

# **Optimization of RRR Niobium Bulk Properties**

**Pushing the Limits of the RF  
Superconductivity**

**ANL RFSC Workshop**

**22-24, Sept. 2004**

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# Types of high RRR bulk niobium

## 1. Polycrystalline niobium from standard process including rolling:

**No control on the microstructure, texture**

**Heterogeneous**

## 2. High Tantalum content niobium may be available at reduced cost

## 3. Single crystal ingots with required orientation

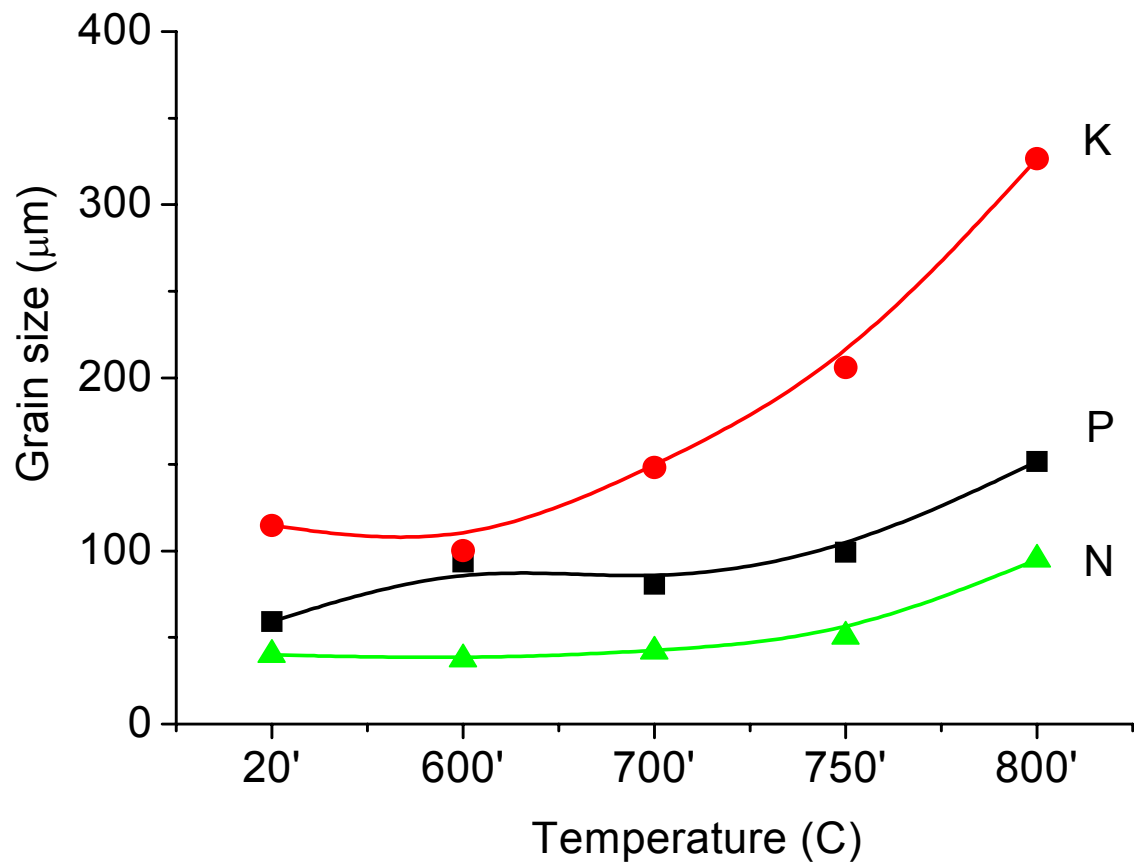
**One obtains the sheets directly from the slicing of the ingots, reduced process steps leading to lower costs**



## What do we know about RRR Nb !?

- We like to have highest RRR (Thermal Conductivity for Thermal Stability)
- 30 – 40 % Percentage of Elongation for forming the half cells
- We specify certain Yield Strength!!!!
- We have Specifications for impurity content (But can't Verify with the available techniques)
- We have no control on the major properties of RRR Nb!!!!
- So there are opportunities to optimize these properties

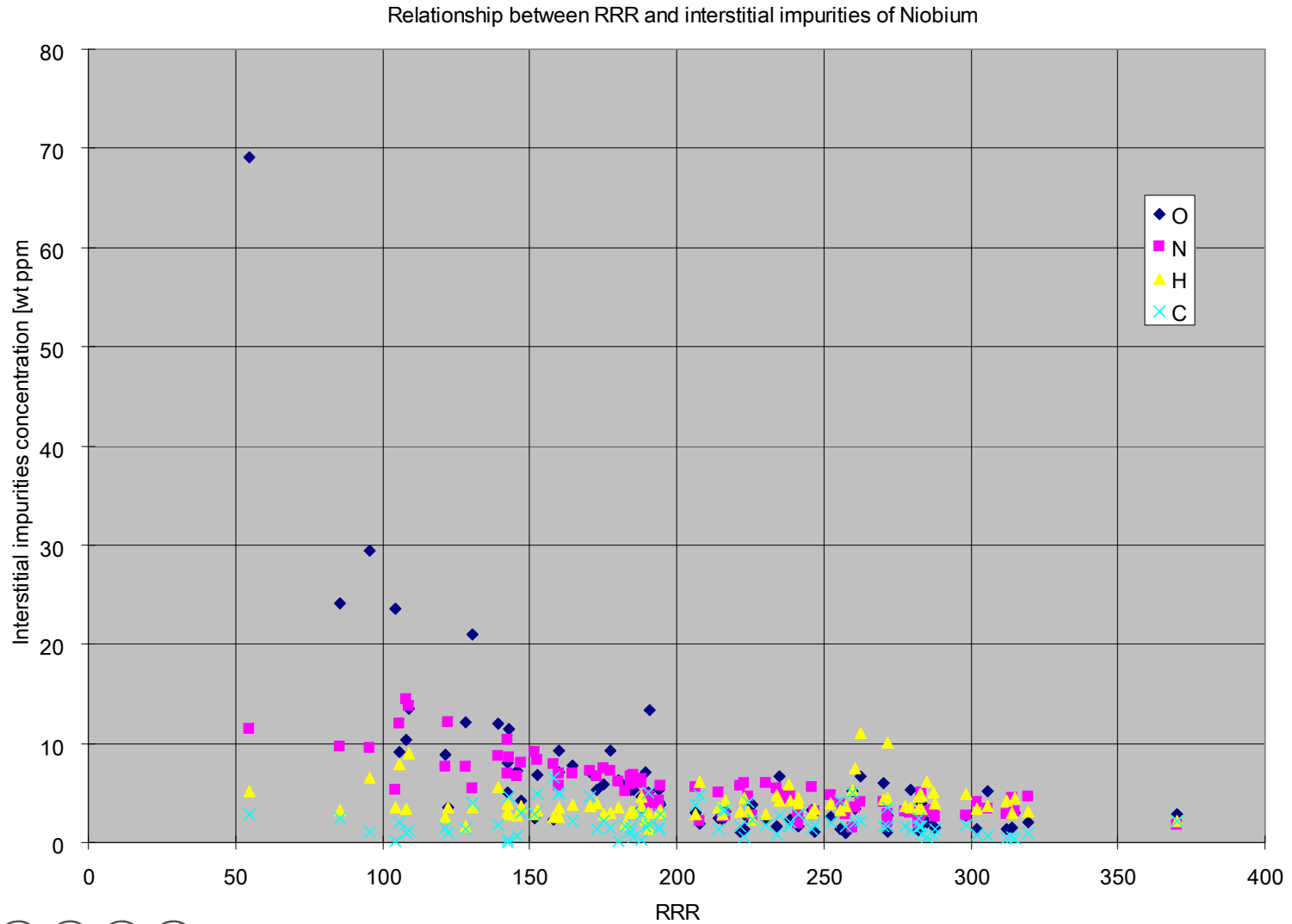




Lineal intercept grain size as a function of annealing temperature for the three lots of high-purity niobium



# Interstitials vs RRR



## Dynamic Mechanical Analysis

### (Internal Friction)

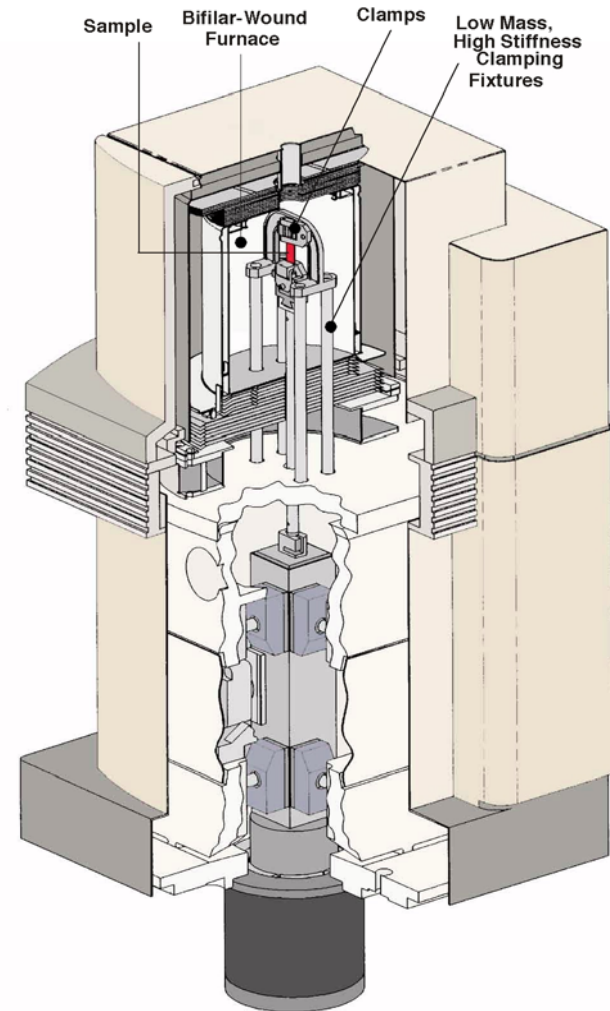
*Measures the phase shift between stress and strain as a function of temperature*

#### 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

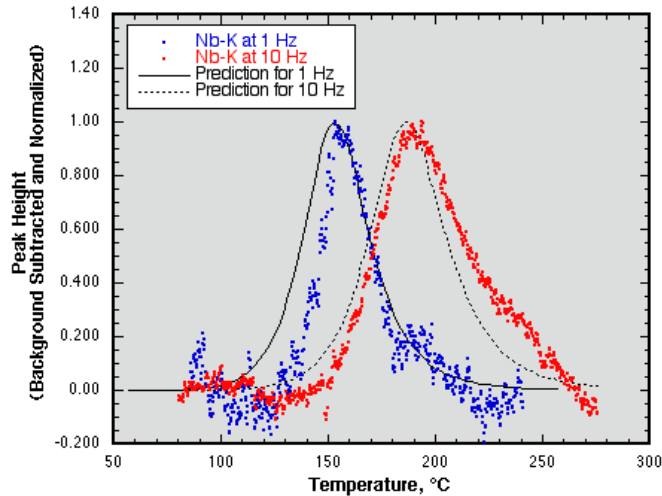


*Note diagram shows tensile grip.  
The measurements of this study were  
conducted in 3 point bend.*



## Dynamic Mechanical Analysis (Contd.)

*Temperature - Relaxation Mechanism Kinetics*  
*Magnitude - Proportional to Concentration*

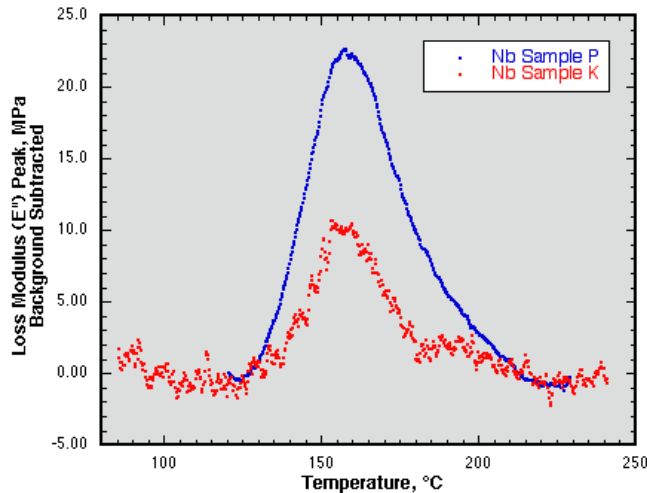


**Peak Temperature = f(Frequency)**

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



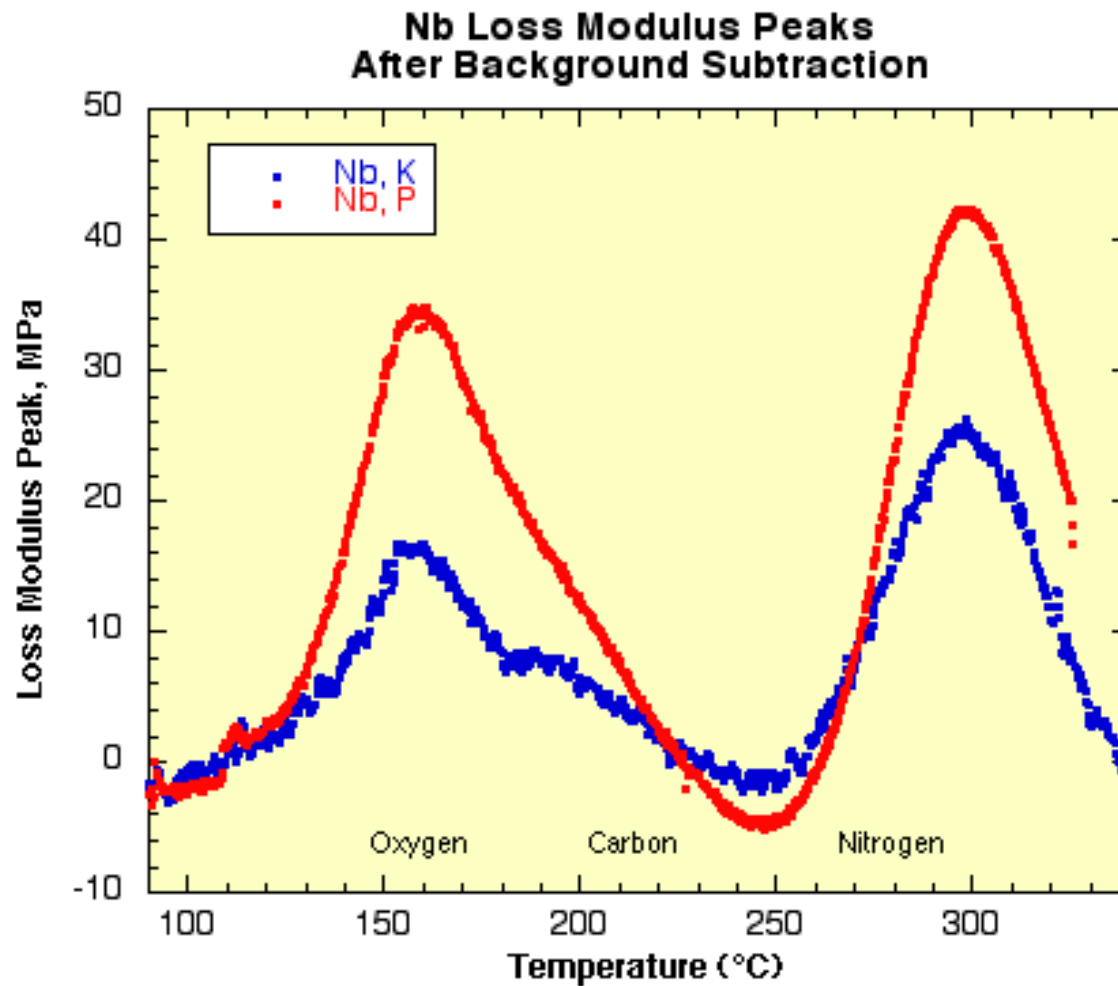
**Peak Magnitude = f(Concentration)**

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



## Dynamic Mechanical Analysis (Contd.)

# Loss Modulus Peaks for Nb



### Peak Ratios

Oxygen: P/K  $\approx$  1.94

Nitrogen: P/K  $\approx$  1.6

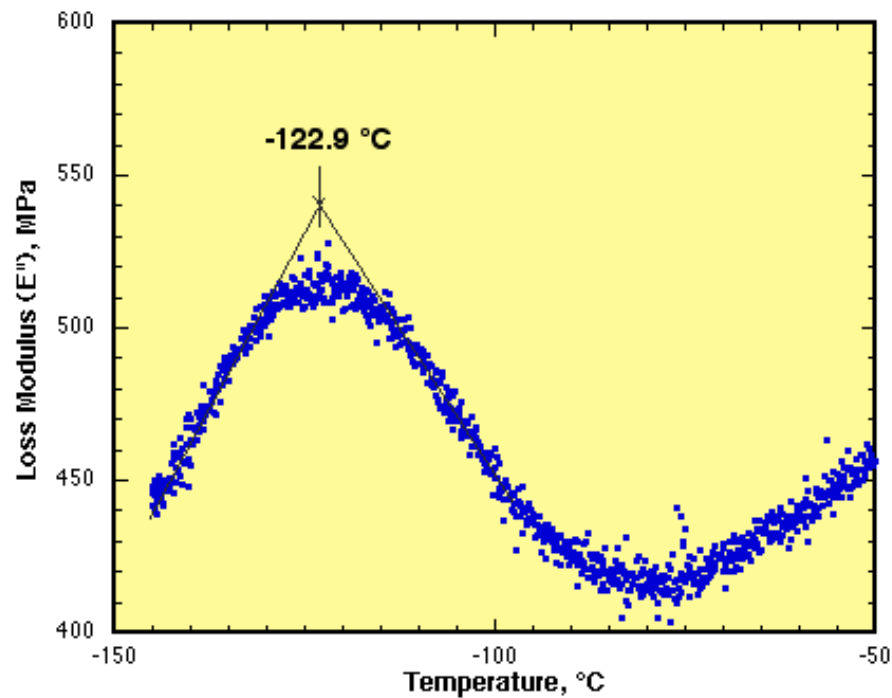
Carbon: P/K NA





## Dynamic Mechanical Analysis (Contd.)

*Example of other types of interactions and peaks*



### H-O Interaction Peak

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.

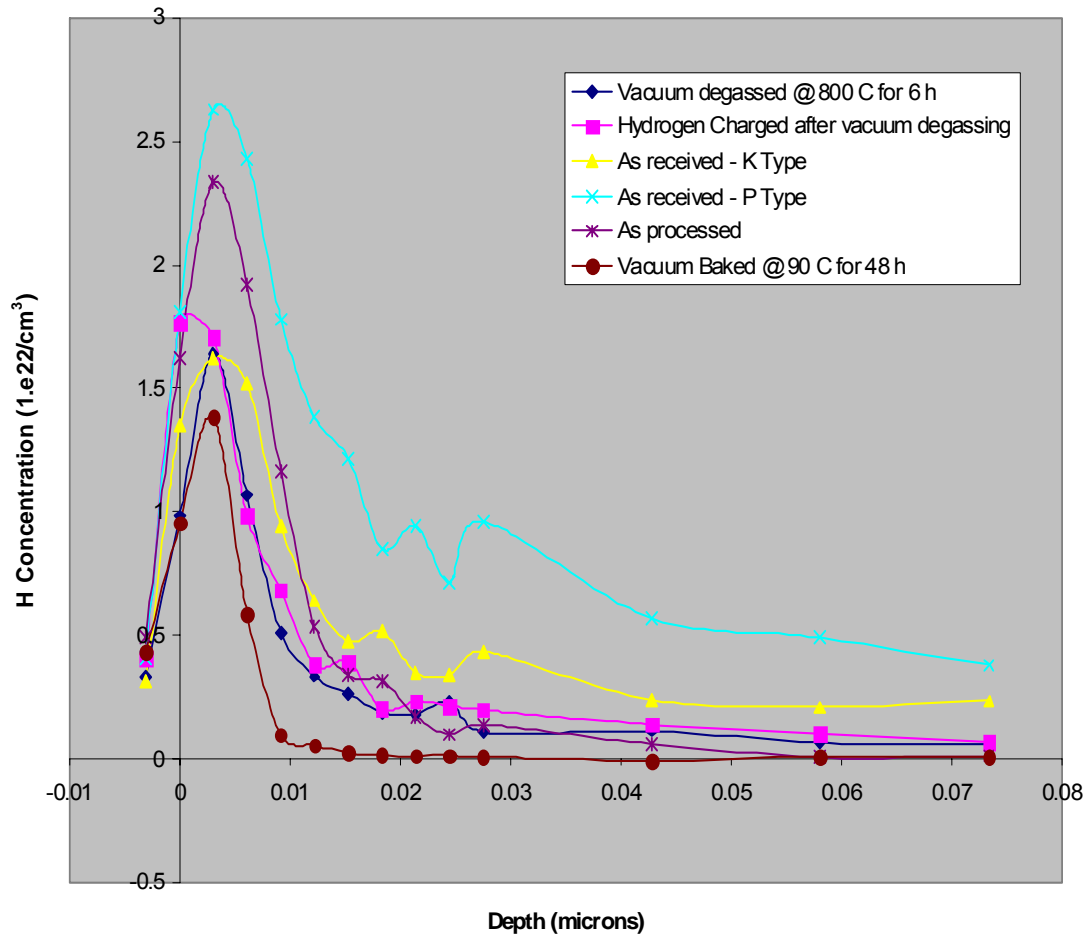
**Estimated Snoek Peak Temperatures (K)  
in Nb for Different Loading Frequencies**

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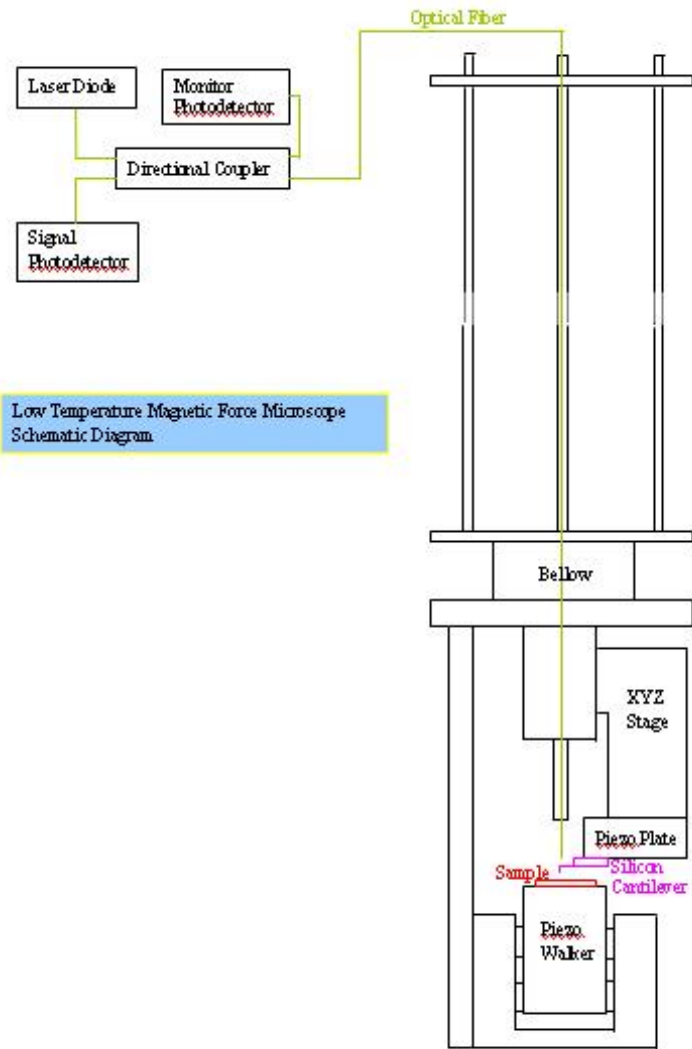


# NRA Hydrogen depth profiles

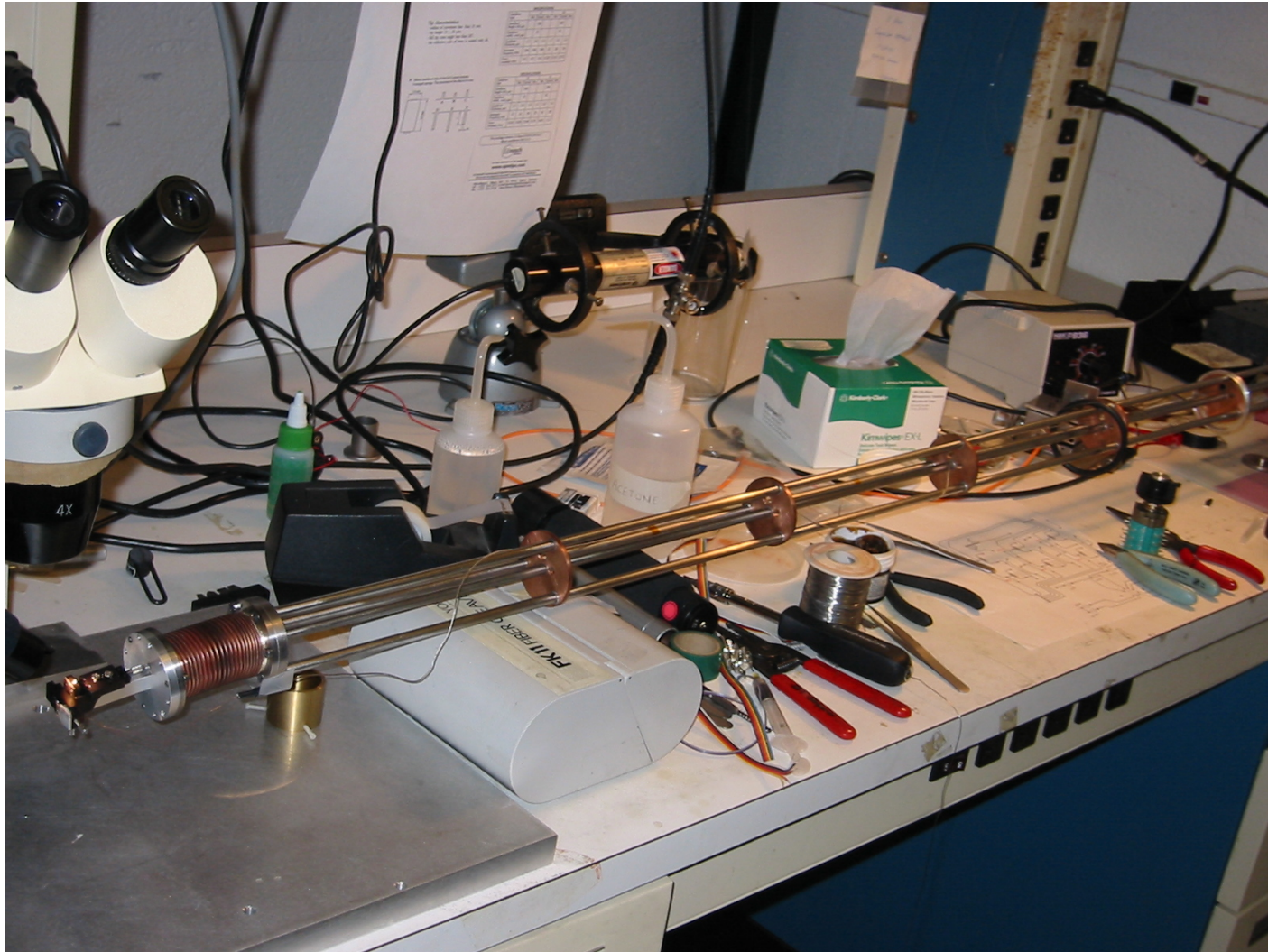
Hydrogen Depth Profile in Niobium



# Schematic of MFM/SQUID Microscope



# MFM/SQUID Microscope

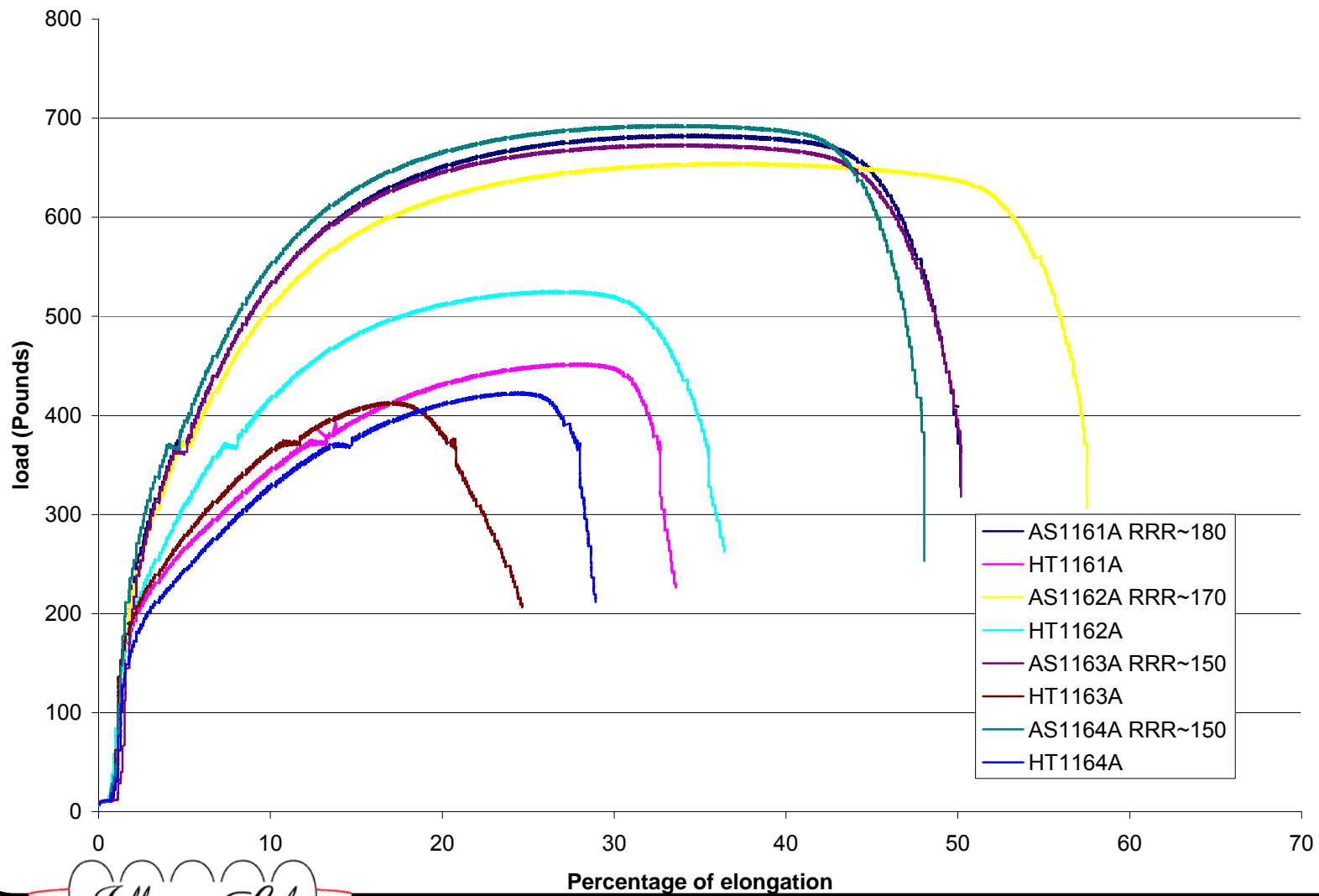


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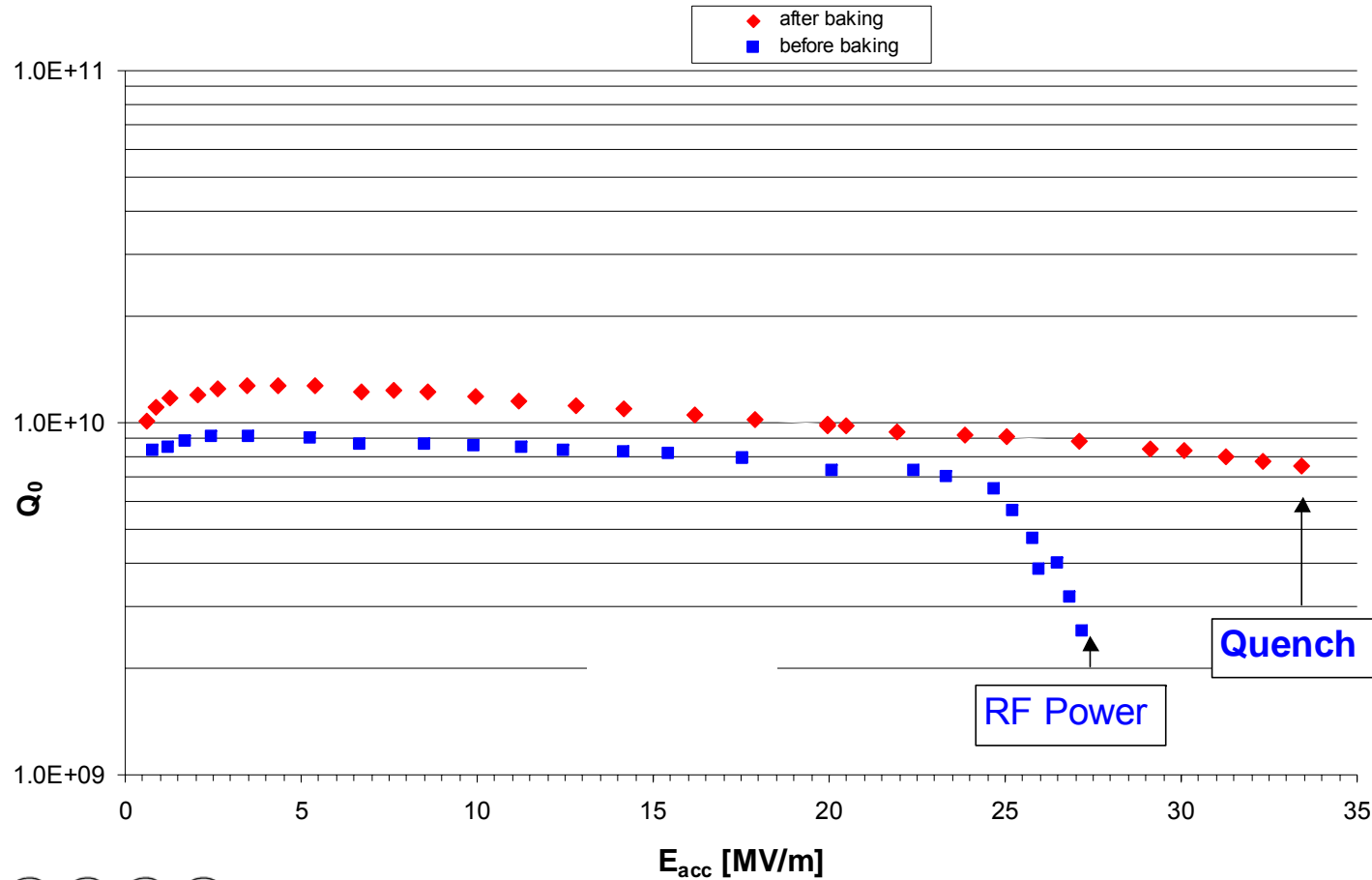
# NbTa (~160 ppm)

Cavity was heat treated with Ti at 1250 C for 3hrs, then 24 hrs at 1000C

CEBAF Single cell cavity Nb/Ta 1161\_32/1161\_33

P. Kneisel

$Q_0$  vs.  $E_{acc}$



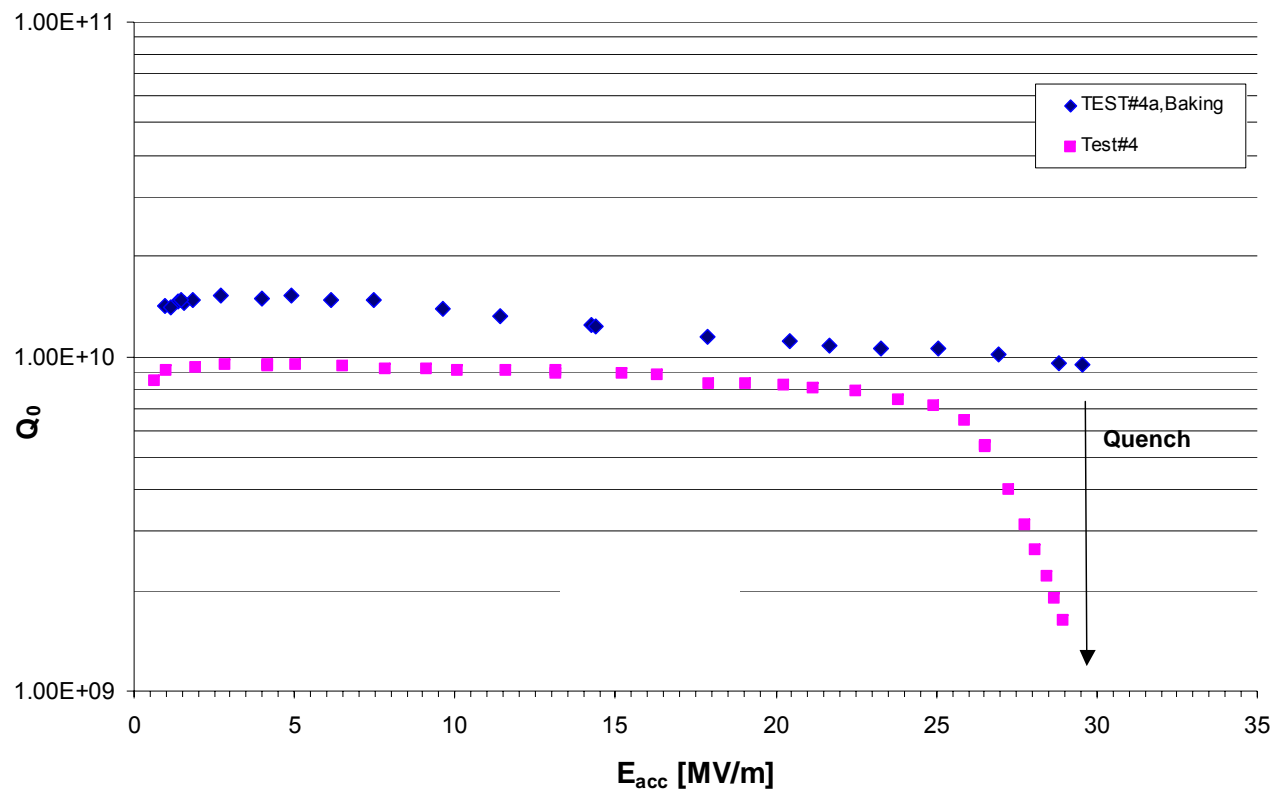
# NbTa (1300 ppm)

P.Kneisel

Cavity was heat treated with Ti at 1250 C for 3hrs, then 24 hrs at 1000C

CEBAF Single cell cavity Nb/Ta 1164\_12/1164\_12

$Q_0$  vs.  $E_{acc}$



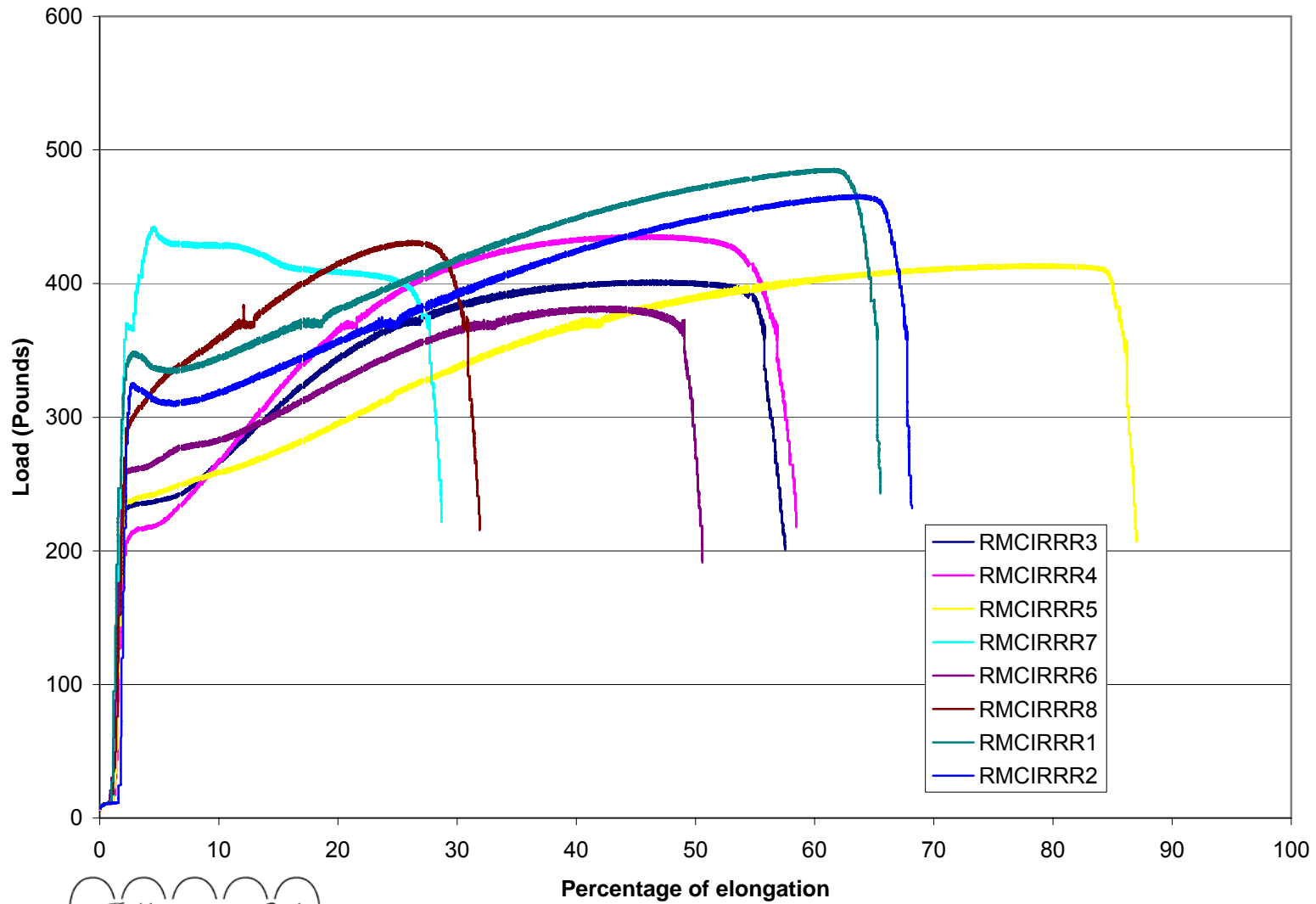
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### Reference Metals RRR Nb from Ingot





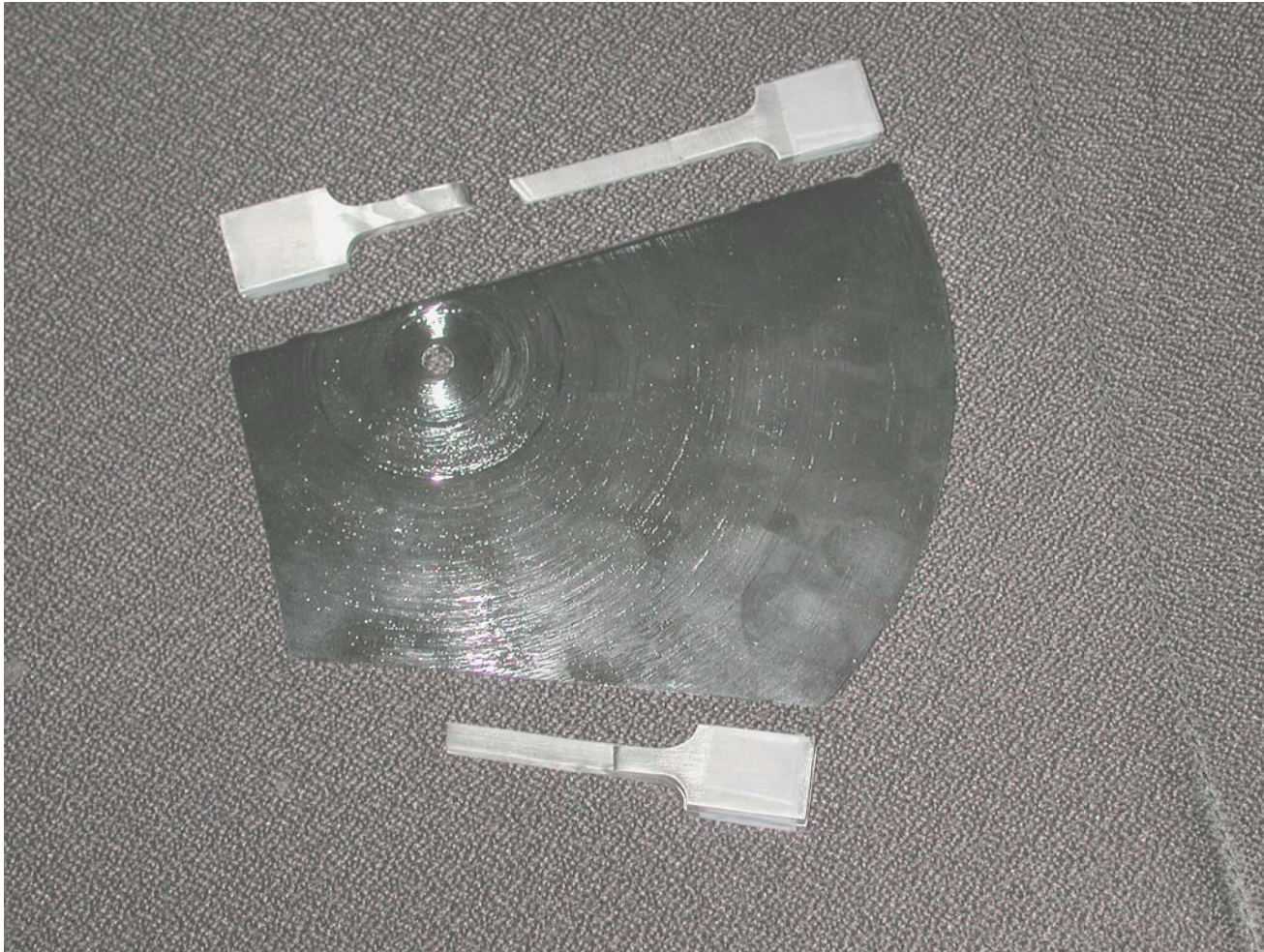


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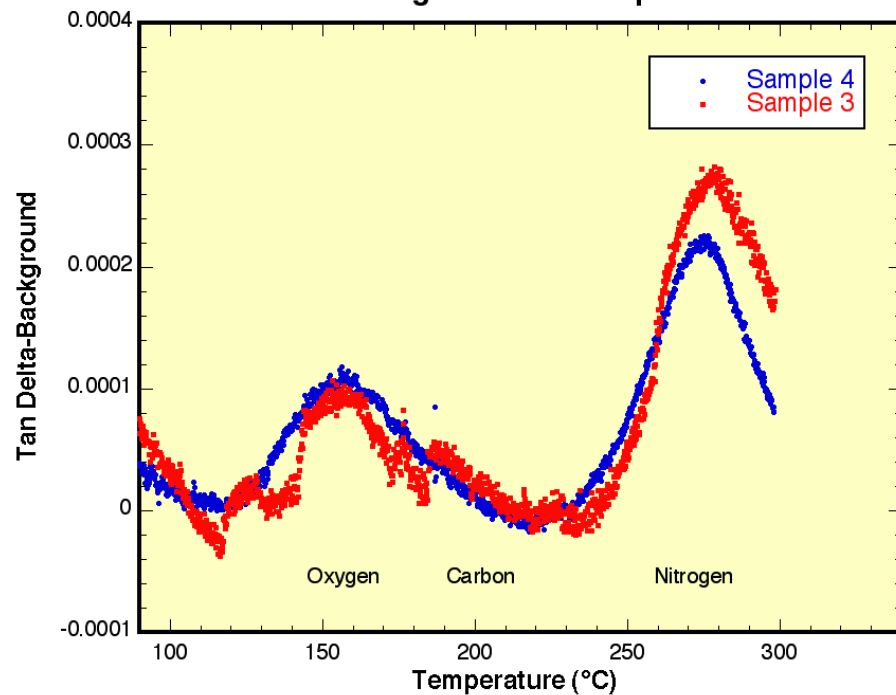
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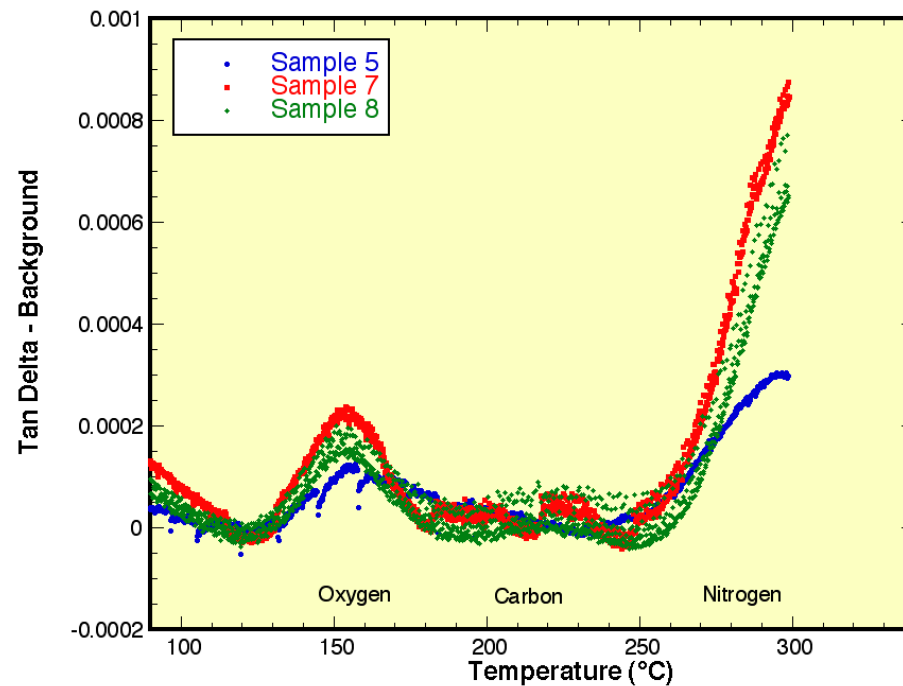
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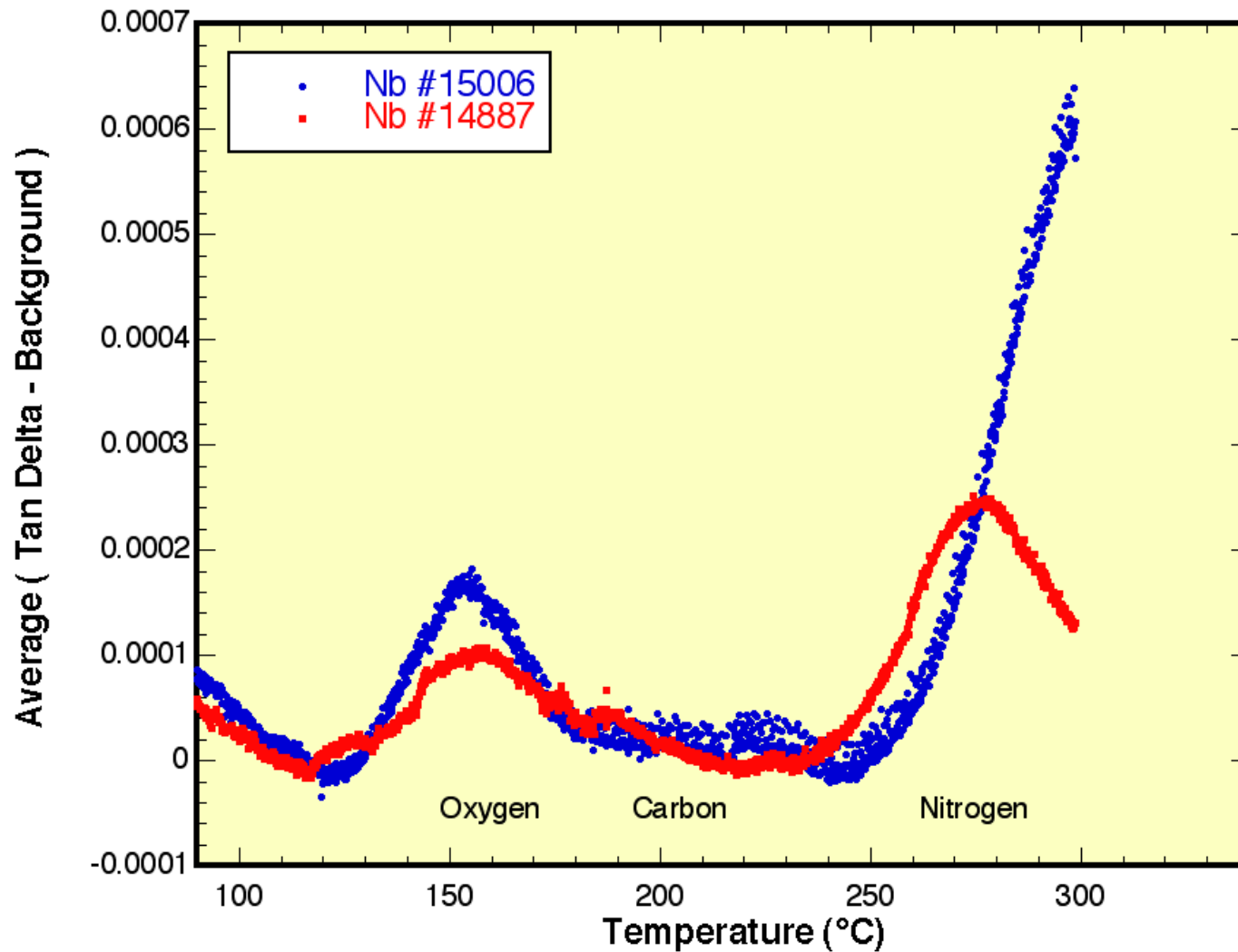
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Pure Nb  
Ingot 14887 03 Top



Pure Nb  
Ingot 15006 03





# Conclusions

- **The presently used high RRR niobium properties appear to be not consistent from batch to batch and/or even from sheet to sheet!!**
- **High tantalum content RRR niobium appears to meet all the needs of presently planned projects except ILC at much reduced costs!!!**
- **Single crystal niobium sheets sliced directly from the ingots can be expected to have consistent properties from batch to batch, less expensive and may provide high performance!!!!**

