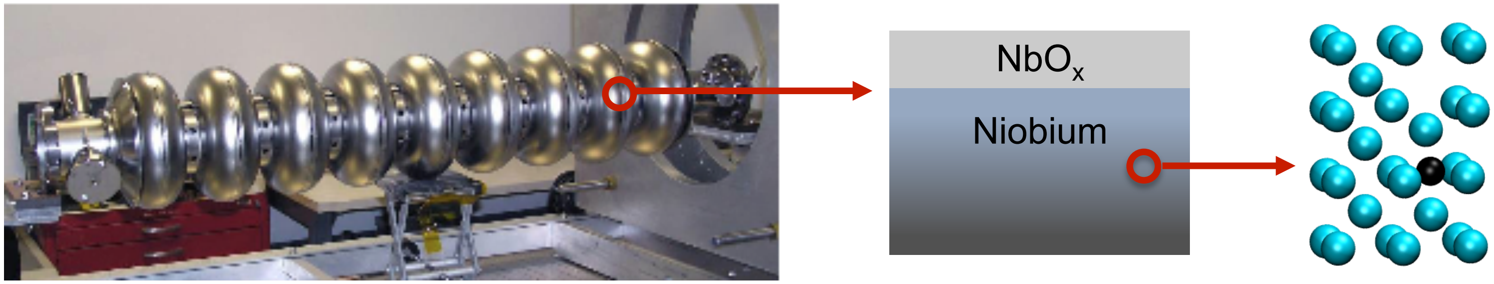
The Materials Science of Niobium Superconducting Radio-frequency Cavities

LARP/HiLumi Collaboration Meeting
Fermilab, May 11, 2015

Denise C. Ford



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Outline of Talk

- My background
- SRF cavity performance and processing
- Electropolishing studies
- Modeling impurity phases in niobium
- Niobium / precipitate interface studies
- Modeling properties related to superconductivity
- Interests in LARP

My Background

- B.S. Chemical Engineering, Certificate Technical Communications
- Part-time job with Computational Surface Science and Catalysis group, also involved some experimental work

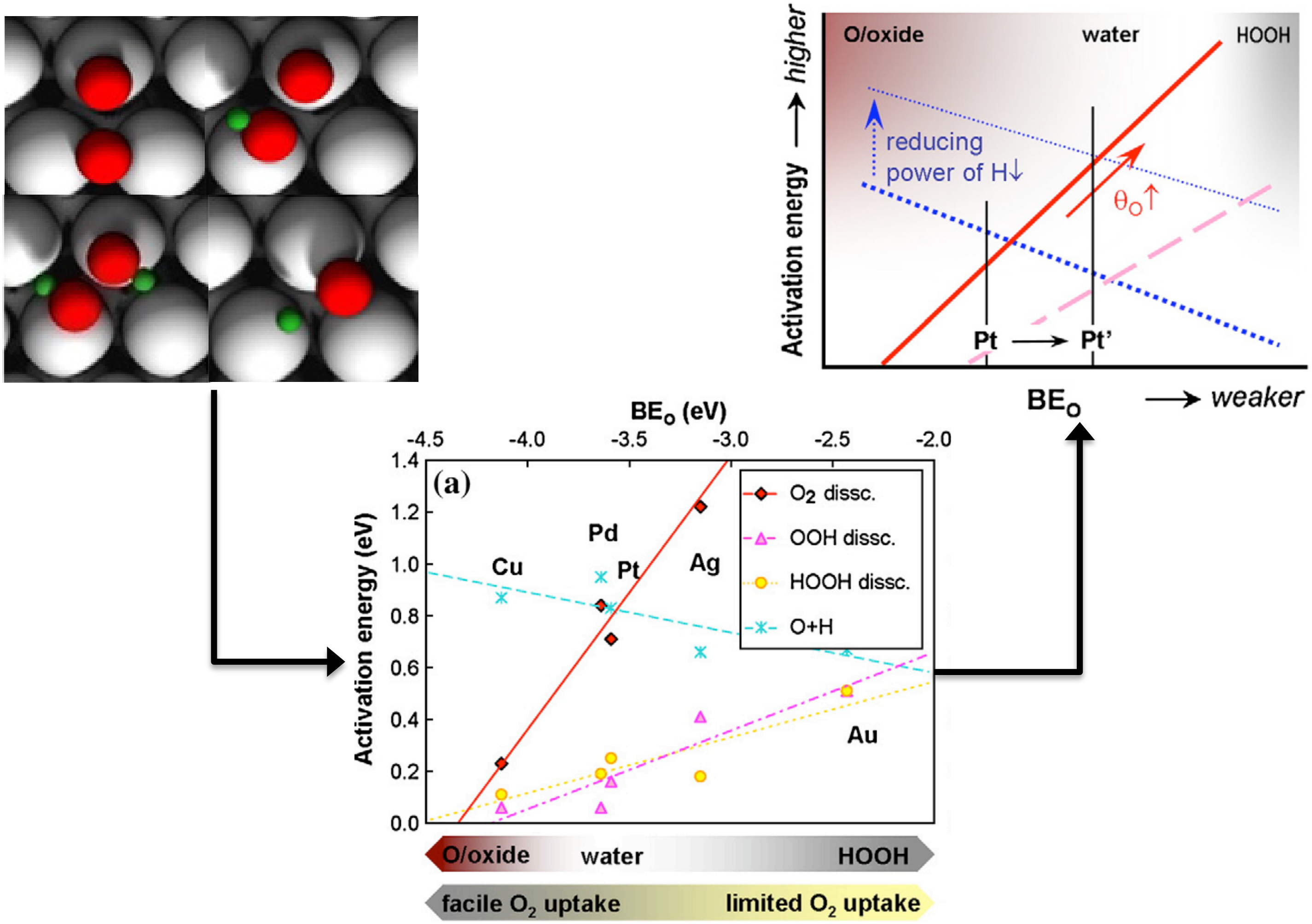
- M.S., Ph.D. Chemical Engineering
- M.S. thesis on diffusion in a porous material

- Ph.D. thesis on niobium SRF cavity processing

- Postdoc doing DFT calculations on niobium carbides and the surface corrosion reactions of glass

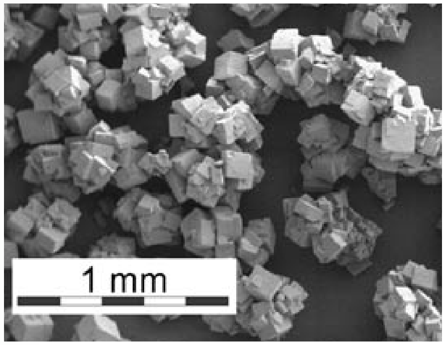
- USPAS, ILC School, and materials modeling courses

Materials Modeling Example – Surface Reactivity

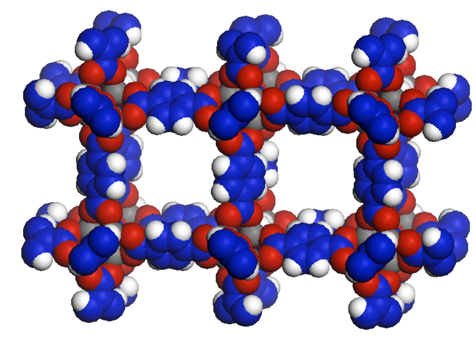


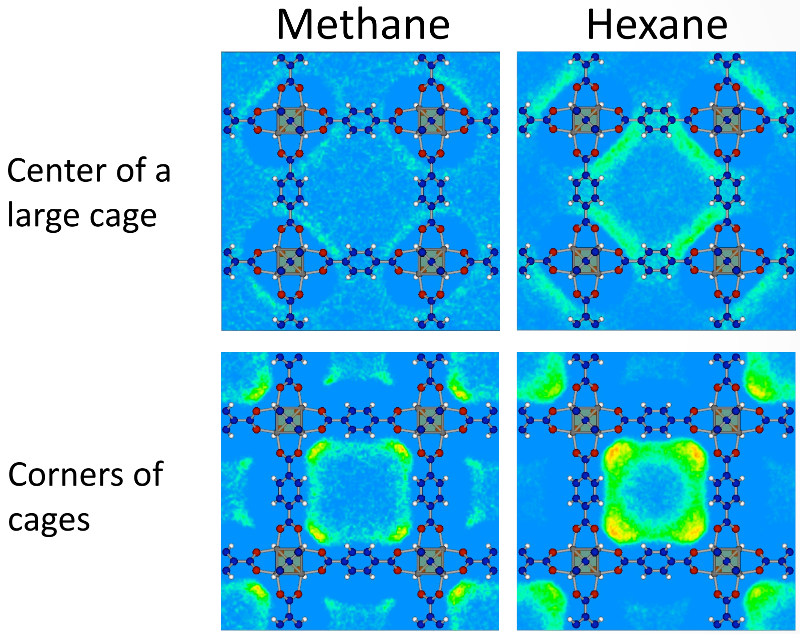
Ford D C, Nilekar A U, Xu Y, Mavrikakis M 2010 Surf. Sci. 604 1565-1575

Materials Modeling Example – Diffusion in Porous Materials



Stallmach F, et al. 2006 Angew. Chem. Int. Ed. 45 2123-2126



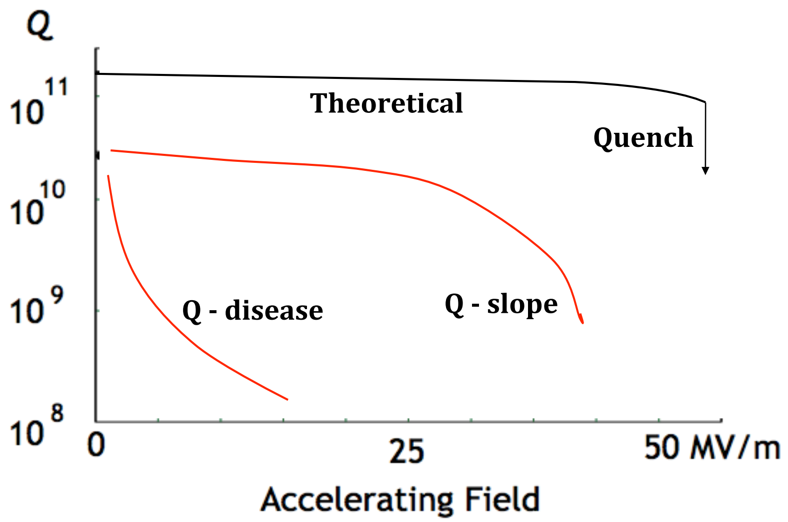


Ford D C, et al. 2012 J. Phys. Chem. Lett. 3 930-933

SRF Cavities

- Provide accelerating gradient for high-performance linear particle accelerators
- Made from ultra pure niobium (>99.98%)
  - Type II superconductor with $T_c = 9.2$ K
- Operation in the superconducting state decreases losses due to surface resistance by ~10^6

SRF Cavity Performance Characterization



$$Q = \frac{stored\ energy}{dissipated\ power / rf\ \mathrm{cycle}}$$

$$= \frac{G}{R_s}$$

- Electron field emission
- Multipacting

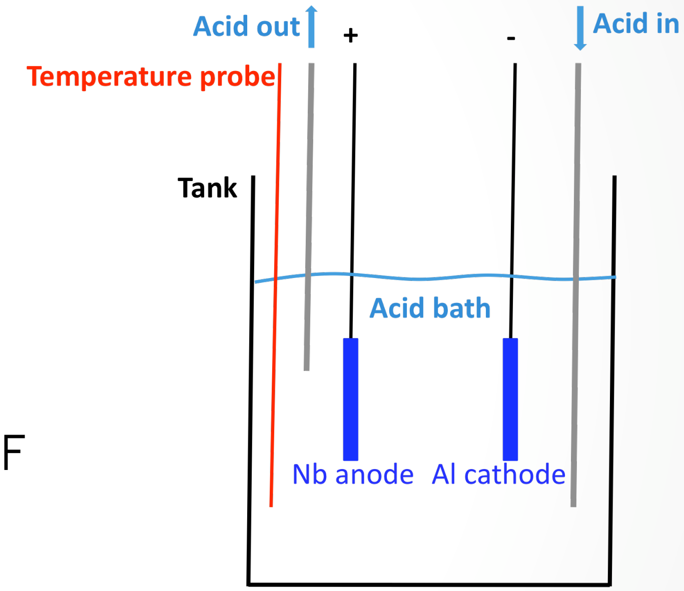
- Q-disease
- Q-slope

Cavity Forming and Processing

- Forming – source of many lattice defects
- Processing – some important techniques
  - **Buffered chemical polishing** of outer surface – increase heat transfer
  - **Bulk electropolishing** (~150 μm) of inner surface – remove damage layer from forming
  - **600-800 °C bake** – eliminate Q-disease
  - **Tumbling** – smooth surface
  - **High pressure rinse** – remove dust (prevent field emission)
  - **100-160 °C bake** – mitigate Q-slope
  - **Nitrogen and titanium impurities** – increase Q
- Procedure is empirical

The Electropolishing Process

- Ideally controlled by F^- diffusion to the niobium surface
  - Avoids crystallographic etching
  - Promotes surface leveling
  - Affected by local temperature, flow, and electrolyte composition



- Standard recipe:
  - 9 parts 98% H_2SO_4 : 1 part 48% HF

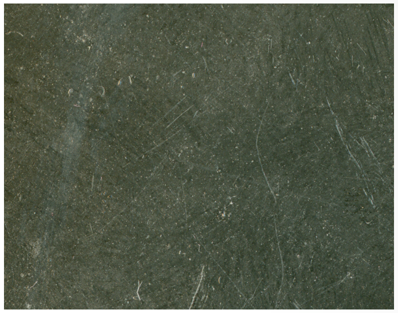
- Chemical processes:
  - Oxidation: $2Nb + 5SO_4^{2-} + 5H_2O \rightarrow Nb_2O_5 + 10H^+ + 5SO_4^{2-} + 10e^-$
  - Dissolution: $Nb_2O_5 + 6HF \rightarrow H_2NbOF_5 + NbO_2F \rightarrow 0.5H_2O + 1.5H_2O$
  - Product formation: $NbO_2F \bullet 0.5H_2O + 4HF \rightarrow H_2NbOF_5 + 1.5H_2O$

The Electropolishing Process – Nb Coupon Studies

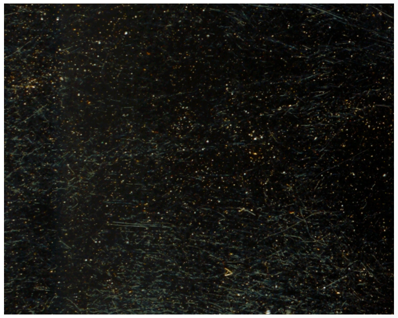
- Strong improvement of gloss
  -> reduction of roughness

- Quality of finish related to Nb pretreatment
  - Cold work strongly promotes pitting
  - Welding promotes pitting

- Quality of finish related to EP process parameters
  - Agitation of bath promotes etching
  - Temperature indirectly effects the process



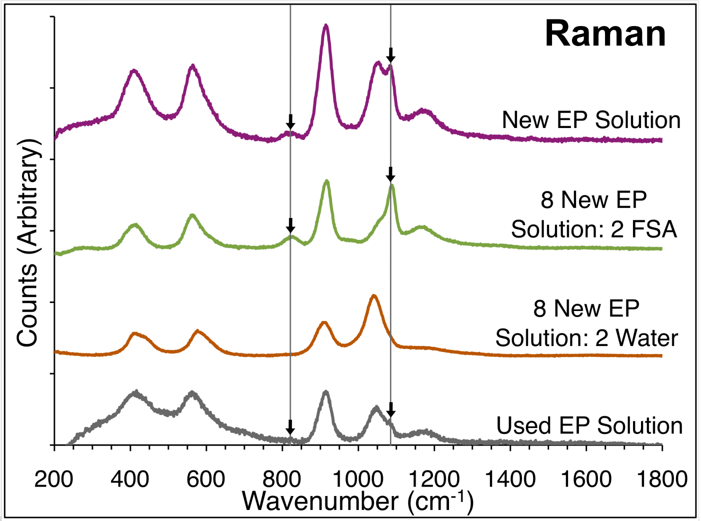
Nb sample before EP

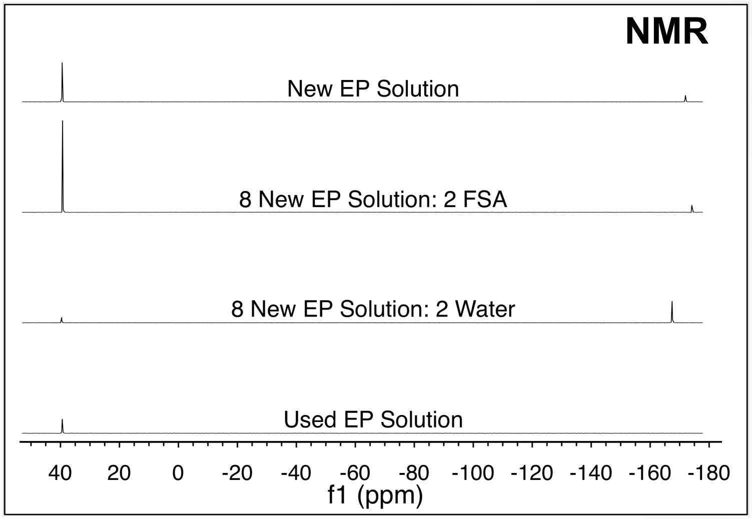


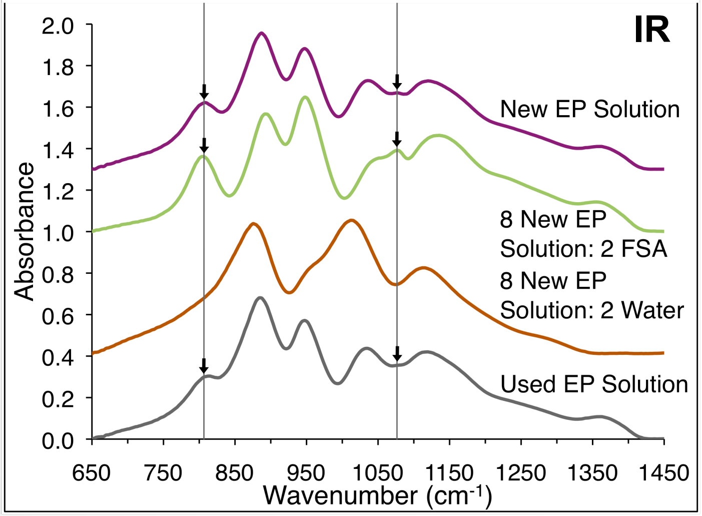
Nb sample after EP

Cooley L D, et al. 2011 IEEE Trans. Appl. Supercond. 21 2609-2014

The Electropolishing Process – Spectroscopy Studies







- $HFSO_3$ is present in fresh mixed EP solution via:

  $HF + H_2SO_4 <-> HFSO_3 + H_2O$

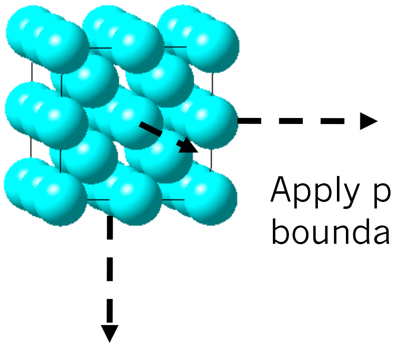
  -> the concentration of $HFSO_3$ can be used to track the progress of EP

Ford D C, et al. 2013 J. Electrochem. Soc. 160 H398-H403

Modeling of Impurity and Defect Structures in Nb

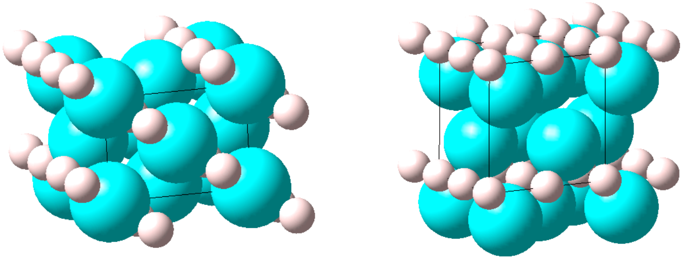
bcc Niobium

Build a crystal structure



Apply periodic boundary conditions

NbH (β niobium hydride)

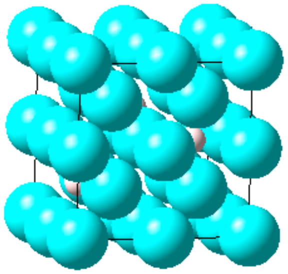


Compare properties of different structures

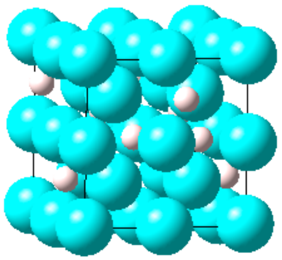
- Solve the electronic structure problem for the model systems using density functional theory in VASP
- Assess properties such as binding energy, electron distribution, and niobium lattice strain

Niobium Hydride Phases

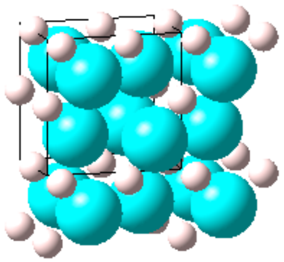
α



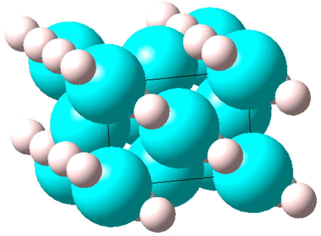
α’

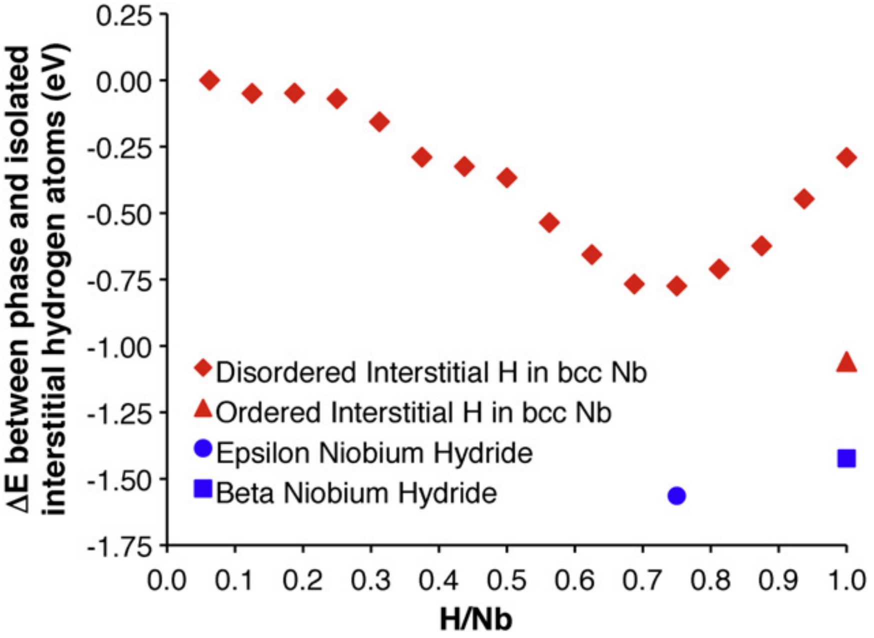


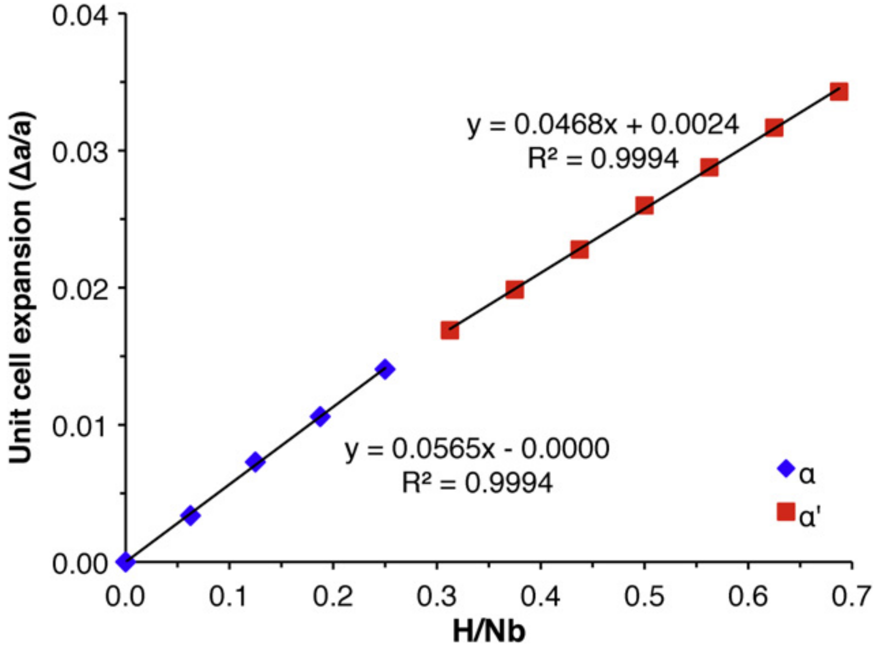
ε



β

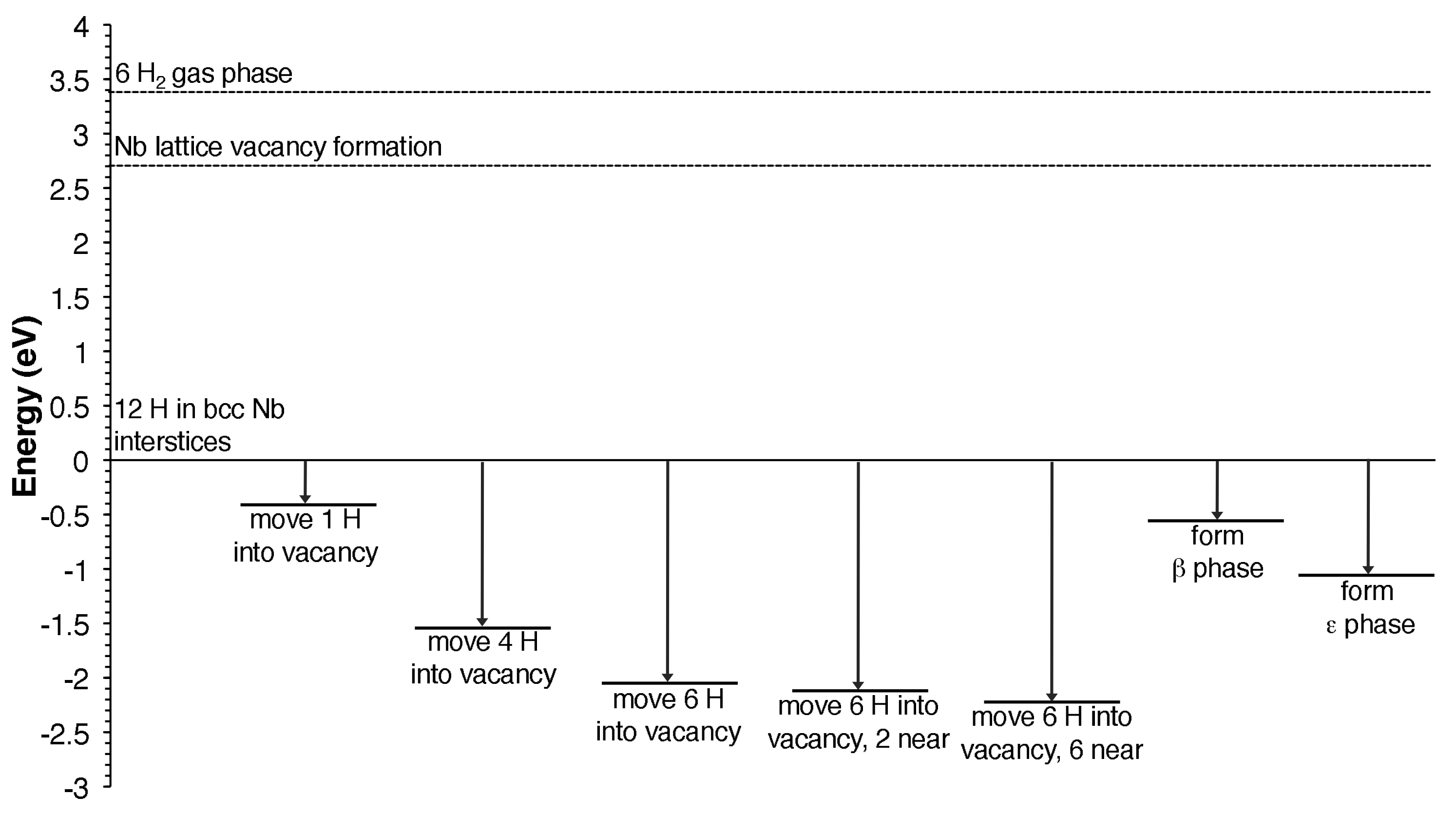






Ford D C, Cooley L D and Seidman D N 2013 Supercond. Sci. Technol. 26 095002

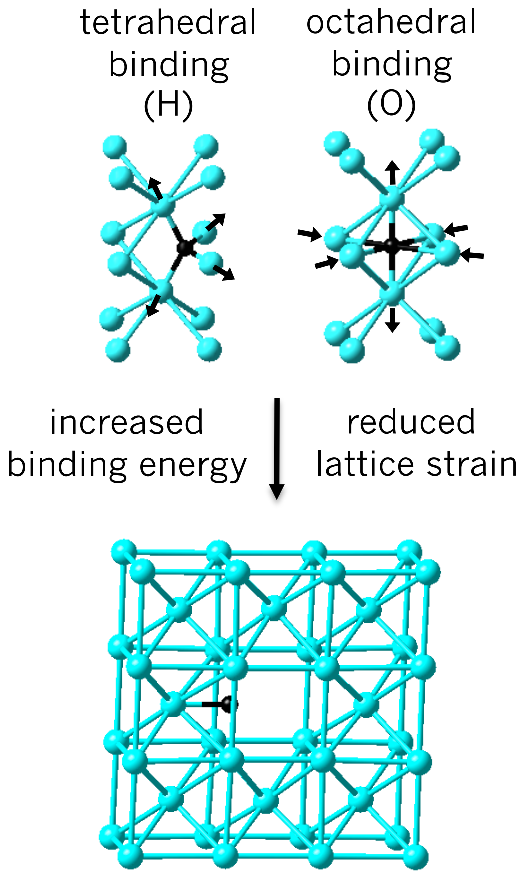
Niobium Hydride Phases



- Niobium lattice vacancies can nucleate ordered hydride phases

Ford D C, Cooley L D and Seidman D N 2013 Supercond. Sci. Technol. 26 095002

Hydrogen and Oxygen in Niobium

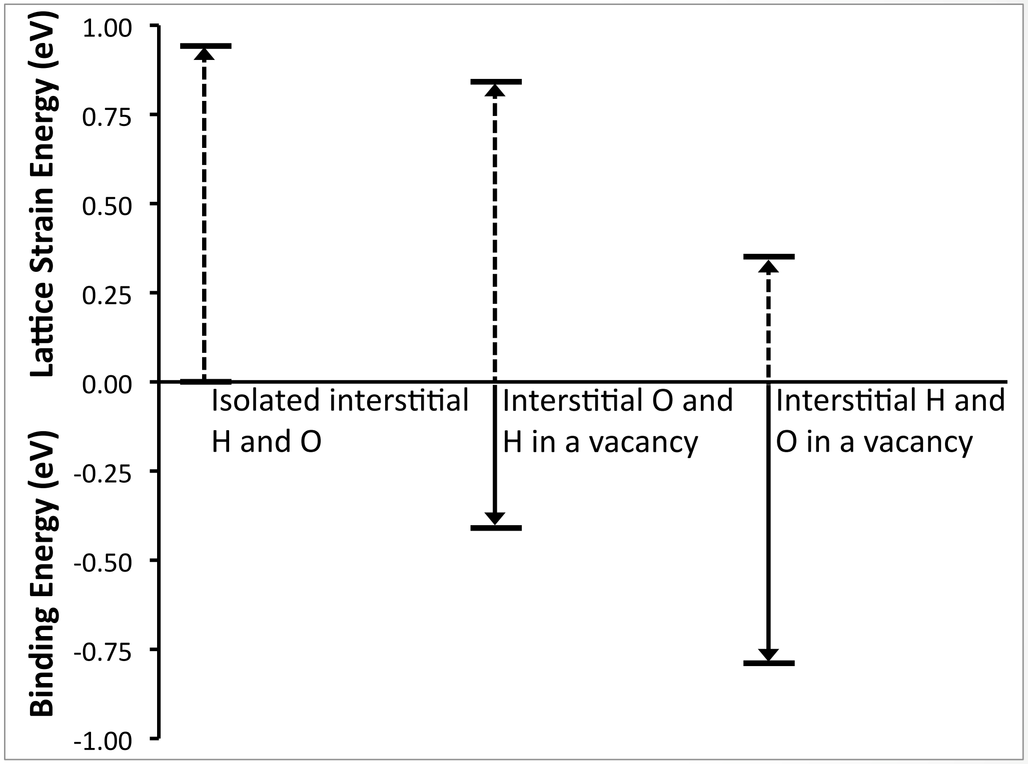


tetrahedral binding (H)

octahedral binding (O)

increased binding energy

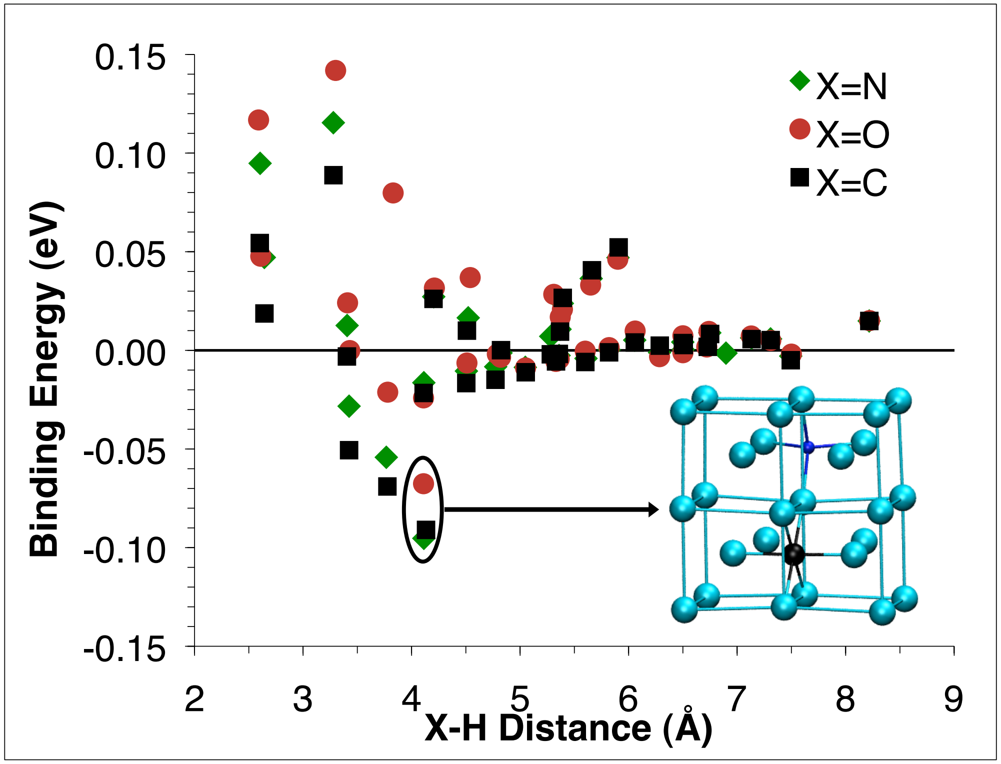
reduced lattice strain



- Oxygen atoms can block hydride nucleation sites

Ford D C, Cooley L D and Seidman D N 2013 Supercond. Sci. Technol. 26 105003

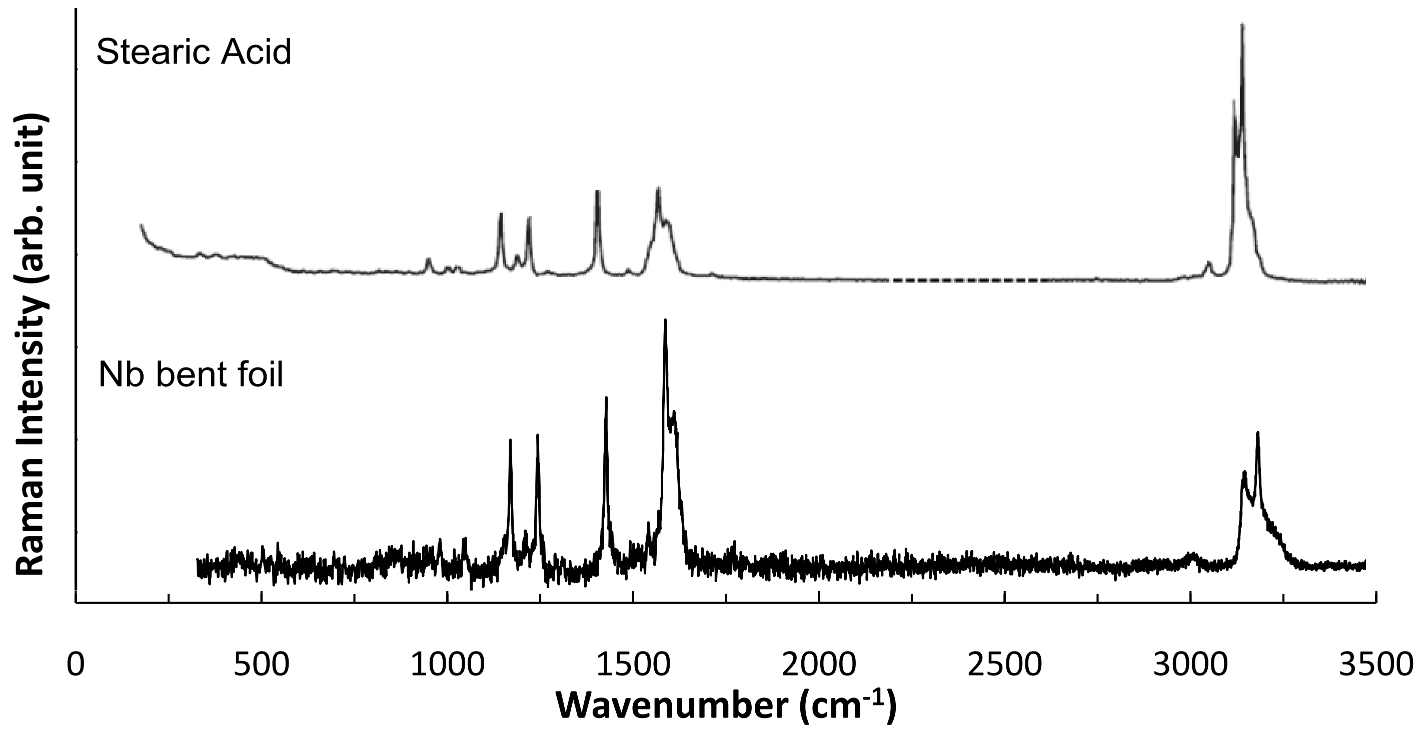
(O, N, C) and H in Niobium



- Interstitial O, N, and C atoms can trap interstitial H atoms and prevent detrimental hydride formation

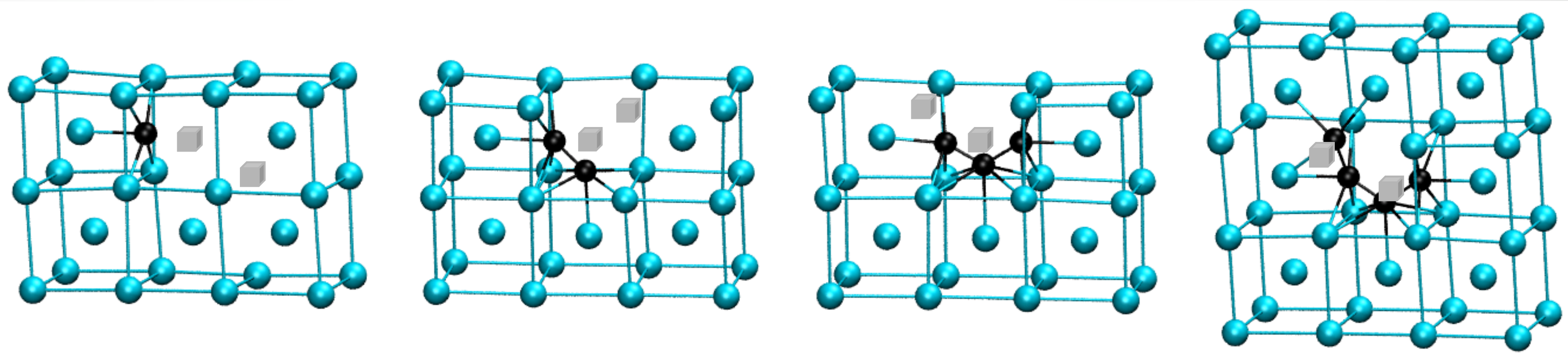
Ford D C, Zapol P, Cooley L D 2015 J. Phys. Chem. C in press

Raman Spectrum of Hydrocarbon Chains in Niobium



C. Cao, et al. 2013 Phys. Rev. ST Accel. Beams 16 064701

Carbon Chains in Niobium

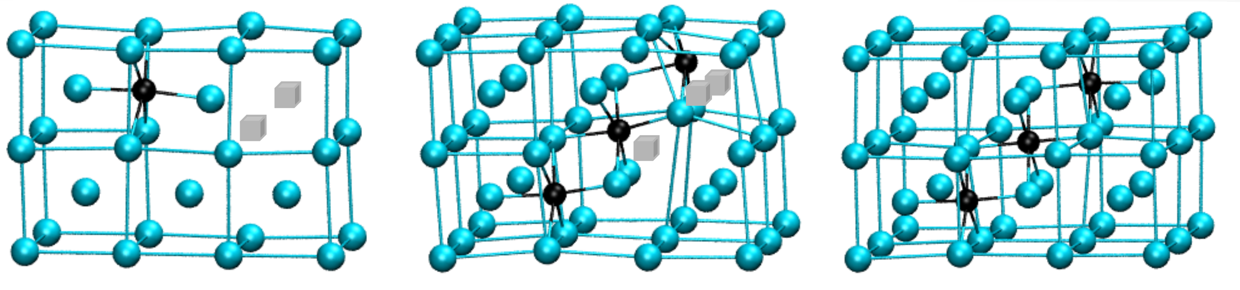


BE_C (eV)	-0.35	-0.88	-0.51	0.07
BE_{NbC} (eV)	-0.03	-0.24	0.45	1.35

- Longer chains quickly become unfavorable
- C-H in Nb spontaneously dissociates
- Is a surface or surface-like defect, such as a grain boundary, required for chain formation?

Ford D C, Zapol P, Cooley L D 2015 J. Phys. Chem. C in press

Carbon Clustering in Niobium

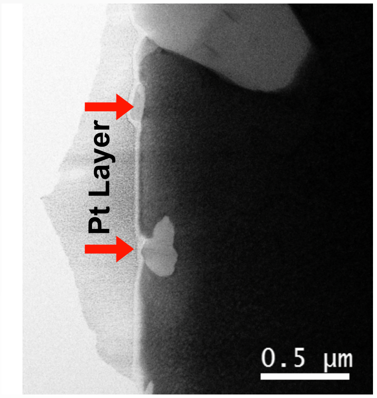


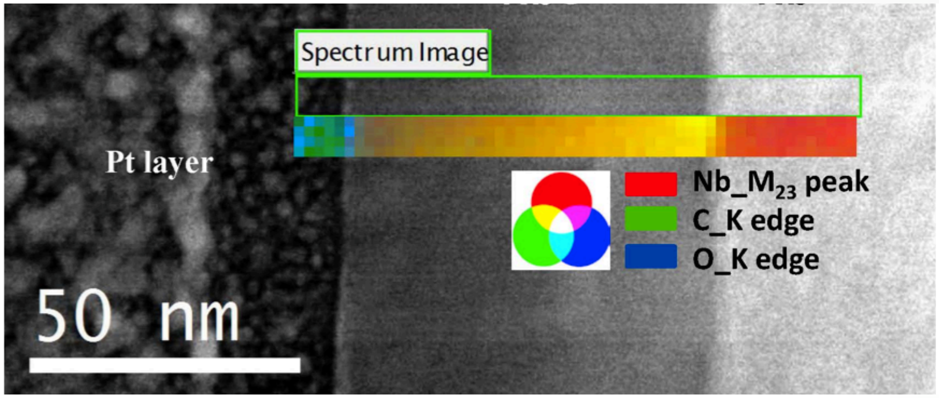
BE_C (eV)	-0.77	-1.84	-0.32
BE_{NbC} (eV)	-0.45	-0.88	0.64

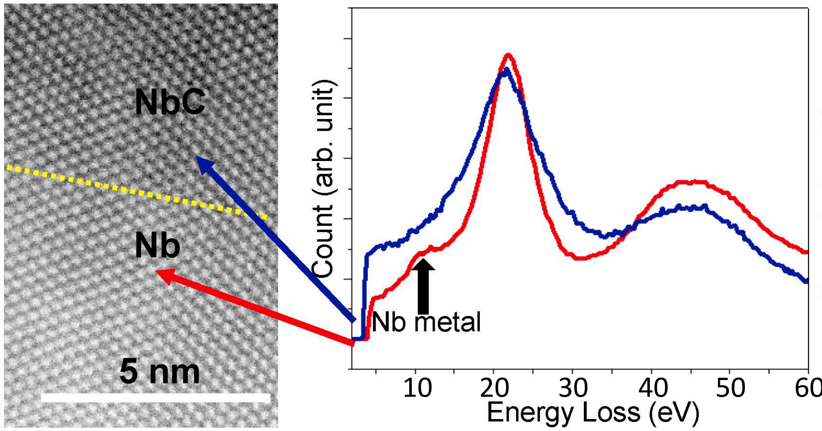
- Favorable for C to cluster
- C can form Cottrell atmospheres around niobium lattice vacancy-type defects

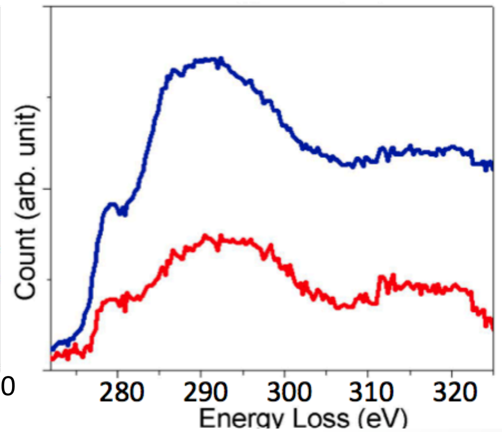
Ford D C, Zapol P, Cooley L D 2015 J. Phys. Chem. C in press

Experimental Evidence for NbC Precipitates in SRF Nb





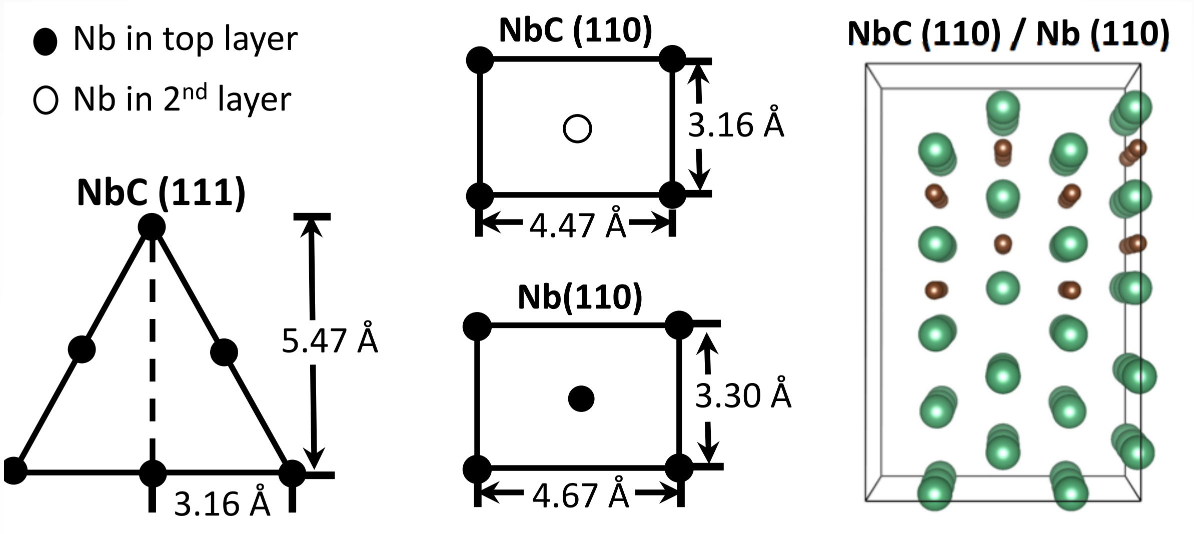




- Coherent Nb / NbC interfaces are found in a variety of SRF Nb samples

Cao C, et al. 2015 Phys. Rev. B 91 094302

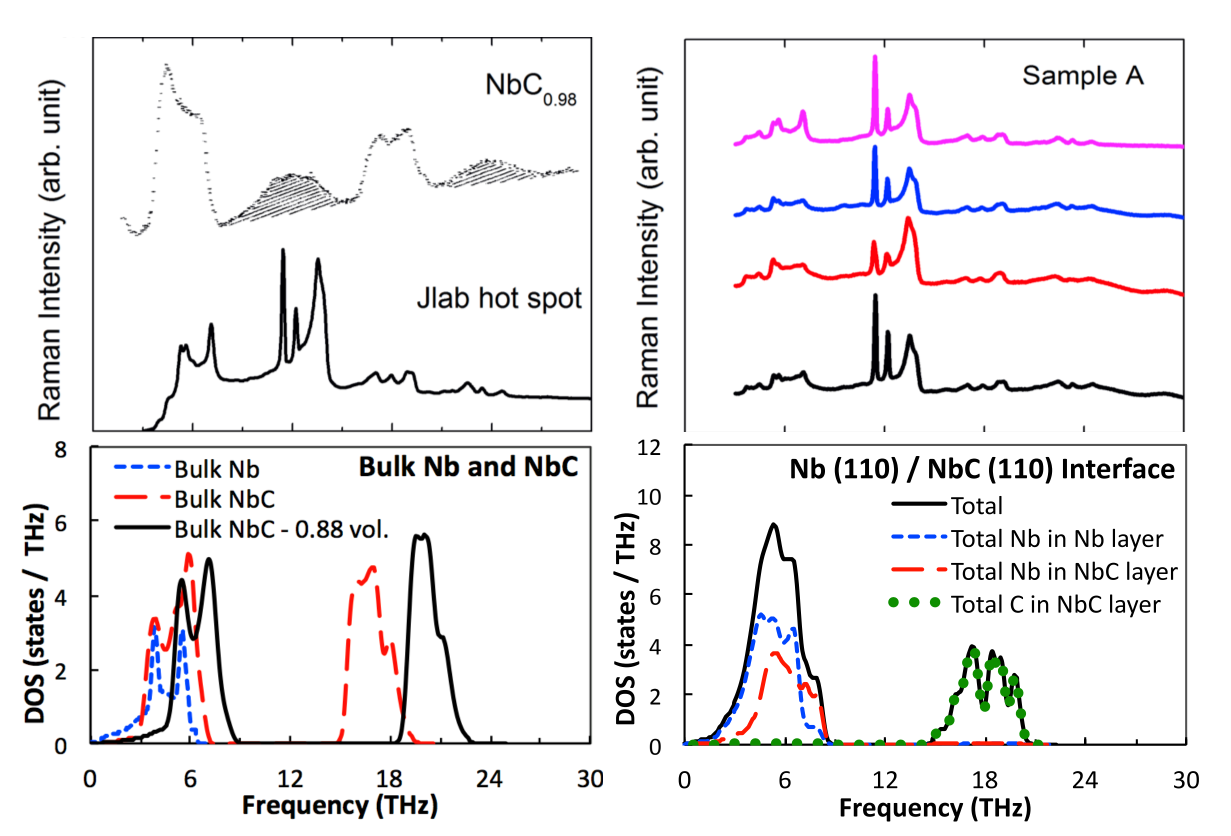
Creating an Coherent Interface Model for Nb/NbC



- Anisotropic compression is required to match the NbC (111) plane to the Nb (110)
- Relatively smaller isotropic expansion is required to match NbC (110) to Nb (110)

Cao C, et al. 2015 Phys. Rev. B 91 094302

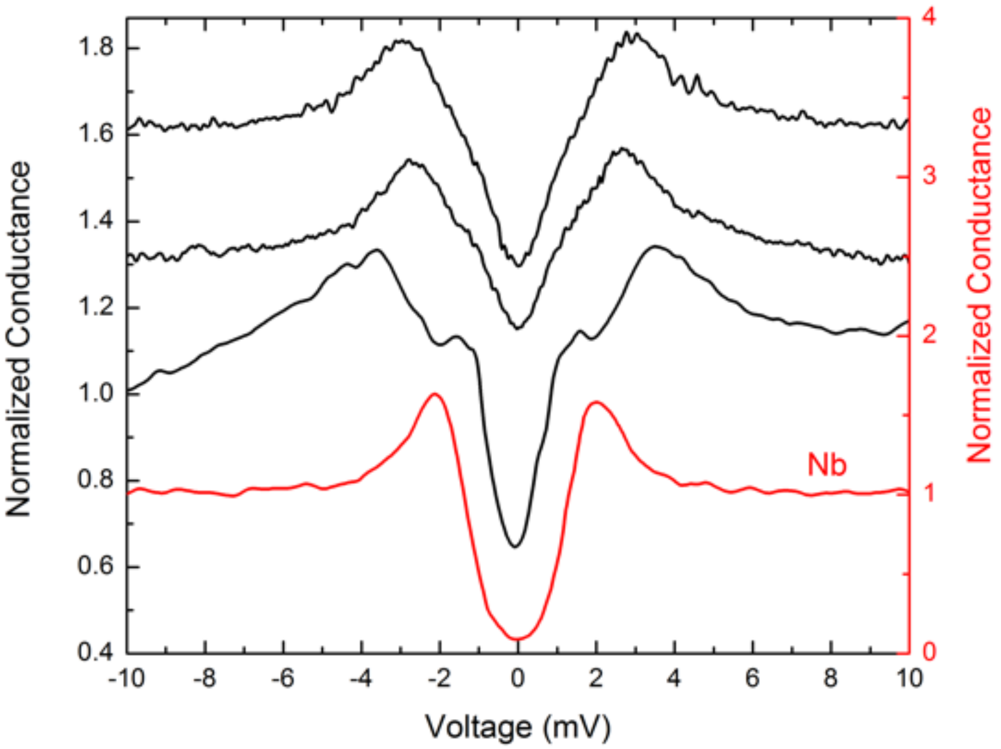
Raman Spectra and Calculated Phonon DOS



- The two-phonon signal dominates the experimental spectra
- The Nb (110) / NbC (110) interface model provides a close match to the experimental spectra

Cao C, et al. 2015 Phys. Rev. B 91 094302

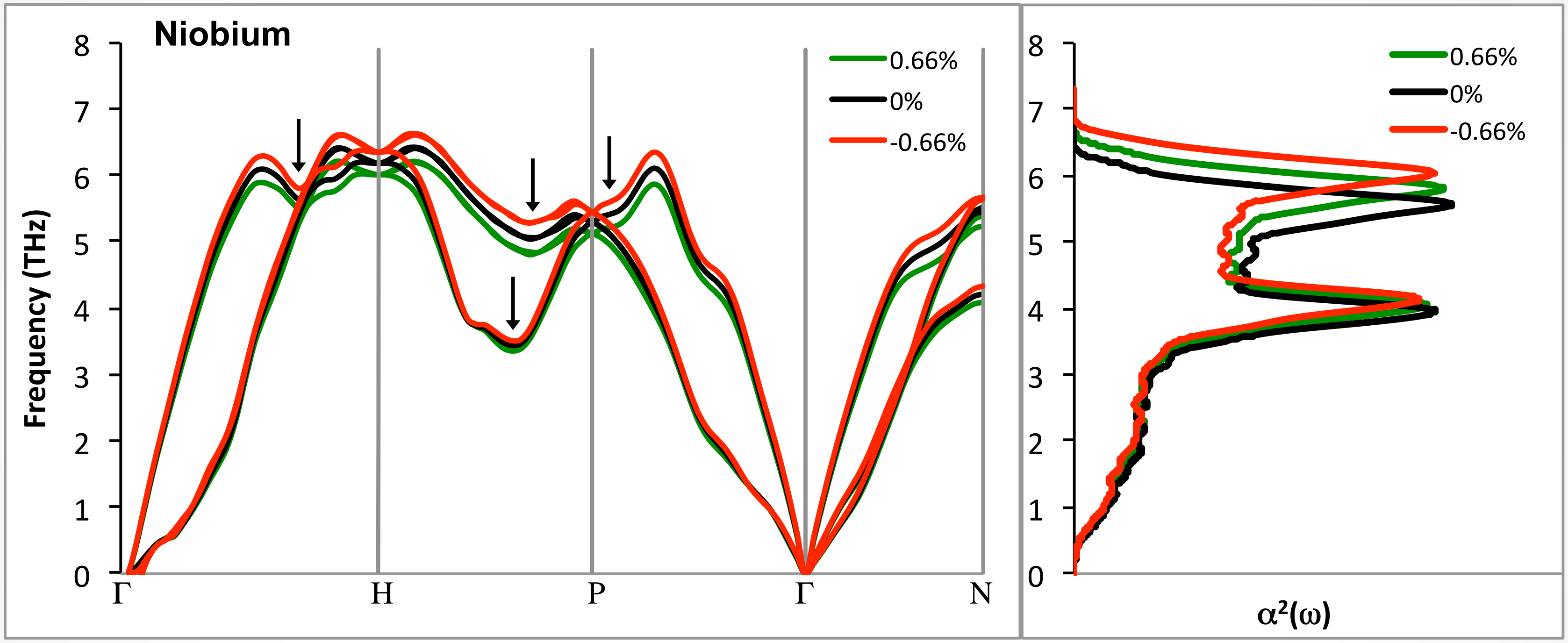
Point Contact Tunneling Spectra



- Increased gap -> possible increased T_c at or near the interface between Nb and NbC or in the NbC precipitates

Cao C, et al. 2015 Phys. Rev. B 91 094302

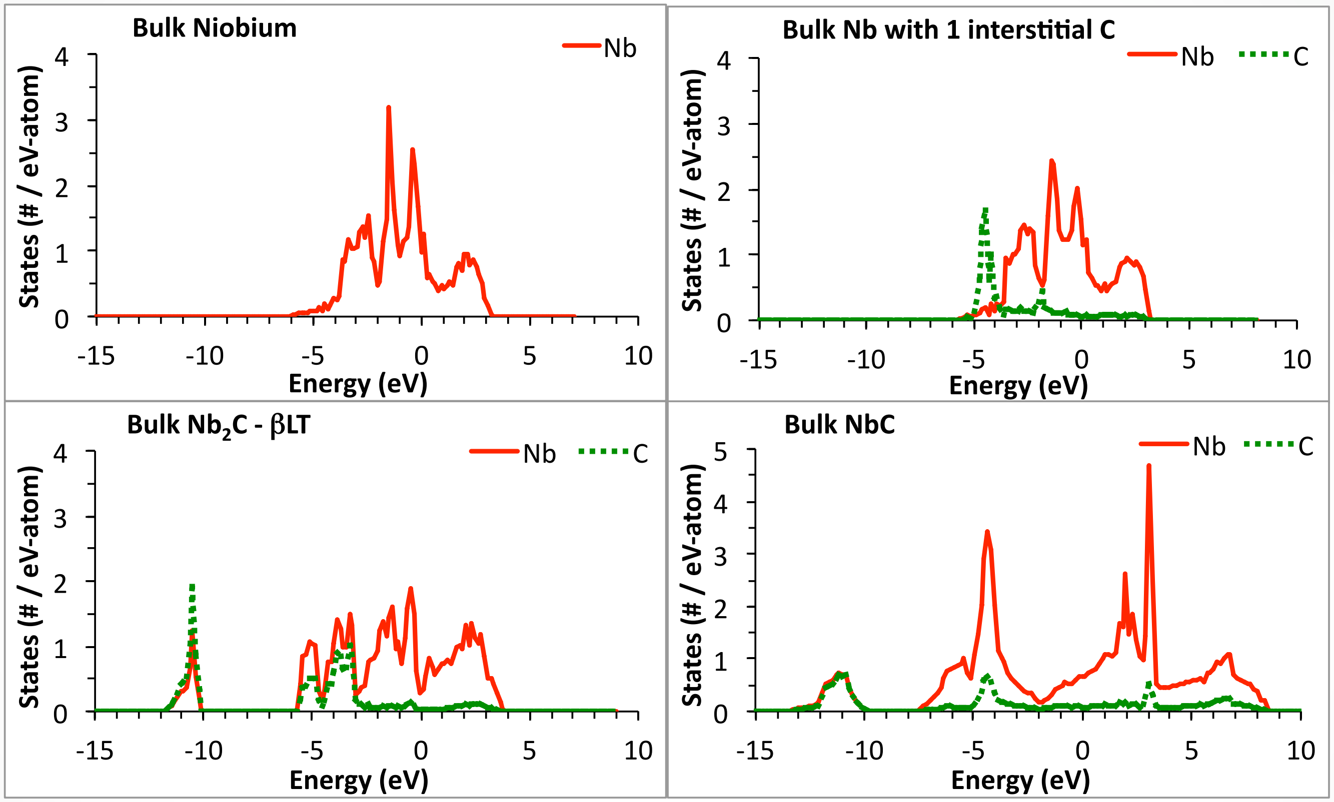
Niobium Lattice Strain and Superconductivity



- Changes in electron and phonon structure lead to increased T_c for small expansive strains and decreased T_c for small pressures

Ford D C, Zapol P, Cooley L D 2014 Appl. Supercond. Conf.

Effect of Carbon on Nb T_c



- Carbon atoms bind with Nb atoms, reducing N_F
- Nb_2C has a lower N_F than NbC and Nb -> likely lower T_c

Ford D C, Zapol P, Cooley L D 2015 J. Phys. Chem. C in press

Interests in LARP

- Materials science studies of the components for the HiLumi upgrade
  - production issues with the crab cavities
  - production issues with the quadrupoles
  - radiation stability
- Advanced accelerator materials

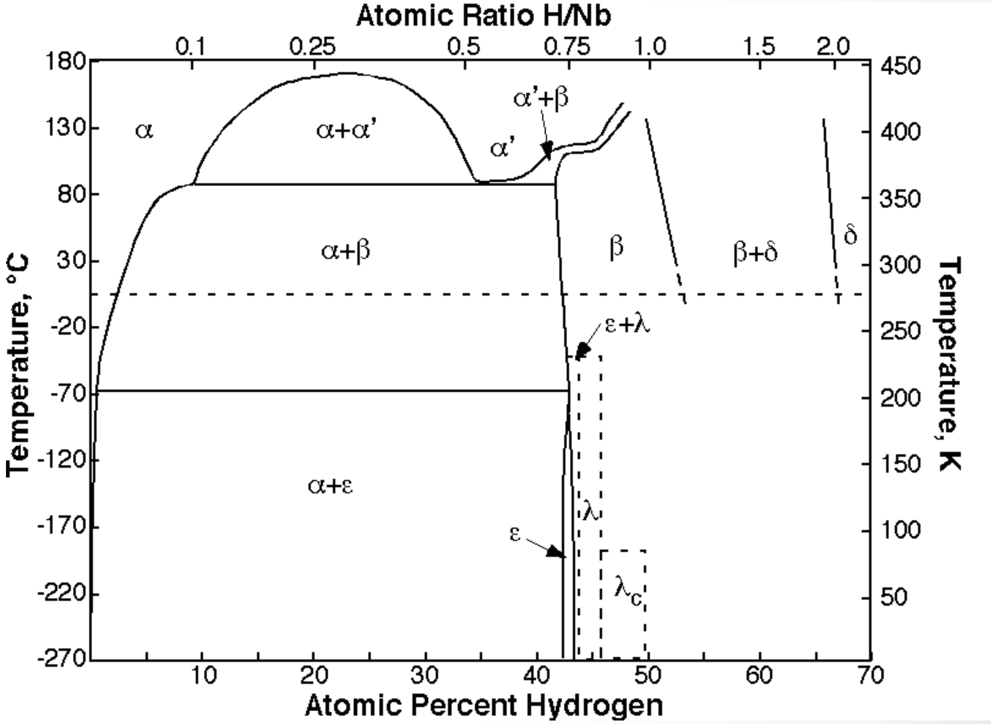
Modeling of Impurity and Defect Structures in Nb

- Calculation parameters
  - Vienna Ab Initio Simulation Package (VASP)
  - Plane wave basis set w/400 eV cutoff
  - PAW pseudopotentials to describe atomic cores
  - PBE-GGA exchange-correlation functional
  - ~0.25/Å gamma-centered k-point mesh for geometries
  - ~0.12/Å gamma-centered k-point mesh for eDOS

- Bader Method to assign local properties

Hydride Phases in Niobium

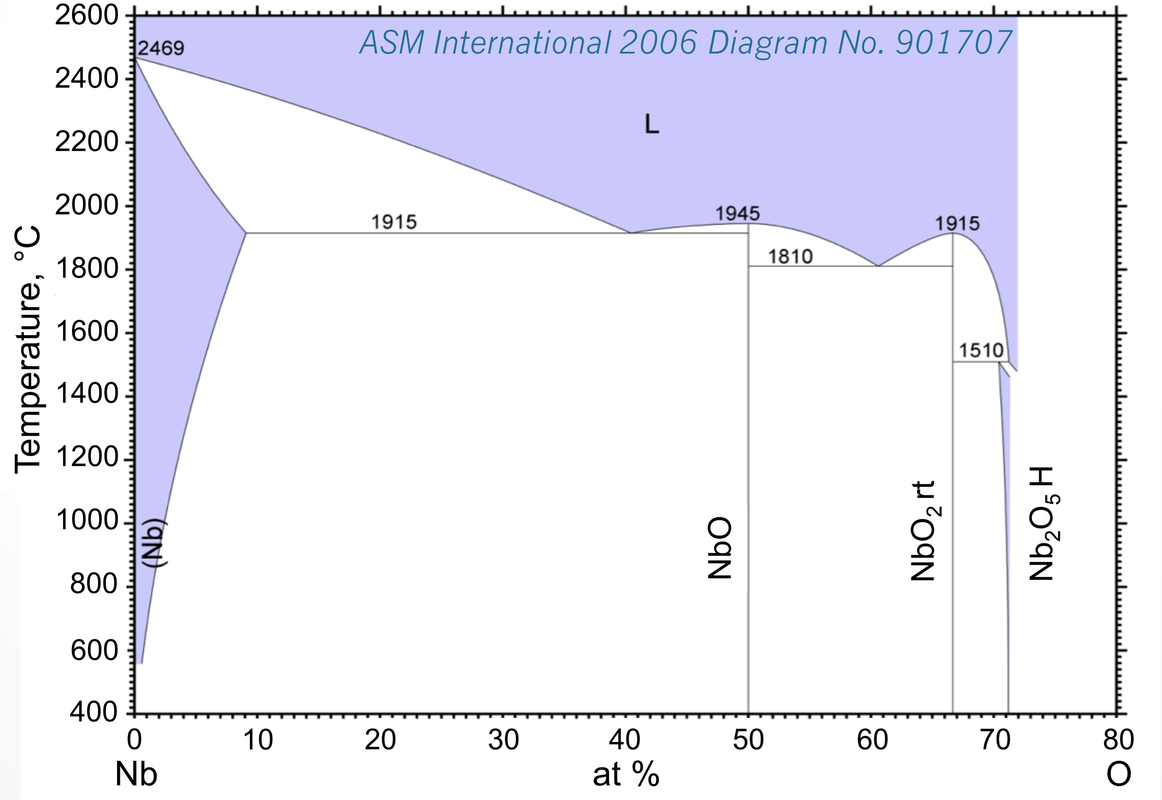
- The niobium – hydrogen phase diagram is very complex
  - **α, α'** – interstitial hydrogen dispersed in bcc niobium
  - **β, ε** – ordered hydrogen interstitials in fco niobium
  - **δ** – hydrogen in the tet. sites of fcc niobium – fluorite structure
  - **λ, λ_c** – experimentally unconfirmed phases



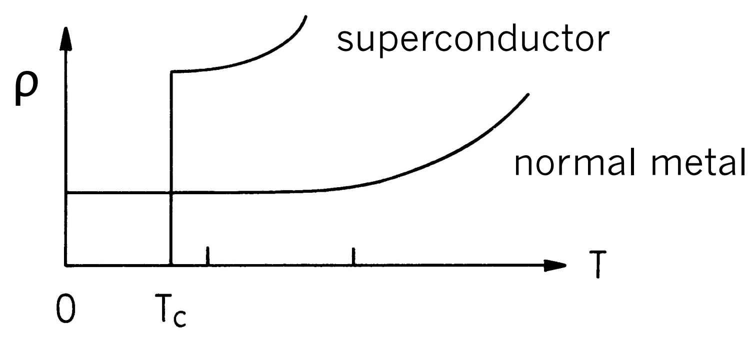
Ricker R E, Myneni G R 2010 J. Res. Natl. Inst. Stand. Technol., 115, 1

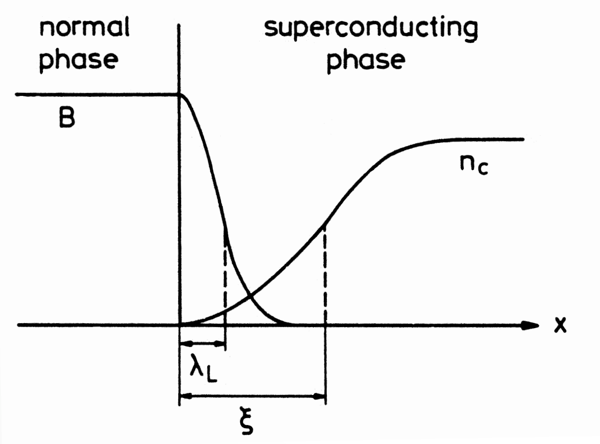
Oxide Phases in Niobium

- The niobium surface is covered with a complex system of oxide layers which changes during processing
- Properties range from metallic to insulating
- Oxygen solubility is much lower than hydrogen

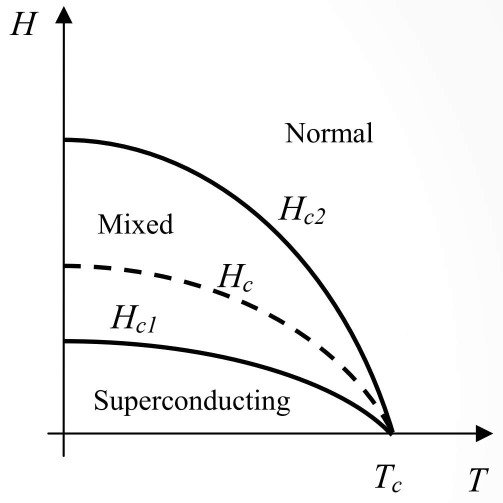


Niobium Superconductivity





$B = \mu_0 H$



Niobium

T_c = 9.2 K

B_{c1} = 174-190 mT

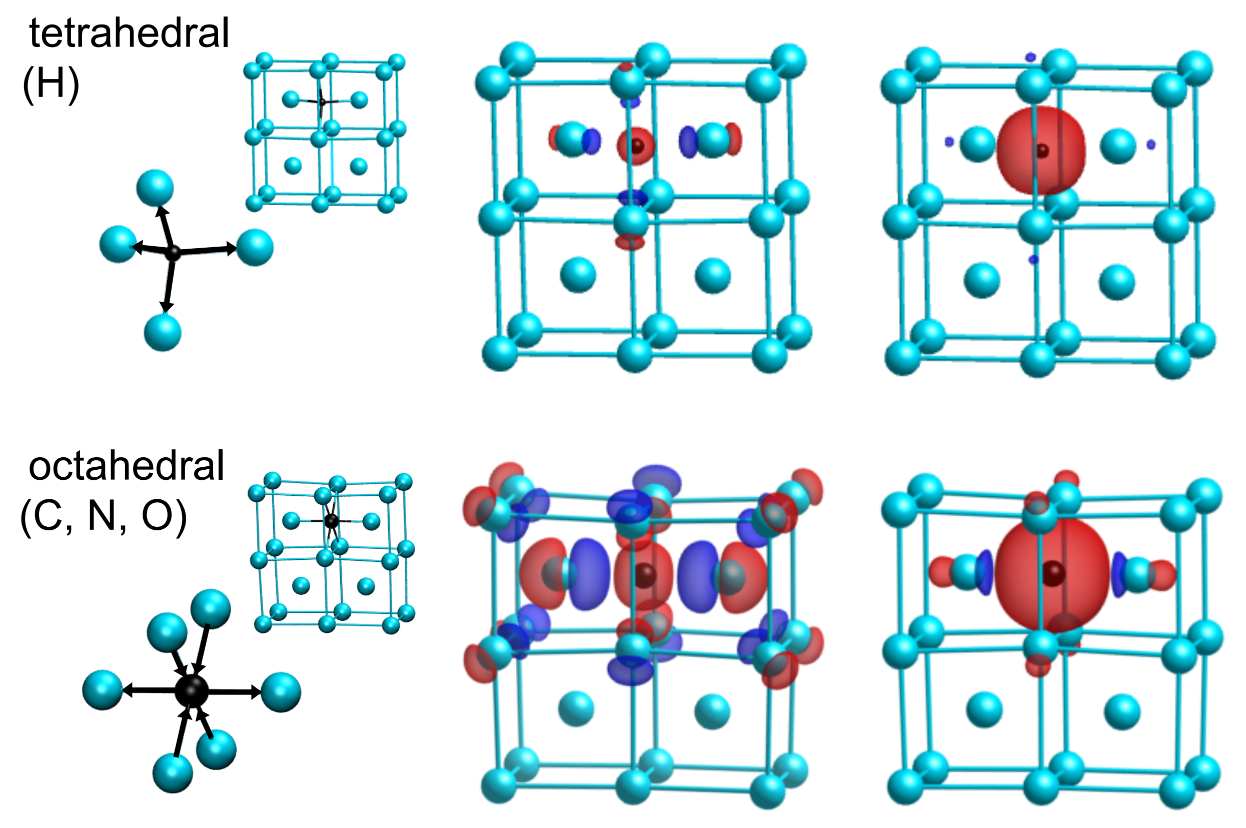
B_c = 200 mT

B_{c2} = 390-450 mT

λ = 32-44 nm

ξ_0 = 30-60 nm

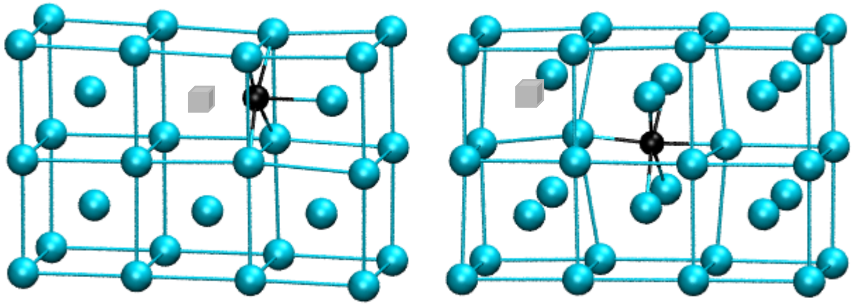
Properties of the Interstitial Impurities



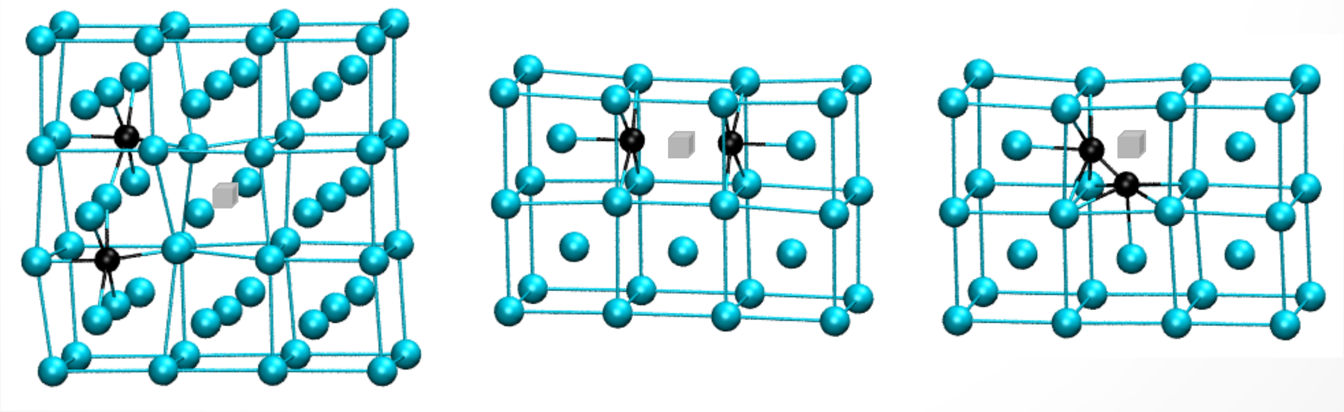
Ford D C, Zapol P, Cooley L D 2015 J. Phys. Chem. C in press

Impurity Binding Near Niobium Lattice Vacancies

One Impurity Near Vacancy



Two Impurities Near Vacancy



Ford D C, Zapol P, Cooley L D 2015 J. Phys. Chem. C in press