Mechanical Properties of High Purity Niobium Novel Measurements

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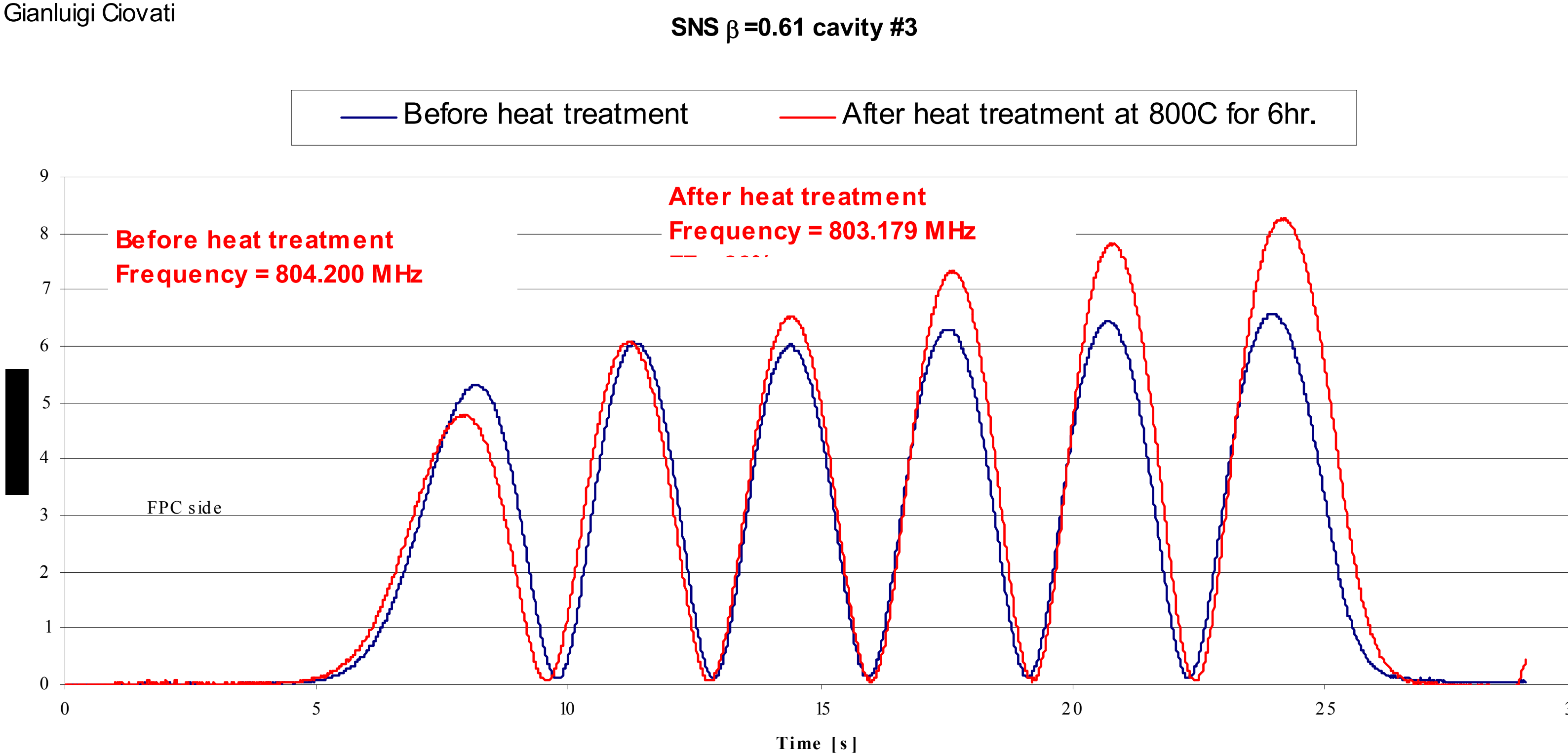
Overview

- Why is an interest in mechanical properties of RRR niobium again?
- What are the Novel Measurements!!
- Where are we going from here?

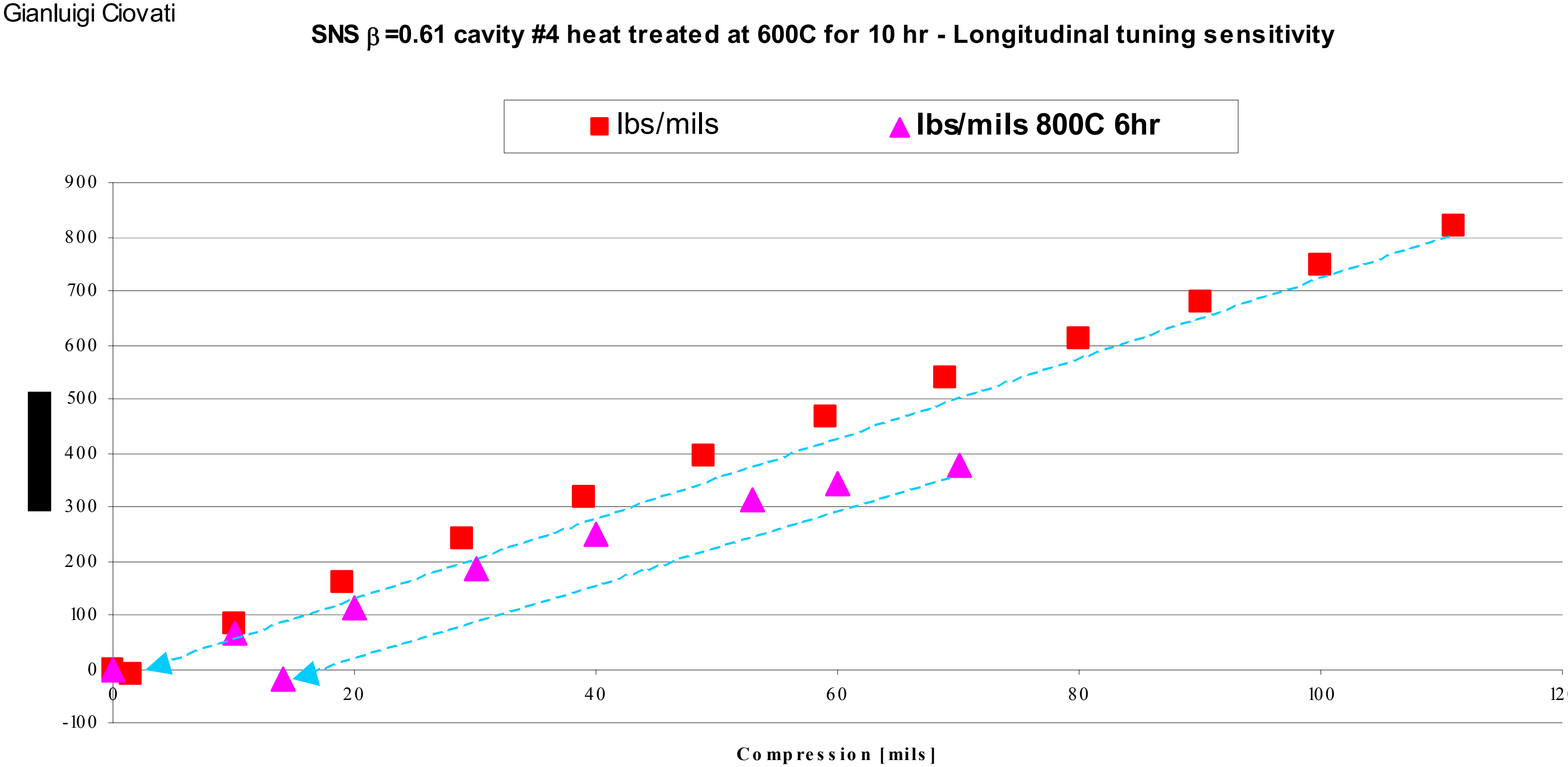
SNS Cavity Field Profile

Gianluigi Ciovati



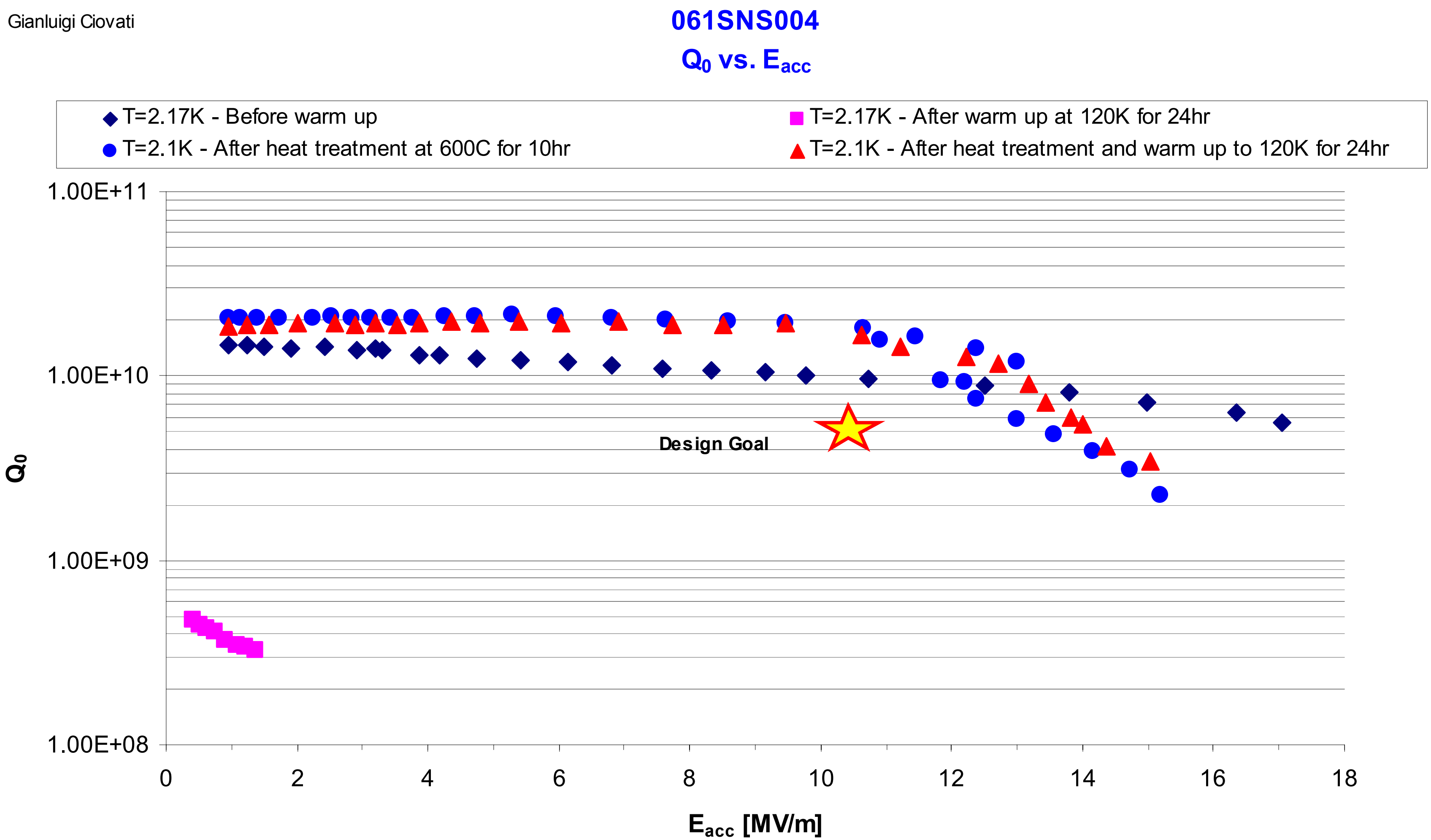
Cavity Tuning Sensitivity

Gianluigi Ciovati

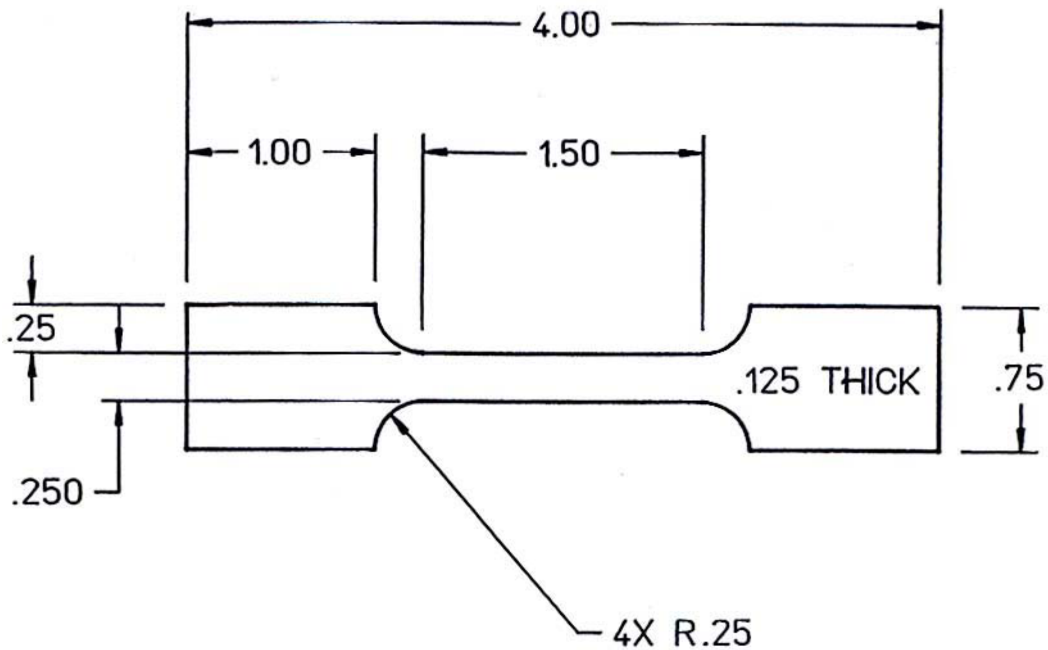


Qo Vs E_{acc}

Gianluigi Ciovati



Tensile sample



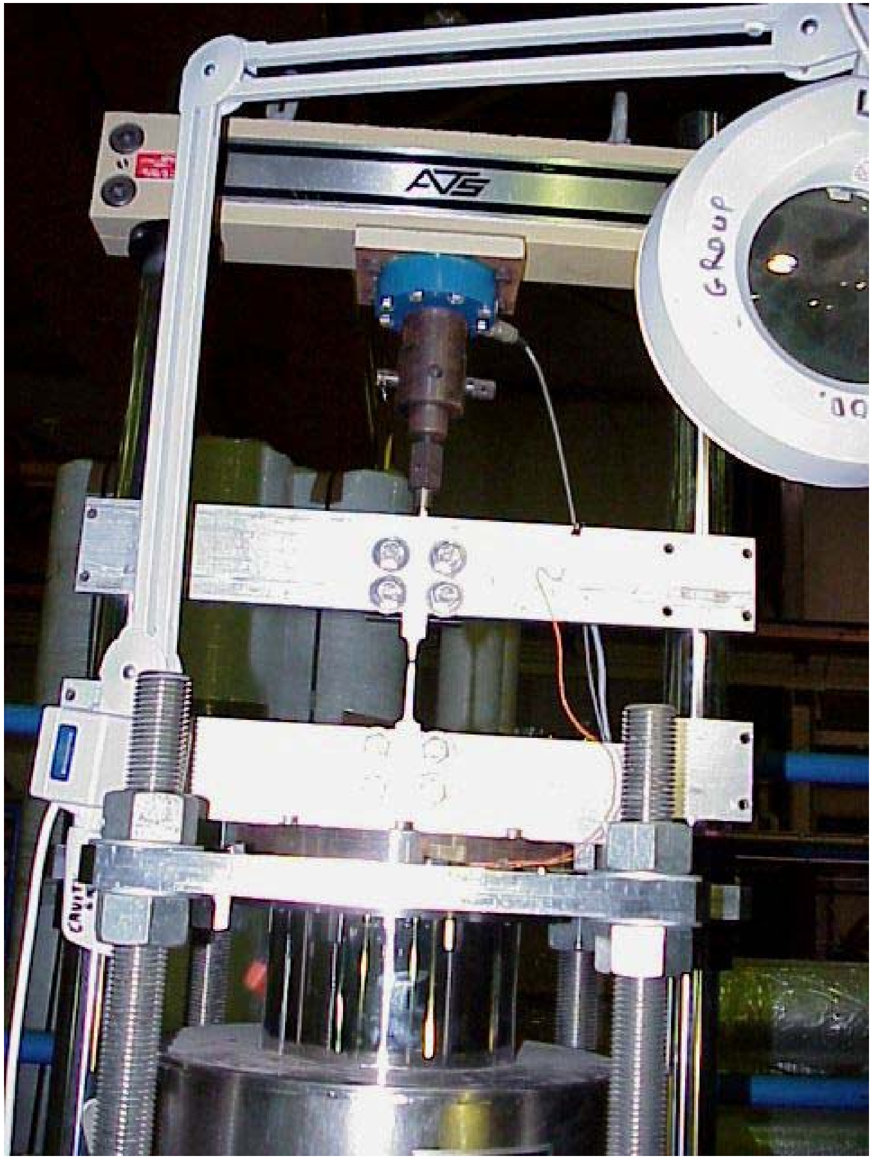
Does not meet ASTM standards but not required since used at very low strain rates

Tensile apparatus

Unique System

Wide temperature range down to 4.2 K

Strain rate variation – six orders



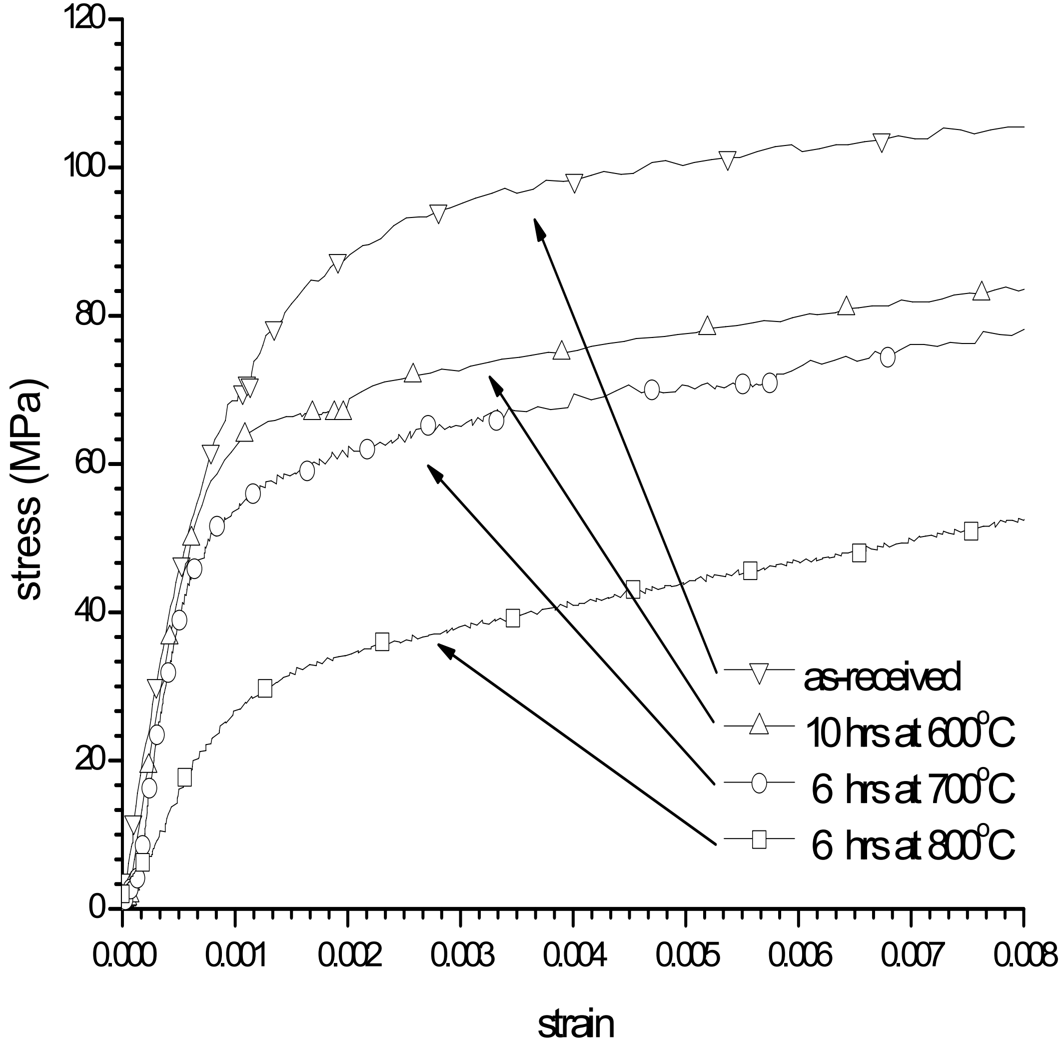
Measurement Accuracies

- Strain Measurement Accuracy ± 2%

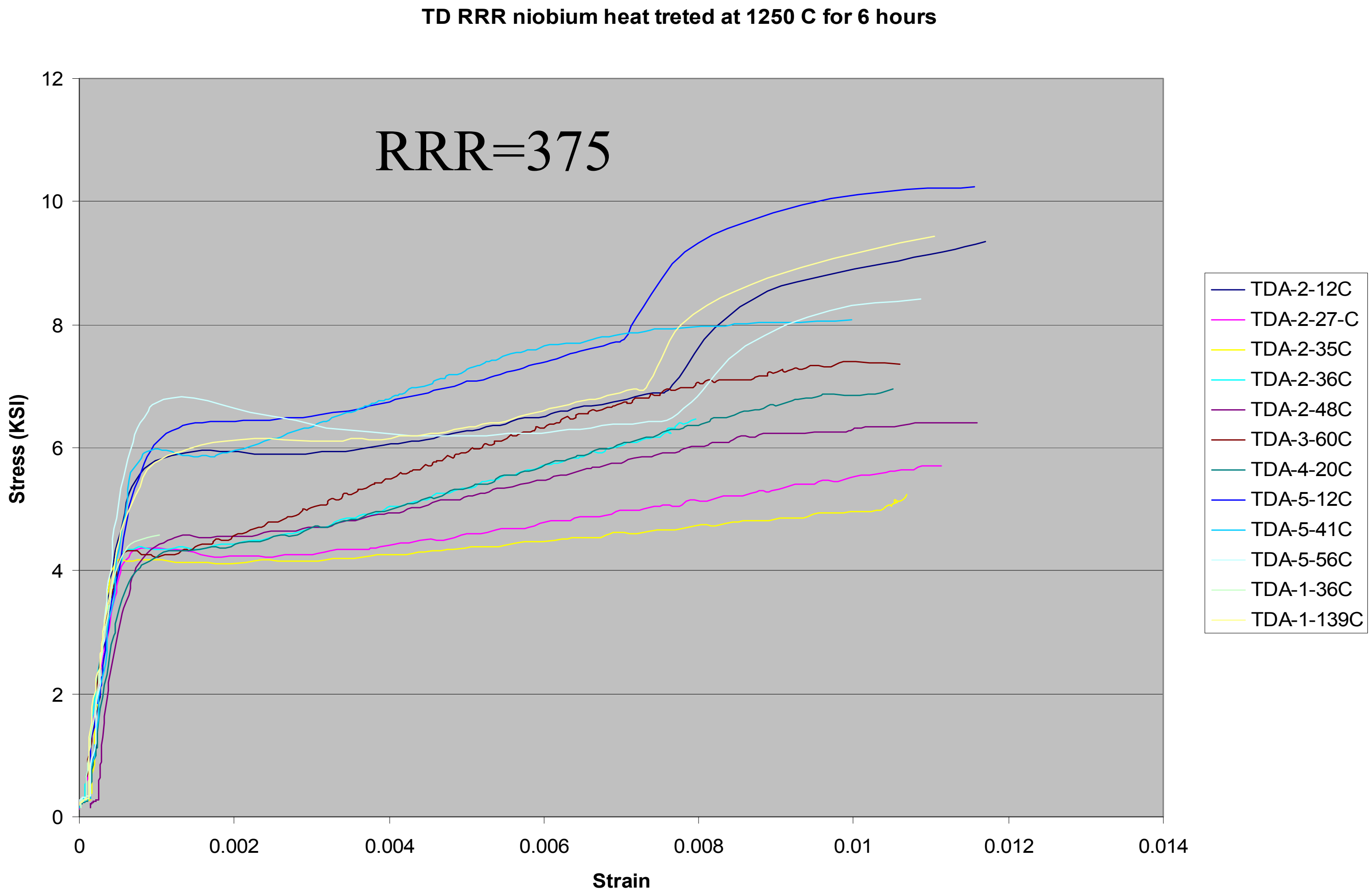
  Strain rate can be varied from 1.e-6 s^{-1} to 10 s^{-1}

- Accuracy of the Stress Measurement ± 1%
- Accuracy of the Percentage of Elongation ± 1%
- Measurements can be made from 4.2 K to ambient temperature

Apparent YM Issue



TD375 stress-strain



TD data summary

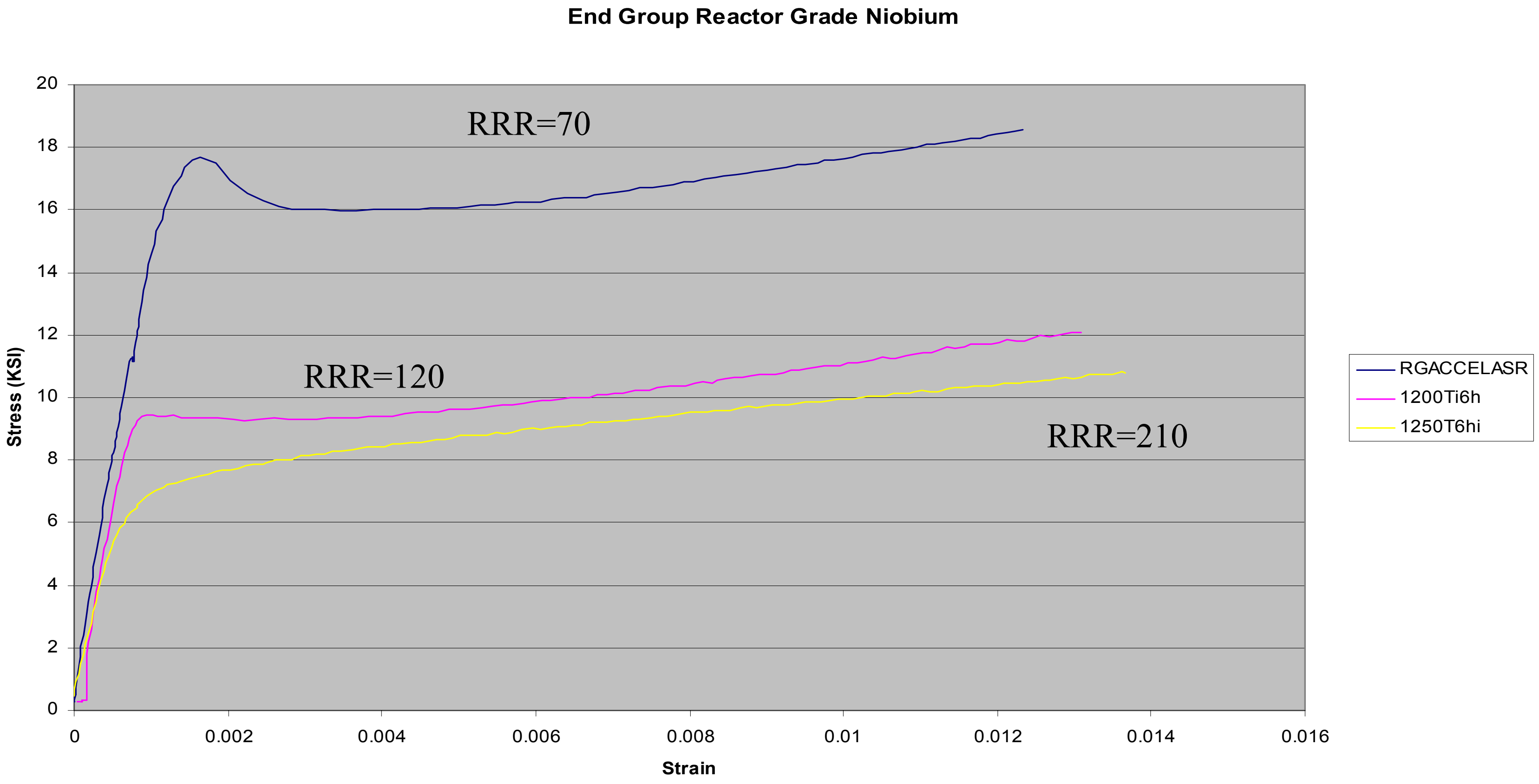
Summary of the TD niobium mechanical properties

Niobium	Yield Strength (KSI)		Tensile Strength (KSI)		% Elongation		RRR	Hv
	SSR	FSR	SSR	FSR	SSR	FSR		
ASR	7.4	7.9	21	24	44	48	260	52
600 C	7.0	7.5	21	22	48	49	300	47
800 C	5.7	--	19	--	47	--	350	43
1250 C	4.5	6.3	15	19	32	33	375	36

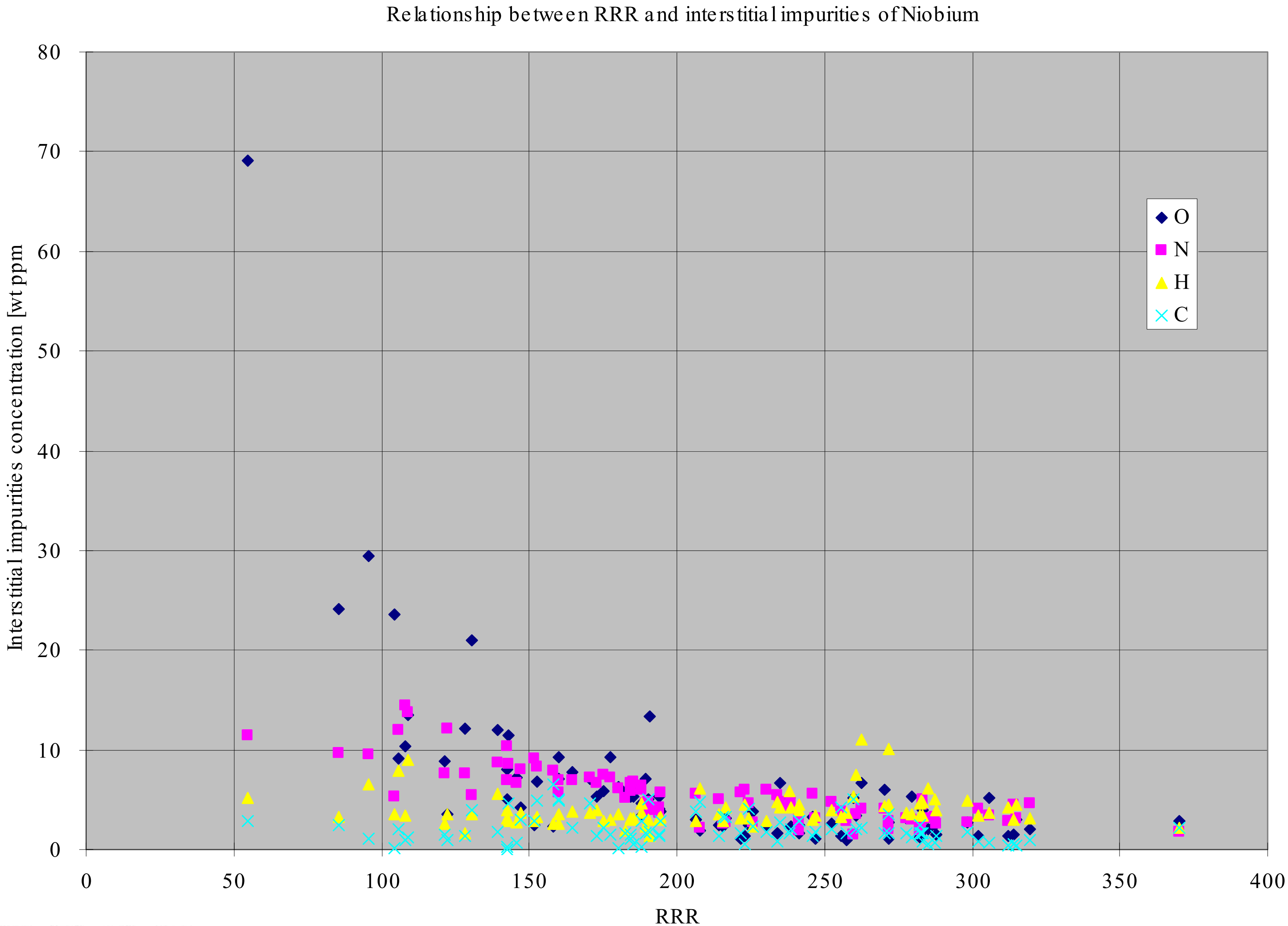
SSR ~ 5.5E-5

FSR ~ 2.0e-4 up to Yield point and 1.0e-3 until break

WC Reactor Grade stress-strain



Interstitials vs RRR



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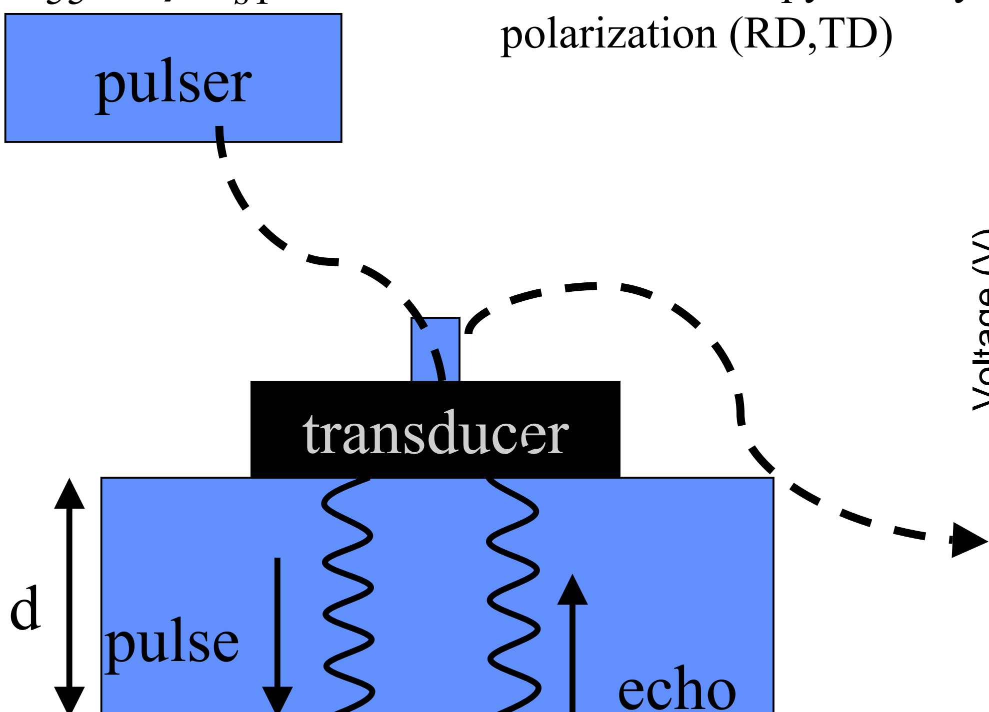
Ultrasonic Velocity Measurements (University of Va)

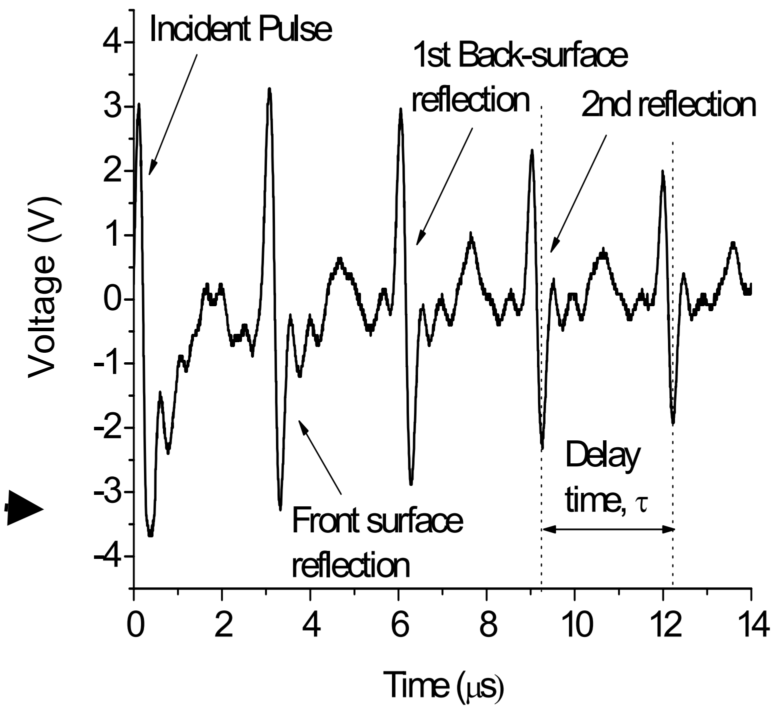
$$C_{33} = \rho v_{l3}^2$$

$$C_{44} = \rho v_{s2}^2$$

$$C_{55} = \rho v_{s1}^2$$

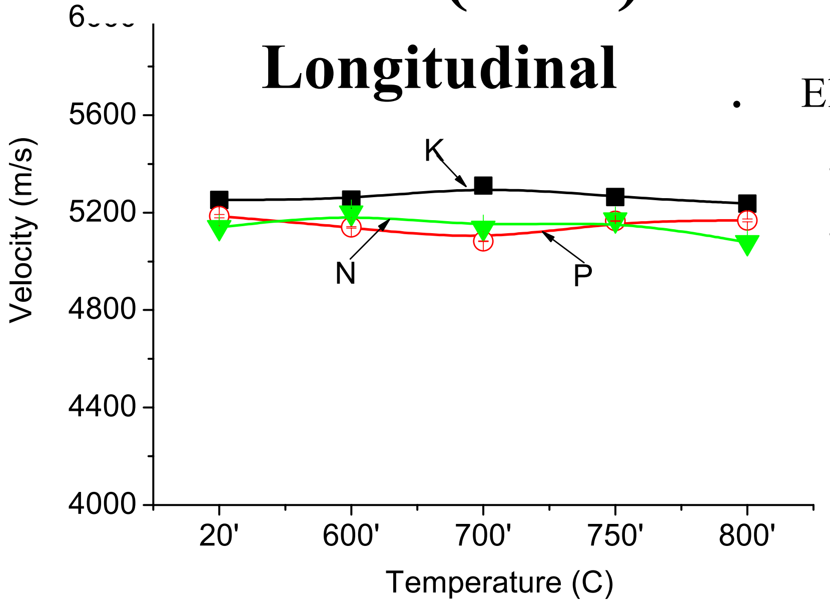
- Elastic properties may be accurately measured by the pulse-echo technique
  - Avoid potential problems of tensile tests (anelasticity, microyielding, etc.)
  - Assess anisotropy directly by changing the direction of shear wave polarization (RD,TD)





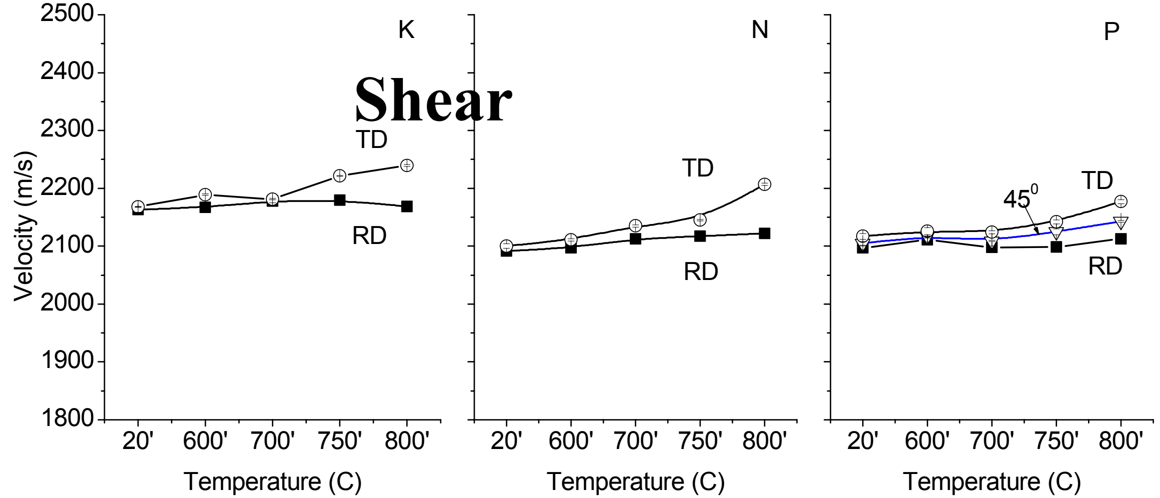
Velocities (UVa)

Longitudinal



- Elastic behavior normal for niobium (E = 110 ± 3 GPa)
  - Anisotropy small (β ≤ 1.03)
  - Reproducible trend with annealing in all lots

Shear



RRR Niobium Rolled Sheet Textured

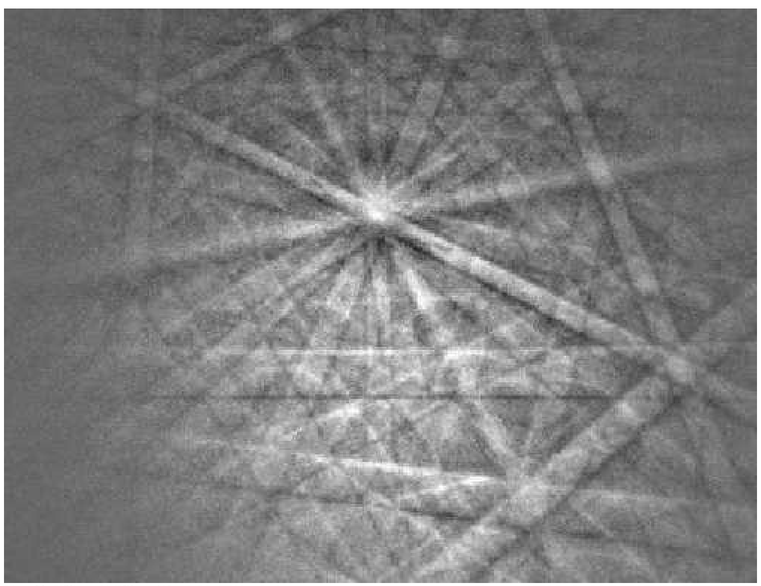
- Due to deformation processing and recrystalization
- Anisotropy of single crystal manifest in textured polycrystal
  - Nb Zener anisotropy parameter, Z = 0.55, is low (many cubic metals Z>1)

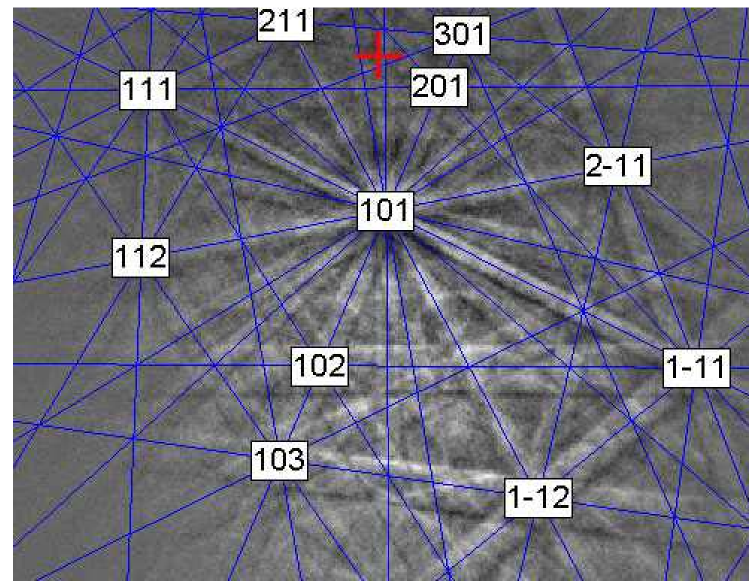
$$Z = \frac{2C_{44}}{C_{11} - C_{12}}$$

- May predict response of polycrystal response if single crystal behavior and texture are known.
  - Using averaging schemes such as Voigt, Ruess, Hill
- Initial studies using x-ray diffraction of the sheet surface showed no change in the texture with annealing.

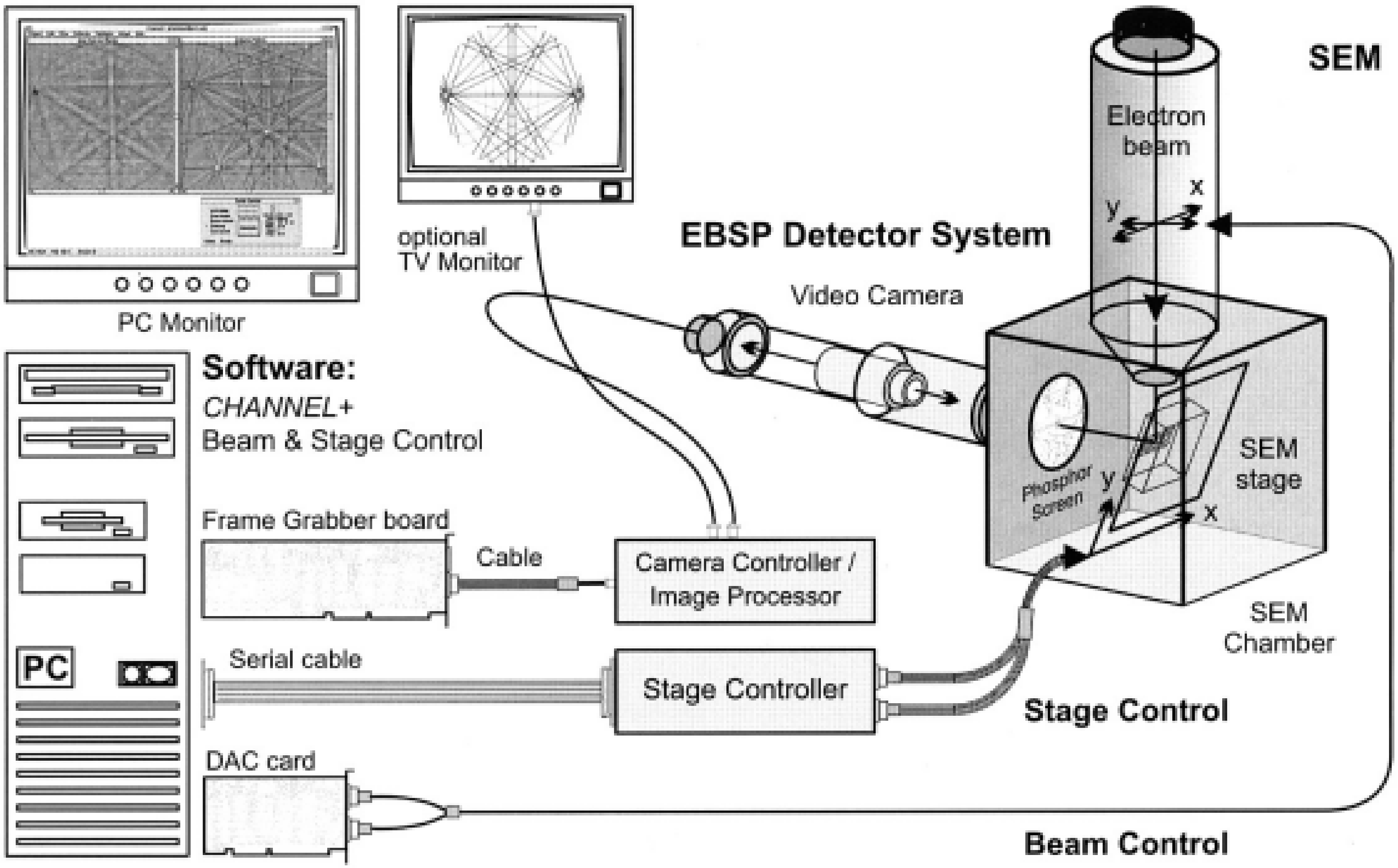
Electron Backscattered Diffraction (EBSD)

- Utilizes automated indexing diffraction patterns
- Provides a method for examining crystallography and crystallographic orientation at a single point in materials
  - grain boundary crystallography
  - intragranular substructure (mosaic)
  - microtexture



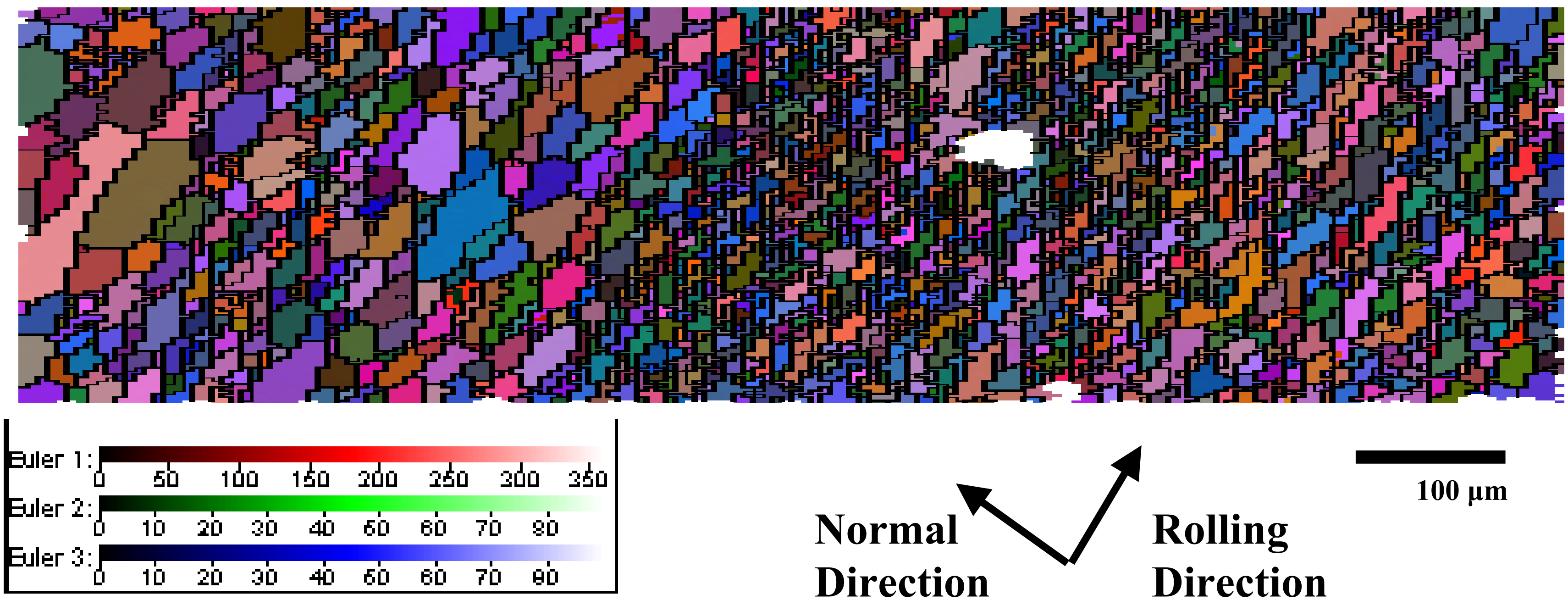


Schematic of EBSD

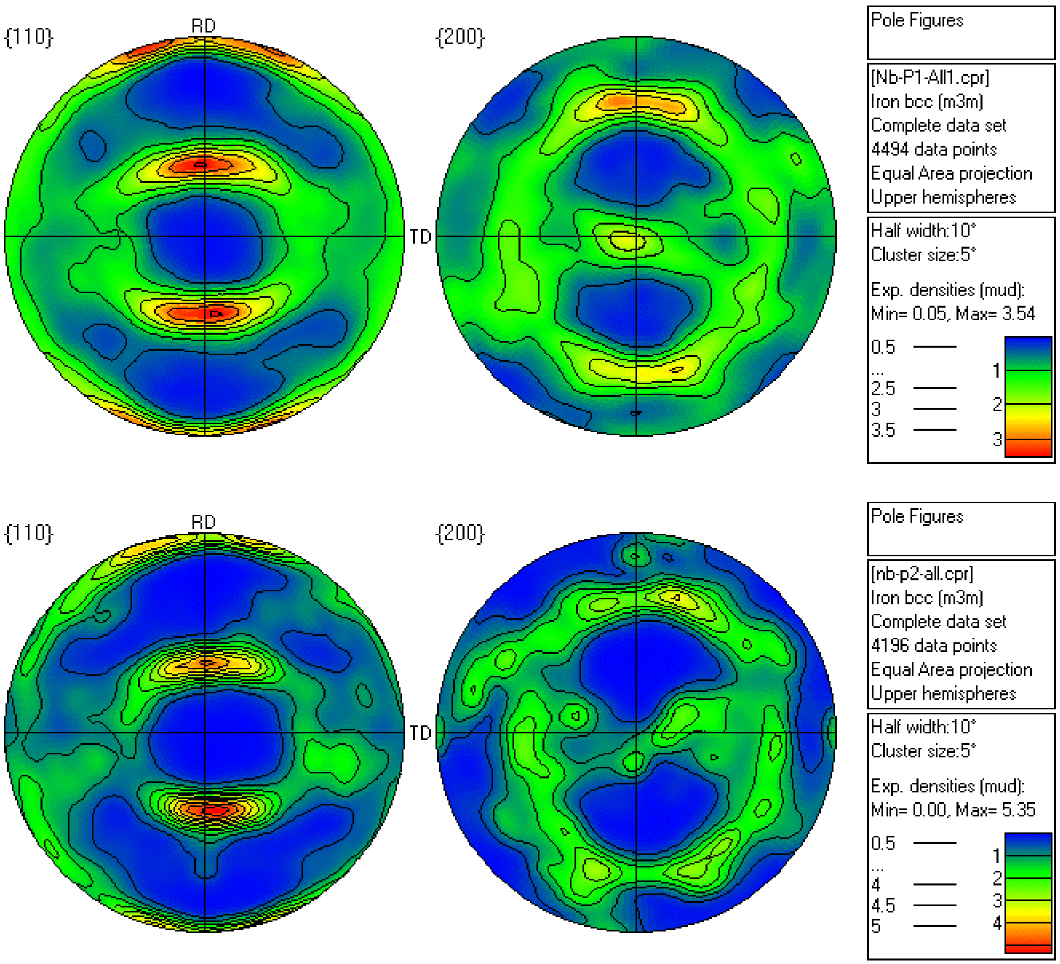


EBSD maps through sheet thickness (UVa)

- As-received P sample exhibits bands of fine and coarse grains typical of these samples.
- Different colors represent orientations as shown in the legend, where the Euler angles are according to Bunge's convention
- White spots represent positions were surface roughness obscured the EBSD detector

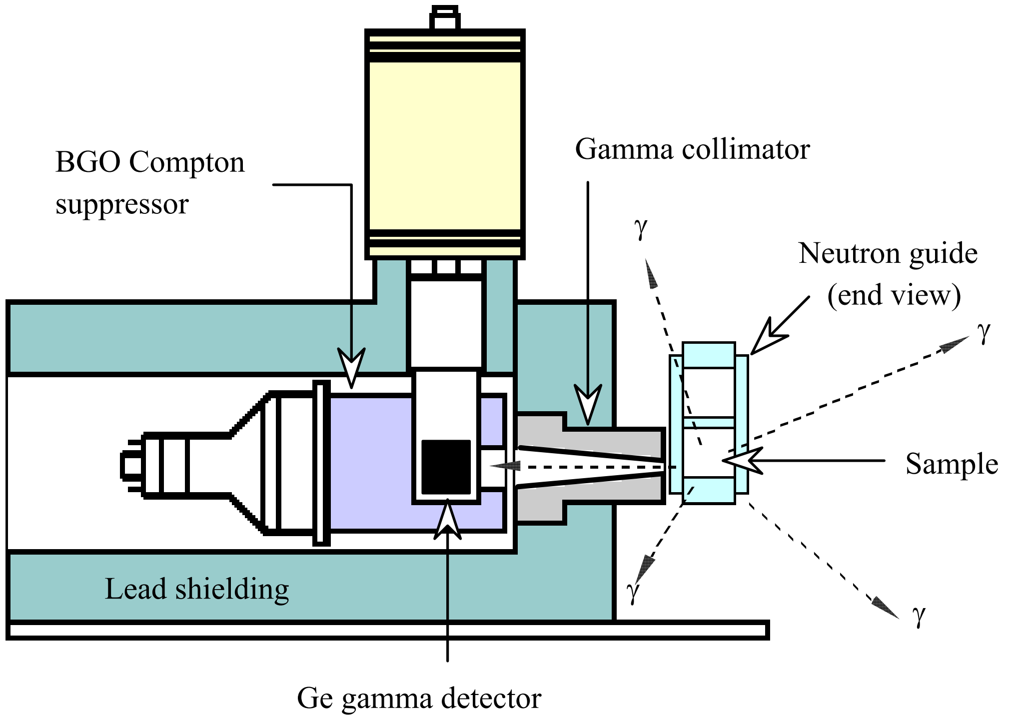


EBSD maps of sheet cross-section provide bulk texture (UVa)

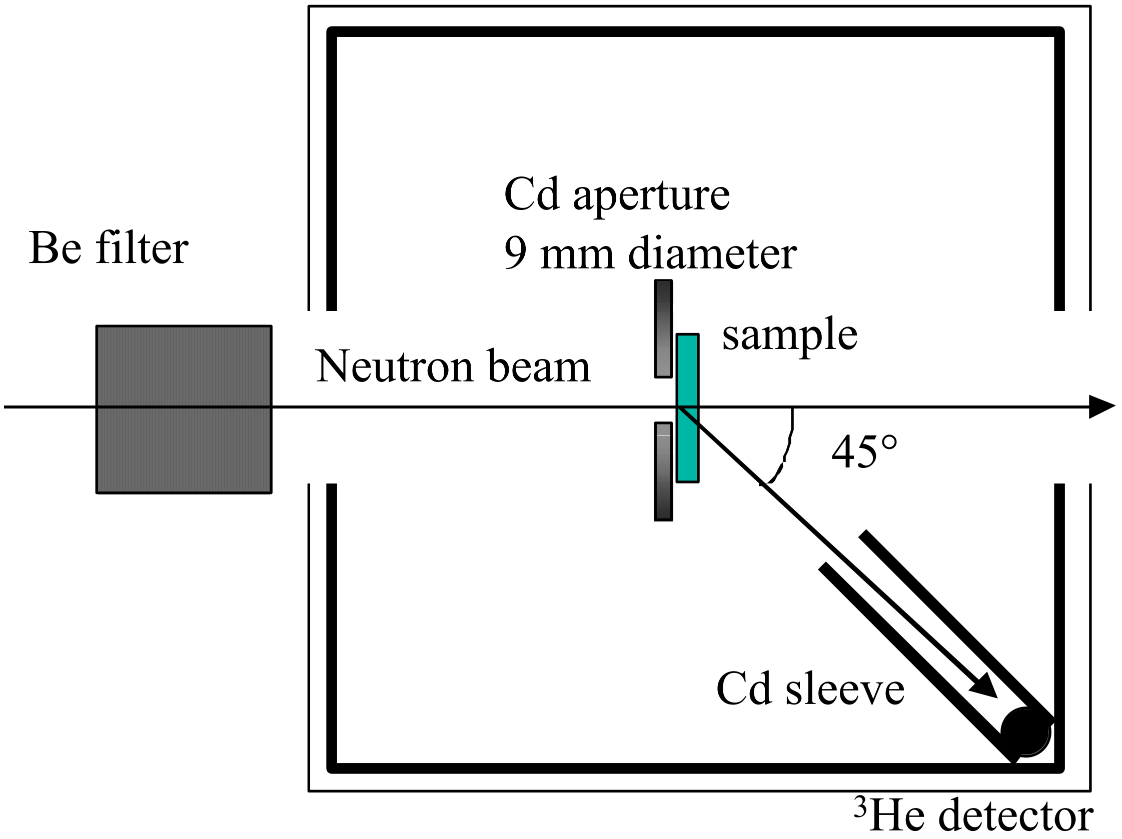


- Pole figures from as-received and annealed (800C) samples (lot P) reveal two facts:
  - ◆Less cube component than in surface (x-ray).
  - ◆Strengthening of texture with annealing

Cold neutron PGAA spectrometer at the NIST Center for Neutron Research



Schematic drawing for the NIS measurement setup



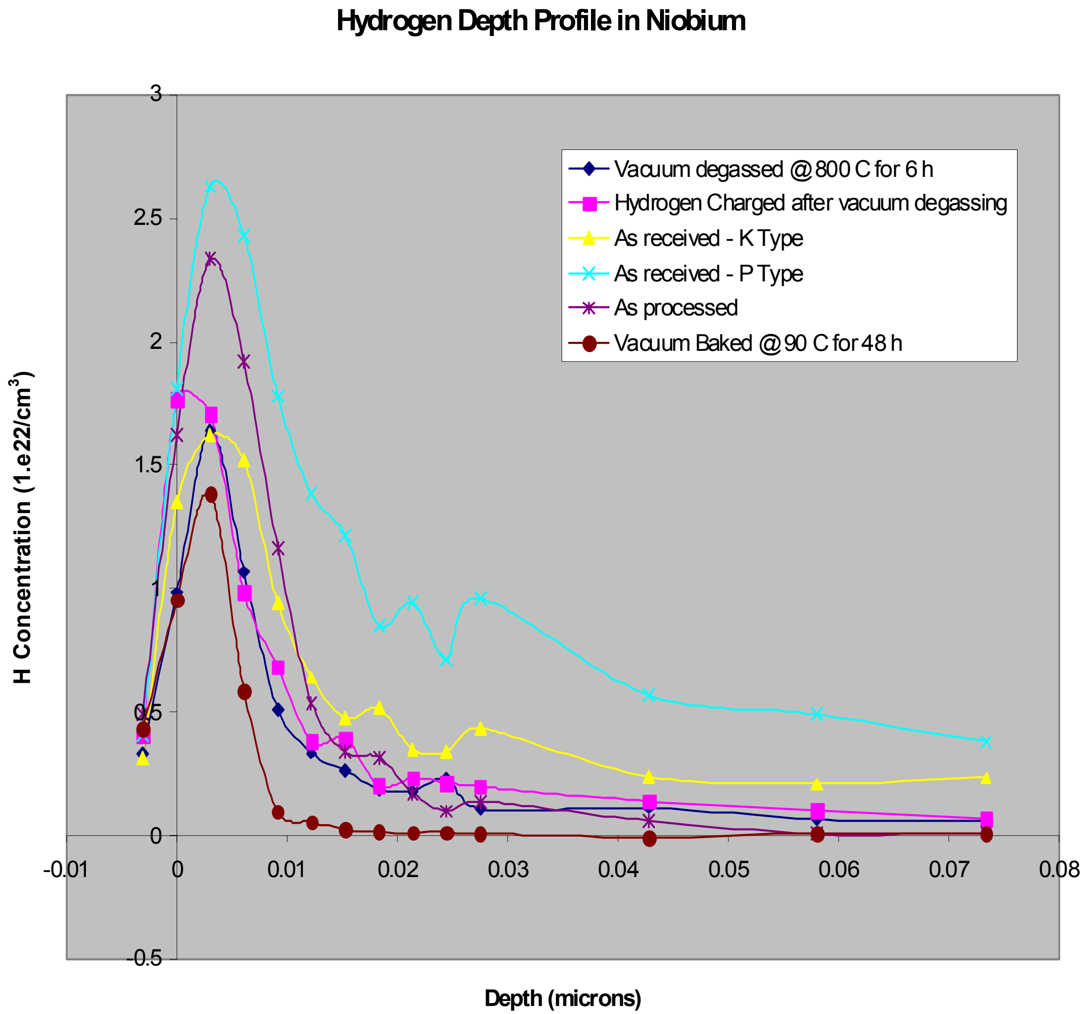
Treatment of niobium samples measured

Sample	Measured as received?	Measured following:	Measured following:	Measured following:	
	(No heating)	Vacuum heating 1	Acid treatment 1 duration (min)	Vacuum heating 2	Acid treatment 2 duration (min)
Nb 2	No	800 °C / 6 h	1	---	---
Nb 3	No	700 °C / 6 h	2	---	---
Nb 4	No	750 °C / 6 h	3	800 °C / 6 h	---
Nb 5	No	600 °C / 10 h	0.5	800 °C / 6 h	5
Nb 6	Yes	----	4	---	---
P1	Yes	800 °C / > 48 h	----	----	----
P2	Yes	800 °C / > 48 h	----	----	----
K1	Yes	800 °C / > 48 h	----	----	----
K2	Yes	800 °C / > 48 h	----	----	----

Hydrogen fractions measured in the niobium samples

Sample	**% atom fraction hydrogen (determined relative blank)**					
	As received	After vacuum heating #1	After acid treatment #1		After vacuum heating #2 (Nb 4) and acid treatment #2 (Nb 5)	
		PGAA	PGAA	NIS	PGAA	NIS
Nb 2	---	0.138 ± 0.025	0.257 ± 0.041	0.153 ± 0.032	---	---
Nb 3	---	0.156 ± 0.026	0.169 ± 0.028	0.167 ± 0.034	---	---
Nb 4	---	0.122 ± 0.028	0.114 ± 0.027	0.083 ± 0.020	≤ 0.03	≤ 0.03
Nb 5	---	0.107 ± 0.025	0.107 ± 0.025	0.113 ± 0.024	0.396 ± 0.037	0.352 ± 0.070
Nb 6	0.126 ± 0.027	---	0.140 ± 0.028	0.150 ± 0.032	---	---
P1	0.03 ± 0.02	≤ 0.03	---	---	---	---
P2	0.03 ± 0.02	≤ 0.03	---	---	---	---
K1	≤ 0.04	≤ 0.03	---	---	---	---
K2	0.03 ± 0.02	≤ 0.03	---	---	---	---

NRA Hydrogen depth profiles



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Dynamic Mechanical Analysis

(Internal Friction)

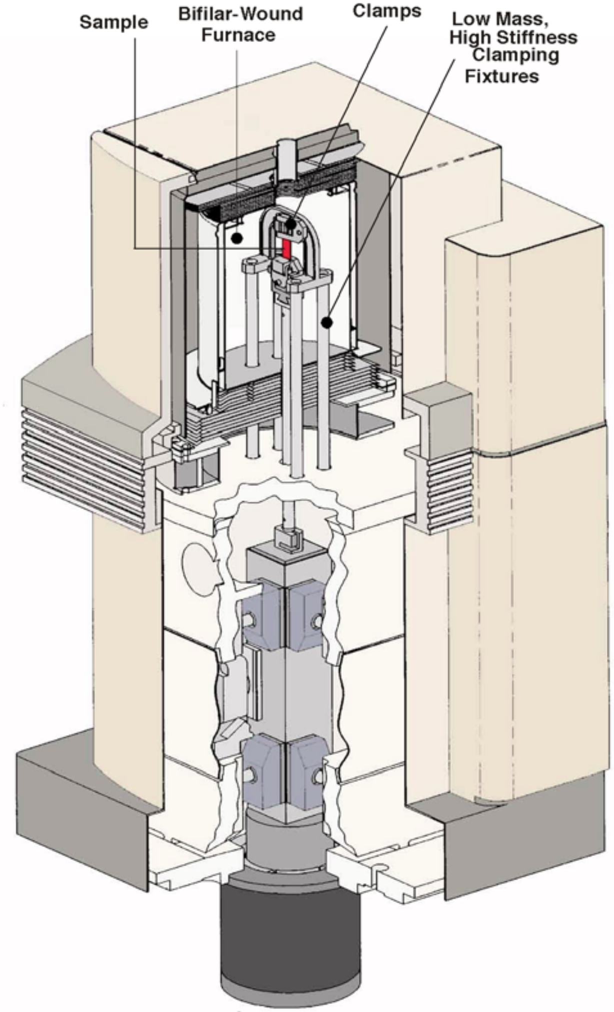
Measures the phase shift between stress and strain as a function of temperature

Hydrogen and Other Interstitials can Have Four Different Types of Interactions:

1. Snoek - Dislocation drag (diffusion)
2. Precipitation - Hydrides, etc.
3. Pairing Effects - solute-solvent or solute-solute pair interactions (e.g. Zener effect)
4. Gorsky - Large scale diffusion effects

Other Peaks:

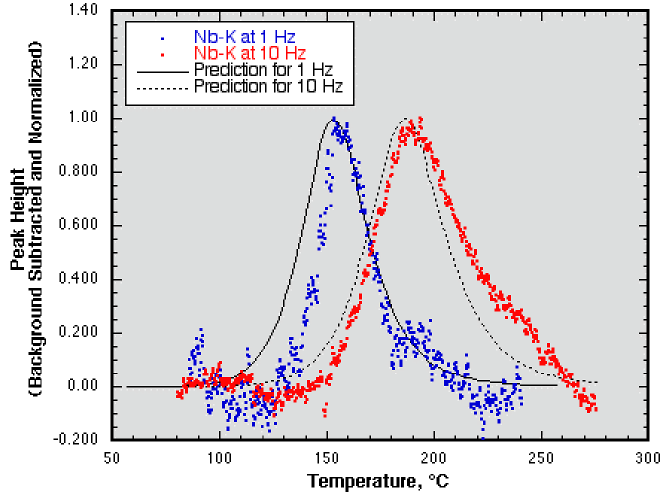
1. Bordoni - Cold work and dislocation density related
2. Grain Boundary - Interface sliding



Note diagram shows tensile grip. The measurements of this study were conducted in 3 point bend.

Dynamic Mechanical Analysis (Contd.)

Temperature - Relaxation Mechanism Kinetics
Magnitude - Proportional to Concentration

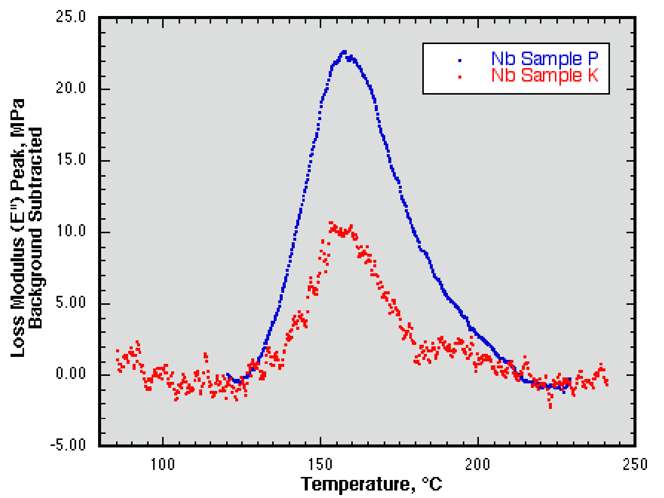


Peak Temperature = f(Frequency)

Comparison of peaks predicted from diffusion data for two frequencies to measurements.

Estimated Snoek Peak Temperatures (K) in Nb for Different Loading Frequencies

Solute	0.1 Hz	1.0 Hz	10 Hz
H	50	53.5	58
C	477	511	550
N	494	528	567
O	397	426	459

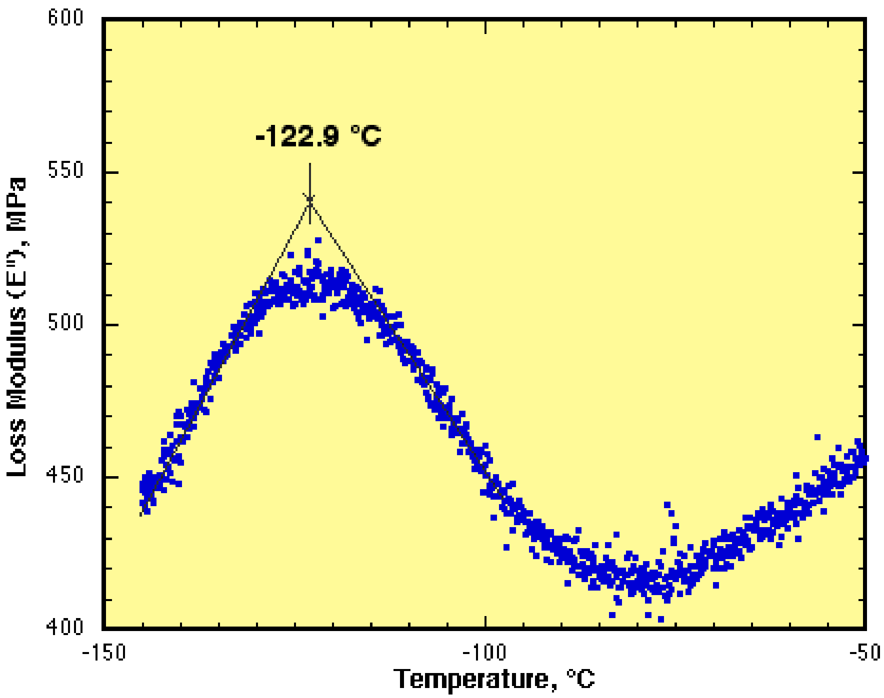


Peak Magnitude = f(Concentration)

Comparison of the oxygen Snoek peak in two samples of Nb indicated a difference in the oxygen concentration

Dynamic Mechanical Analysis (Contd.)

Example of other types of interactions and peaks



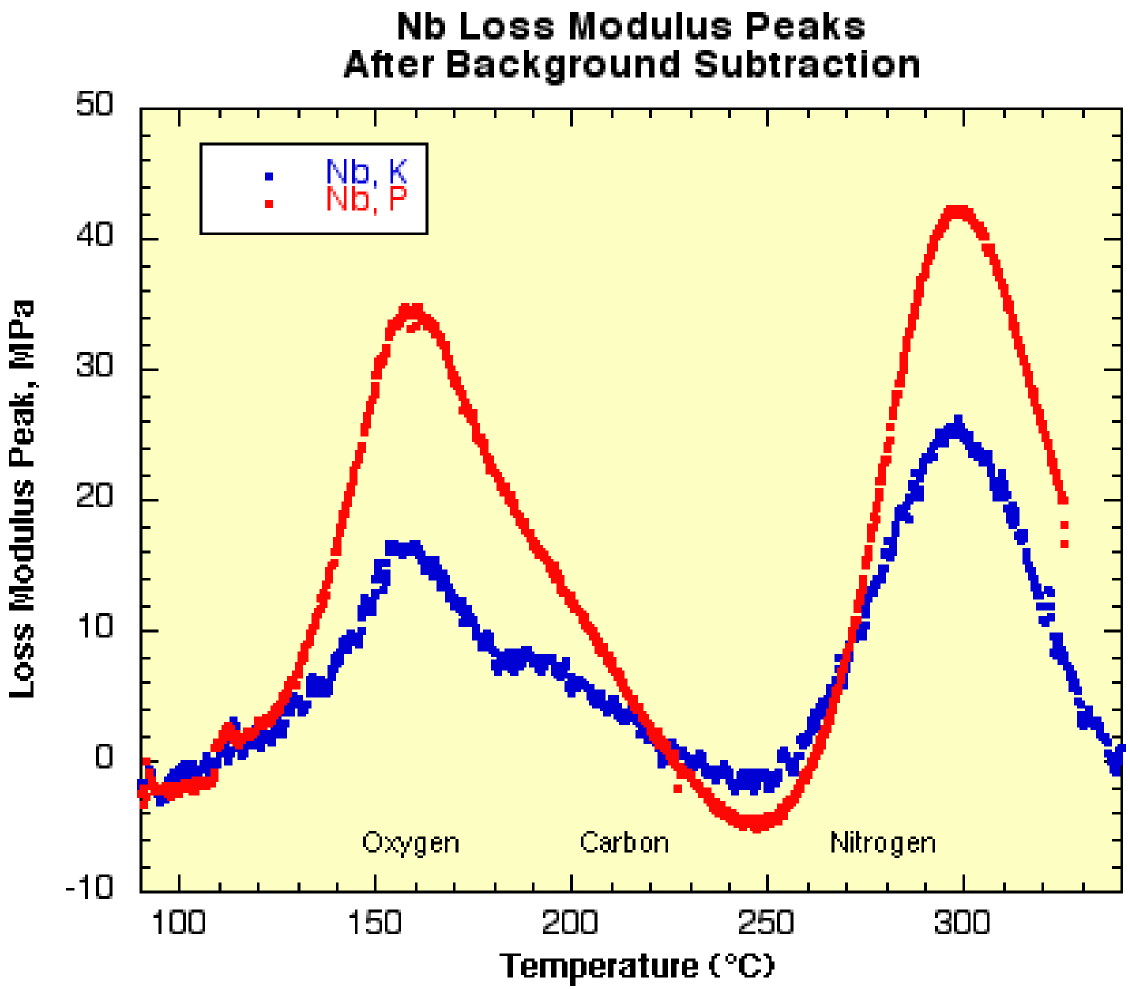
<u>H-O Interaction Peak</u>

A peak was found at a temperature between that expected for the H and O Snoek peaks. Previous investigators postulated this peak to be due to H-O interstitial interaction.

Estimated Snoek Peak Temperatures (K) in Nb for Different Loading Frequencies

Solute	0.1 Hz	1.0 Hz	10 Hz
H	50	53.5	58
C	477	511	550
N	494	528	567
O	397	426	459

Loss Modulus Peaks for Nb



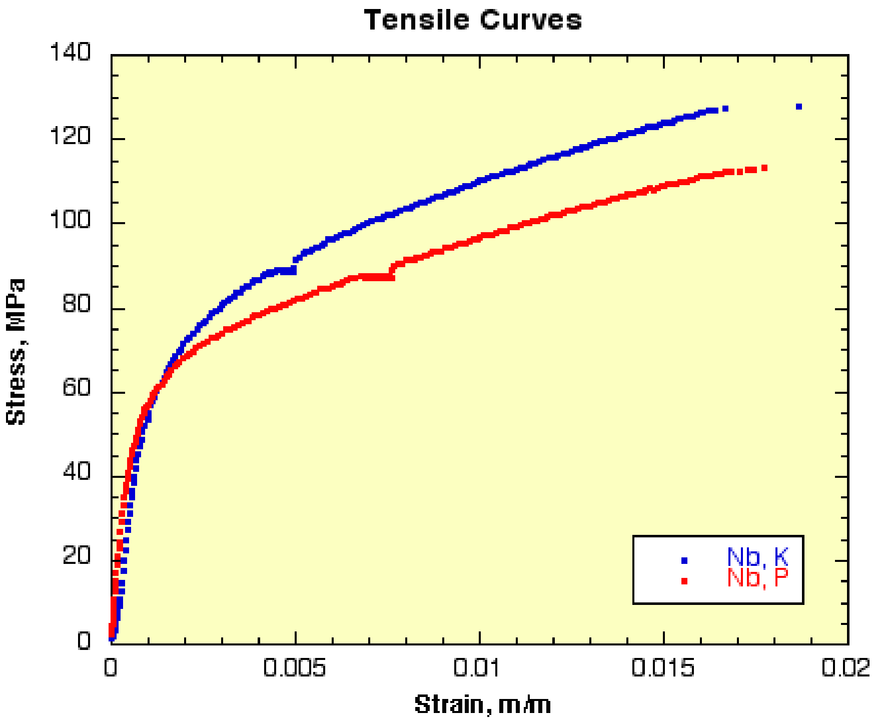
Peak Ratios

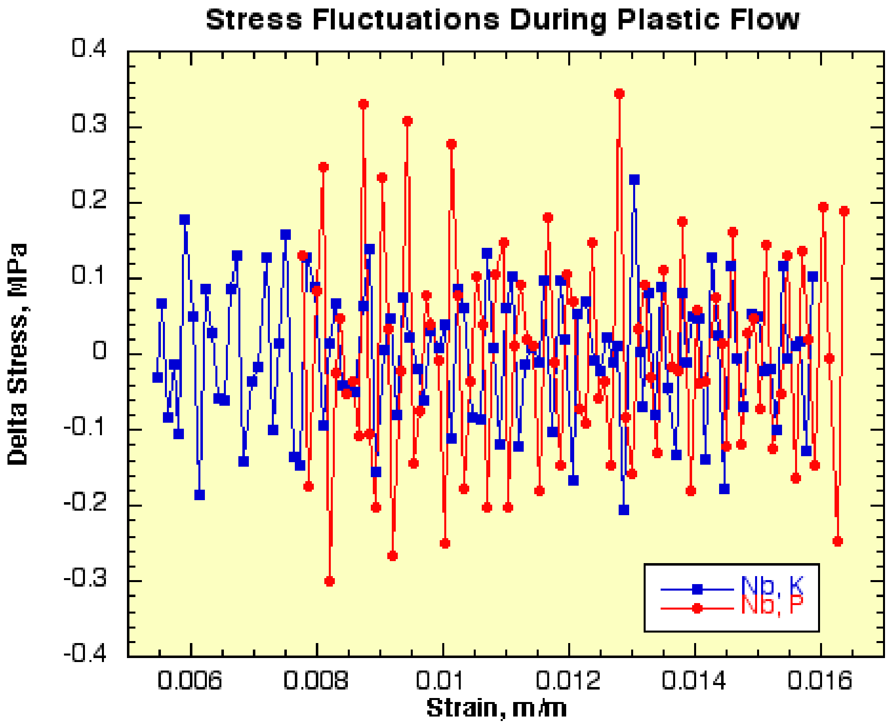
Oxygen: P/K ≈ 1.94

Nitrogen: P/K ≈ 1.6

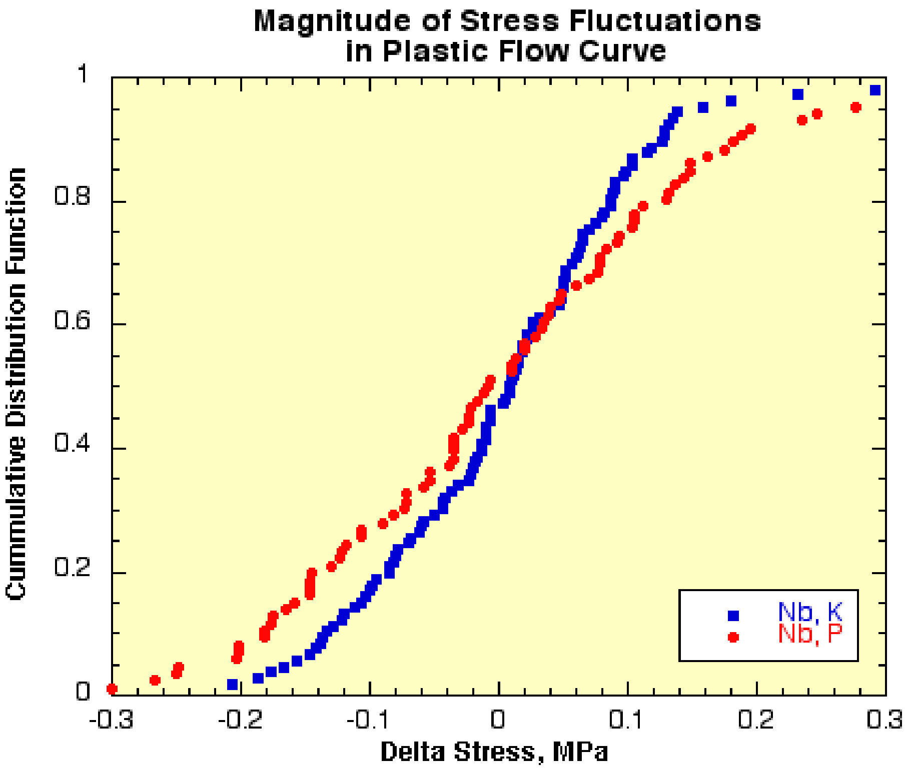
Carbon: P/K NA

Tensile Deformation and Stress Fluctuations During Plastic Flow





Distribution of Stress Fluctuations During Plastic Flow



Standard Deviations

Nb, P = 0.1427 MPa

Nb, K = 0.0907 MPa

Ratio P/K = 1.57

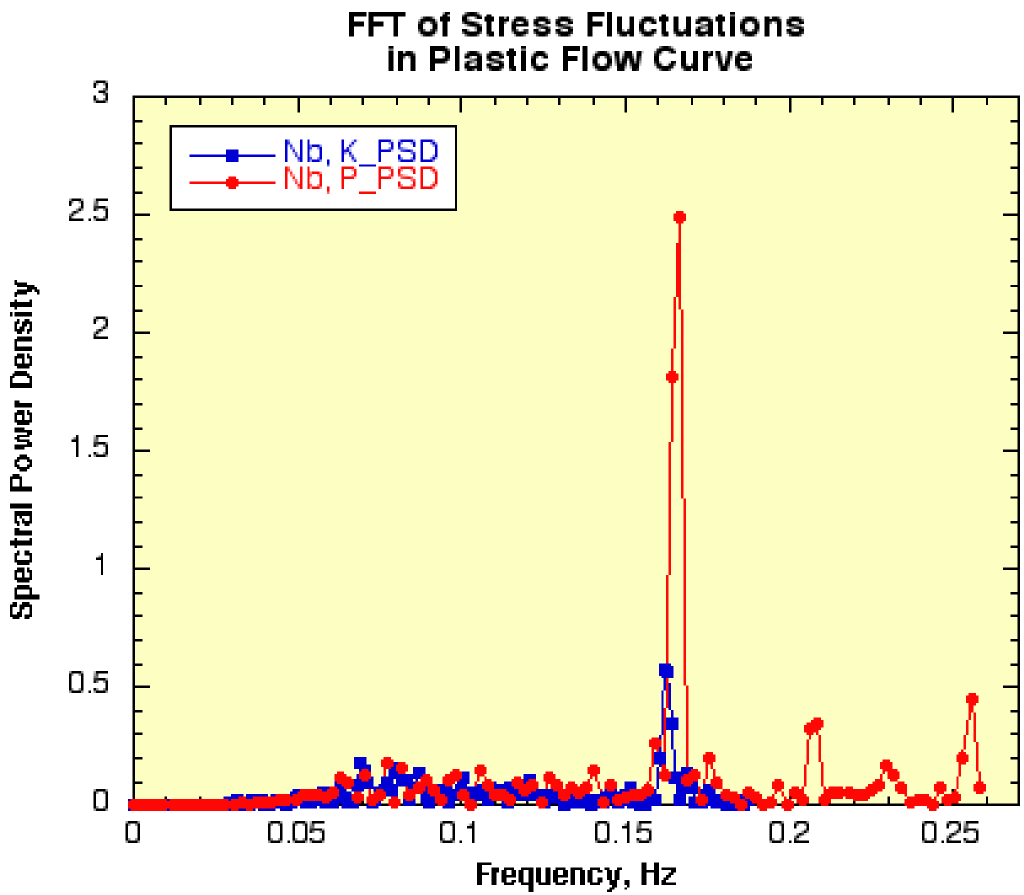
Loss Modulus Peak Ratios

Oxygen: P/K ≈ 1.94

Nitrogen: P/K ≈ 1.6

Carbon: P/K NA

Analysis of Periodicity of the Stress Fluctuations During Plastic Flow



SPD Peak

Nb, P = 2.49

Nb, K = 0.56

Ratio P/K = 4.44 (2.11)

Standard Deviations

Nb, P = 0.1427 MPa

Nb, K = 0.0907 MPa

Ratio P/K = 1.57

Loss Modulus Peak Ratios

Oxygen: P/K ≈ 1.94

Nitrogen: P/K ≈ 1.6

Carbon: P/K NA

2002 Hydrogen in Materials and Vacuum Systems Intl. Workshop

- **This International Meeting, the First of its Kind, was Jointly Sponsored by**
  - **The Mid-Atlantic Chapter of the American Vacuum Society,**
  - **The Old Dominion University (Physics Department),**
  - **Deutsches Electronen – Synchrotron (DESY),**
  - **The College of William and Mary,**
  - **Reference Metals Company Inc.,**
  - **Tokyo Denkai Co., Ltd. (Japan),**
  - **Wah Chang (USA) and**
  - **The Jefferson Lab.**
- **Success Lead to the Formation of International Hydrogen in Matter Symposia Board (ISOHIM): Chair – Dr. Jim Miller, ANL; Co-Chair – Dr. G. Myneni JLAB**
- **Second International Symposium on Hydrogen in Matter 2005 – Uppsala University, Sweden**
- **AIP Published the Proceedings of the workshop as CP #671 in July 2003**

High RRR Nb–Hydrogen Research Collaborations

JLAB – ODU Physics Hydrogen Interactions in Materials – Theory

. JLAB – UVa Physics	H in Nb Grain Boundaries – SQUID Magnetic Microscope
. JLAB – NIST	H–PGAA–Neutron Scattering Studies of high RRR Niobium
. JLAB – NIST	Internal Friction Measurements of H and Interstitial Content in High RRR Niobium
. JLAB – SUNY Albany	Hydrogen Depth Profile with Nuclear Reactions & Oxide Thickness in High RRR Niobium
. JLAB – DESY, Germany	Thermal and Mechanical Properties of Niobium With Heat Treatments – H Degassing Affects
. JLAB – FZK, Germany	Extreme High Vacuum Science & Technology – H
. JLAB – William & Mary	Hydrogen in Semiconductors
. JLAB–LANL–UVa – Reference Metals – MOU	High RRR Niobium Microstructure – Tuning with H
JLAB – Uppsala Univ.	Fundamental Understanding of H-Matter Interactions

Conclusions

- High purity niobium mechanical properties vary from batch to batch and are very sensitive to various treatments and handling
- Elastic behavior of high purity niobium is normal and is unaffected by heat treatments
- Hydrogen bulk content and depth profile studies are yielding interesting results
- Internal Friction measurements are expected to provide quantitative interstitial concentrations
- Stress-Strain curves may provide the high RRR Nb purity
- Fundamental hydrogen-matter interactions knowledge from various communities will enhance the understanding and improve the SRF Technology

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