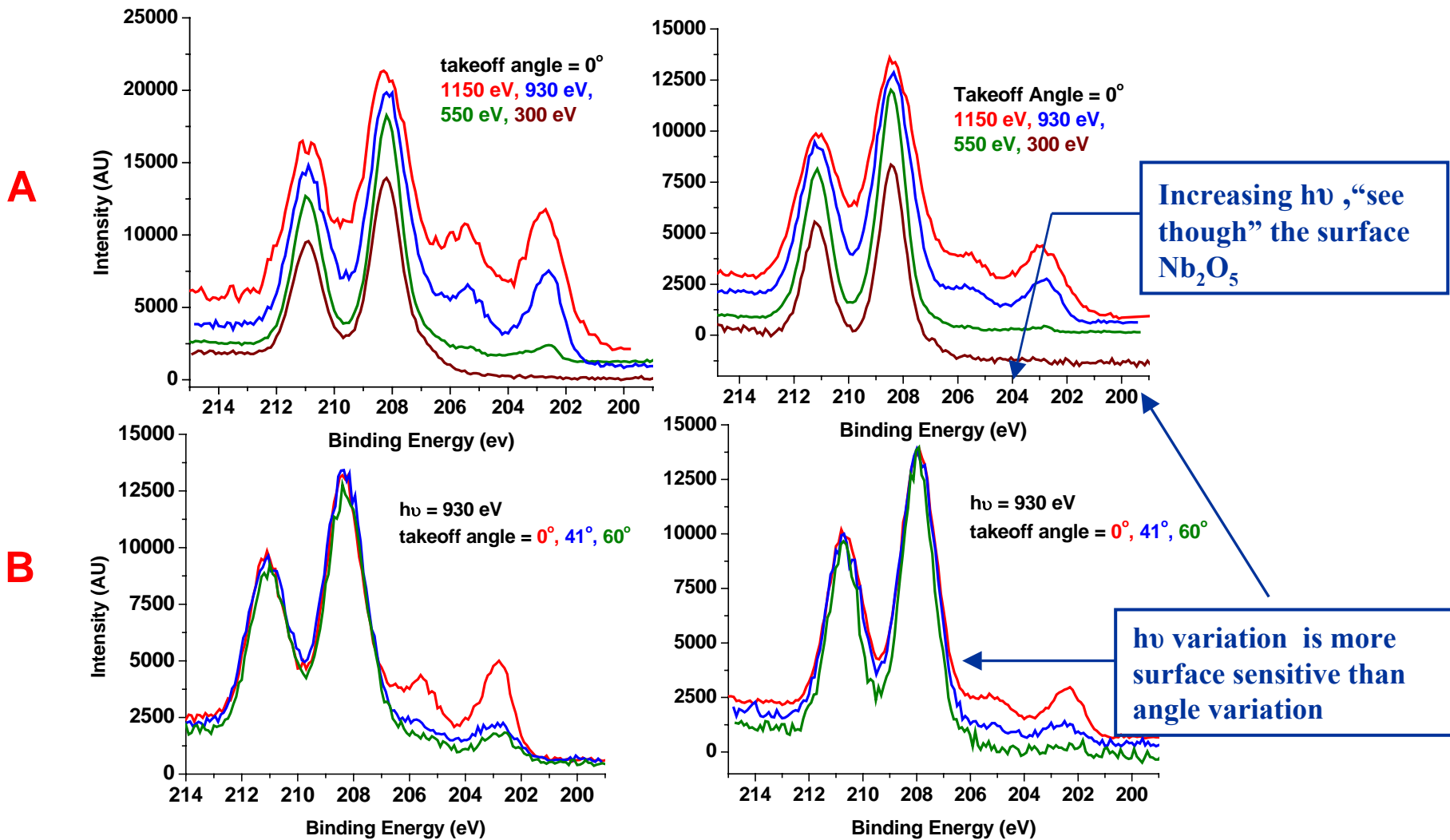


Angle Resolved vs. Variable Photon Energy XPS -Depth Profiling



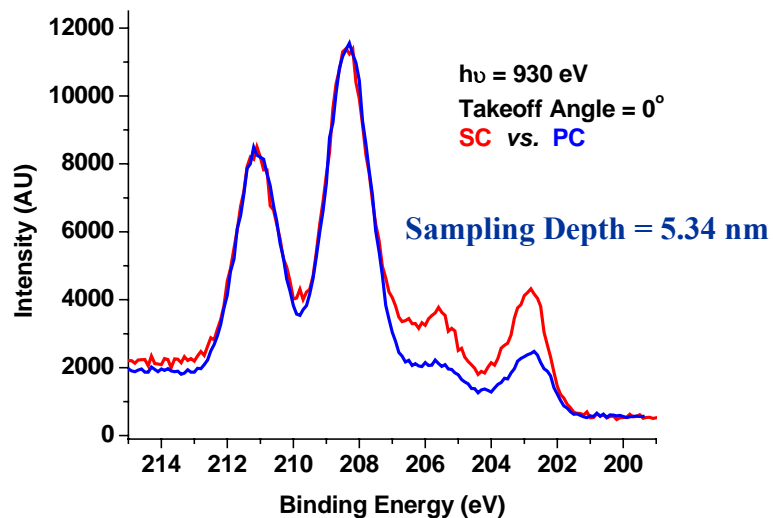
Surface Chemistry (BCP 1:1:2 ,static)

Single Crystal vs. Polycrystal



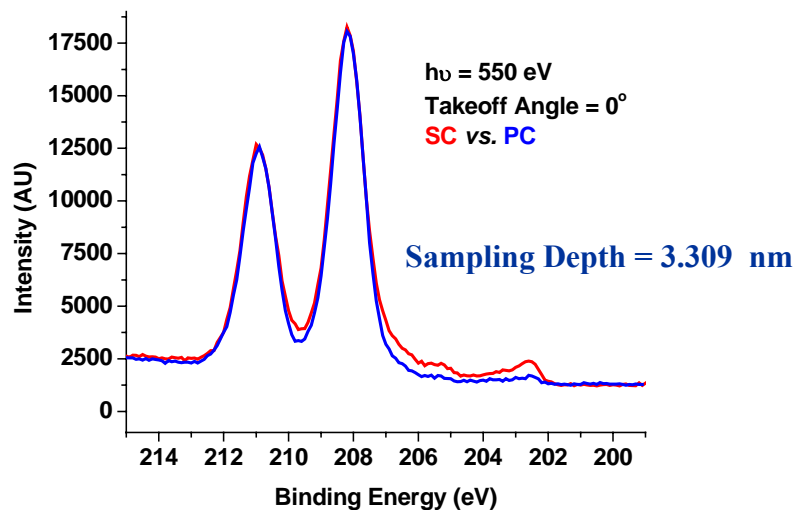
Surface Chemistry II (BCP 1:1:2 ,static)

Single crystal vs. Polycrystal



□ The intensity ratio of $\text{Nb}_{5+}/\text{Nb}_{\text{total}}$ of polycrystal is larger than that of single crystal .

□ Detailed deconvolution is in progress.



□ The single crystal surface is smoother ($R_a = 0.6 \mu\text{m}$) than the polycrystalline surfaces ($R_a = 1.66 \mu\text{m}$).



Conclusions

- ❑ **Reproducibility studies with 40 samples provide first known assessment of XPS measurement variation significance on Nb; Scattering in the $\text{Nb}_{5+}/\text{Nb}_{\text{total}}$ is about 5% of ratio; variation of intensity ratio of $\text{O}_{\text{total}}/\text{Nb}_{\text{total}}$ is smaller than variation of surface roughness.**
- ❑ **Different solution flow rate causes different surface chemistry , the surface roughness shows no significant change.**
- ❑ **Variable photon energy XPS is more surface sensitive than angle resolved XPS .**
- ❑ **Single crystal is smoother than polycrystal , Nb_2O_5 of single crystal appears thinner than that of polycrystal.**



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