Moderated discussion: Surface Measurements.

\[ \lambda_L \] for Nb:
~ 40-50 nm

*Theoretical Surface*:

- Native oxide: Nb\(_2O_5\) 5-10 nm
- Interface: sub oxides + interstitial oxygen: some monolayers.
- Interstitials: what concentration, what depth profile?
- Grain boundaries

*“Real” Surface*:

- Inclusions
- Chemical residue

SRF 2005
Limits of RF measurement

- RF measurements @ 2K => ~ 50-100 nm,
- RF measurements @ 10K => ~1µm

But

- (baking) => nm scale modifications?

=> We cannot measure very local modification of SC parameters with cavities.
Topics.

- Classic basic tools:
  - e.g.: SIMS, XPS, AUGER, X-Ray techniques... Other profiling techniques
- Extension of these techniques to high level precision:
  - e.g. use of synchrotron sources, time of flight analysis etc...
- Surface morphology:
  - @ atomic resolution STM, AFM...
  - @ larger scale: profilometry, replicas...
- Dedicated techniques for RF Superconductivity:
  - magnetic measurements, RRR, I, field emission.
- Emerging techniques:
  - 3D Atom Probe Tomography
  - \(\Delta\) measurement by photoemission
What surface technique?

Need for sensitivity and depth resolution

too superficial => STM, LEED, REED…
- need to cross ~ 5 nm Nb$_2$O$_5$, only indirect info on the SC matrix; STM/AFM = OK for morphology

too “deep” => EDX, electron probe…
- Explores ~1 µm depth
- Only relative information

roughness sensitive => X-Rays, reflectometry…
- Work on monoXstal, special sample preparation
Limits of classic techniques (SIMS, XPS)

- 1\textsuperscript{st} golden rule: what you see is not (always) what’s in your sample
  - e.g. XPS:
    - very sensitive to the detection angle $\Rightarrow$ to compare spectra the sample holder must be at same location!
    - Very sensitive to ion sputtering: creates suboxides

- 2\textsuperscript{nd} golden rule (Murphy’s law variant)
  
  (sensitivity x accuracy) goes like (time x money)$^{-1}$
  
  - e.g. SIMS
    - Standard: explores $\sim$some µm, with $\sim$100 nm depth resolution
    - Same set up, more accurate settings + UHV $\Rightarrow$ some nm resoln
    - TOF-SIMS: atomic ML resolution + indirect access to chemical info You can observe significant changes upon surface treatments BUT if you change location on the sample … changes too! (≠ grain orientations ?)

  $\Rightarrow$ Very useful for qualitative exploration, not trivial for quantitative

  (cf J. Kauffman, A. Wu/G. Rao)
Gain in sensitivity/resolution: «exotic» techniques

- 3D Atomic probe tomography (D.N Seidman)
- Grazing X-Ray depth resolved techniques (M. Delheusy)
- \( \Delta \) measurement by photoemission (myself)
How can Oi influence superconductivity?

- $[O_i]$ might affect very locally the superconducting gap $\Delta$ ($d_{\text{char}} \sim 1\text{nm}$)

<table>
<thead>
<tr>
<th>Not baked</th>
<th>Baked</th>
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<tbody>
<tr>
<td>$\Delta$ vs $x$</td>
<td>$\Delta$ vs $x$</td>
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<td>$\sim 40$ nm</td>
<td>$\sim 40$ nm</td>
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Need for a nm sensitive probe!

=> Ultrahigh resolution laser photoemission spectrometer
Angle resolved method + in situ baking => profiling should be possible with nm resolution!
As received

EPV.eps Valence structure of electron polished sample without preparation at 5K.

EPEF.eps Spectrum near EF of electron polished sample without preparation at 5K.

After Ar Sputtering

EPVsp.eps Valence structure of electron polished sample after Ar sputtering at 5K.

EPEFsp10K.eps Spectrum near EF of electron polished sample after Ar sputtering at 10K.

EPEFsp5K.eps Spectrum near EF of electron polished sample after Ar sputtering at 5K.
After Ar Sputtering + annealing

EPspan5K.eps Valence structure of electron polished sample after Ar sputtering and 2h anneal at 5K.

EPspan10K.eps Valence structure of electron polished sample after Ar sputtering and 2h anneal at 10K.

EPVspan.eps Valence structure of electron polished sample after Ar sputtering and 2h anneal at 5K.

CPV.eps Valence structure of chemical polished sample without preparation at 5K.

CPEF.eps Spectrum near EF of chemical polished sample without preparation at 5K.
Profiling techniques

- Ion sputtering: troubles with preferential O sputtering...
  - Reduces depth sensitivity to ~ >10 nm
  - Only relative information, no way to tell oxide/Oi

- Thus: be careful with profiling (XPS, Auger.... and SIMS in standard conditions)

- Profiling with depth sensitivity:
  - TOF-SIMS (but tricky to tell oxides / O_i)
  - Angle-resolved techniques

- Angle resolved photoemission (XPS/ESCA)
  - Chemical sensitivity
  - Profiling

- But ....
Photoemission (≡ ESCA ≡ XPS) / Auger

A interesting way to get info from depth under oxide

\[ E_B = h\nu - E_C \]

\( E_B \) (bonding energy) is characteristic from one element; and is influenced by electronegativity of bonded neighbors ⇒ chemical environment information.

For \([x_i] < 10\%\), ∃ deconvolution signal ≠ ∃ physical cpd !!!