Characterization of Ingot Materials for SRF Cavity Production

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Talk Overview

- Superconducting Property studies of LG-Nb samples

➢ **System Design.**

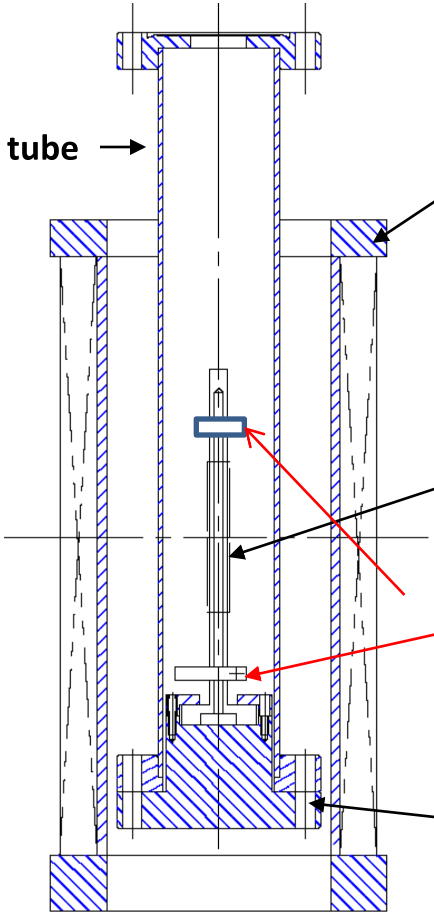
➢ **Thermal conductivity measurements.**

➢ **Magnetization Measurements.**

➢ **Penetration Depth & T_C Measurement.**

➢ **Summary and Conclusions.**

System Design



THIS INTEGRATED SYSTEM HAS BEEN DESIGNED TO MEASURE,

- DC MAGNETIZATION
- THERMAL CONDUCTIVITY
- PENETRATION DEPTH
- SURFACE PINNING CHARACTERISTICS

SUBSYSTEMS OF THE APARATUS:

SAMPLE: Φ 6mm, L=120mm, NIOBIUM ROD

HEATER: Made with constantan wire glued on a Cu block with Epoxy is clamped near the base and on the upper side of the sample rod.

TEMP. SENSOR: Two calibrated Cernox resistors are soldered with indium on two small Cu blocks which are clamped to the rod at a distance of about 40 mm.

PICK UP COIL: ~ 200 turns, 0.29 mm diameter Cu wire coil is inserted in the middle of the sample, between the two Cernox .

SC MAGNET: 1T (0.1% field homogeneity over the sample length - made by Cryomagnetics)

Samples Treatments

- Samples are cut by wire EDM and then machined by lathe to Φ6mm, L=120mm.
- After the machining the samples are degreased in ultrasonic bath with the micro solution for 30 min.
- Then BCP(1:1:1) was done to remove about 180 μm.
- After that the samples are again degreased for 30 min.
- Then the samples are degassed at 600^0C for 10 Hour at a vacuum $<10^{-6}$ Torr.
- After degassing all the samples were degreased and BCP(1:1:2) were done to remove about 24 μm.
- Degreased and baked at 100^0C, 120^0C, 140^0C at a vacuum better $<10^{-6}$ Torr for 12 Hour.

Thermal Conductivity Measurement

- The thermal conductivity as a function of the average temperature of the sample is calculated using Fourier's law,

$$K_S = \frac{P}{\Delta T}\frac{d}{A}$$

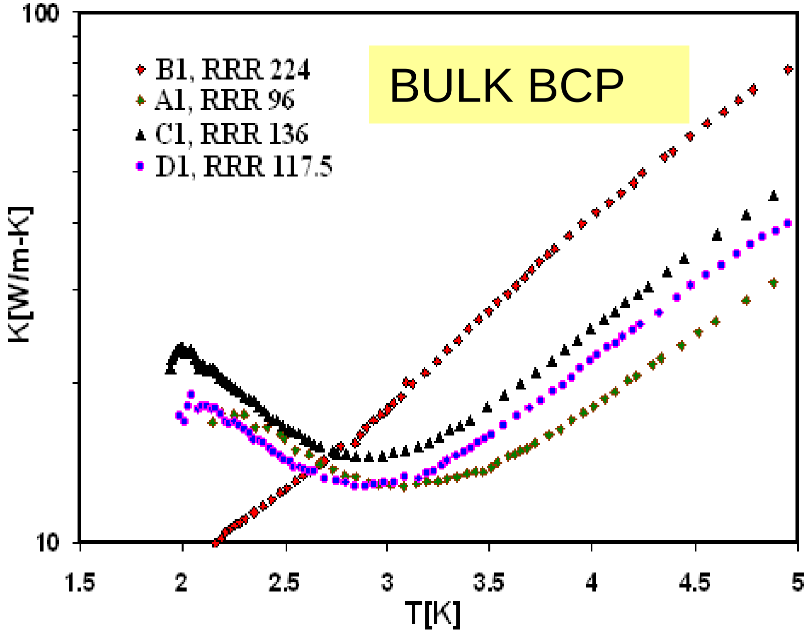
- Measurement error is ~ 6%.

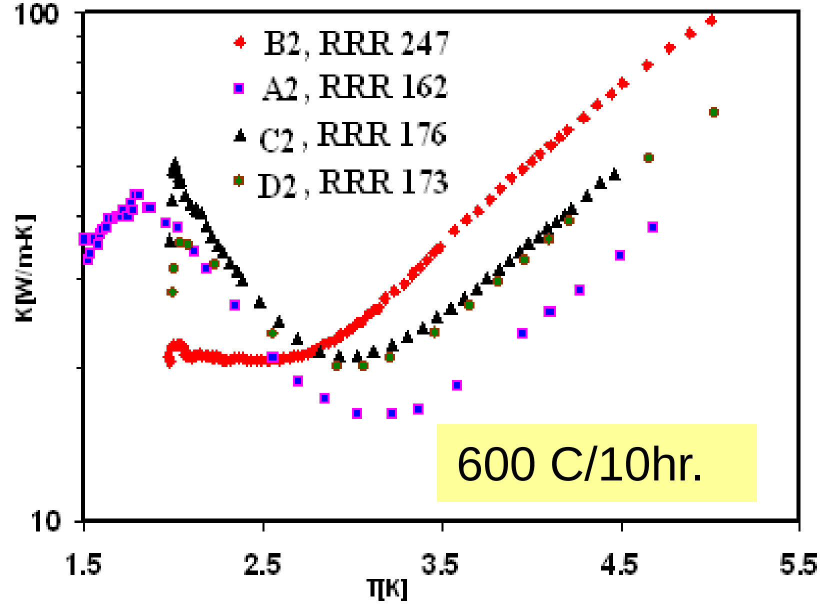
A. *Measurement of Thermal Conductivity and Evaluation of RRR*

- **The RRR of the sample is defined by RRR = α K_S (4.2K).**
- $\alpha = \frac{\rho_{295K}}{R(y)L_0 T}$ **is the experimentally measured parameter.**
- $\rho_{295K} = 1.44E-7\Omega-m$ **Resistivity at room temperature and L_0 Lorentz Constant.**

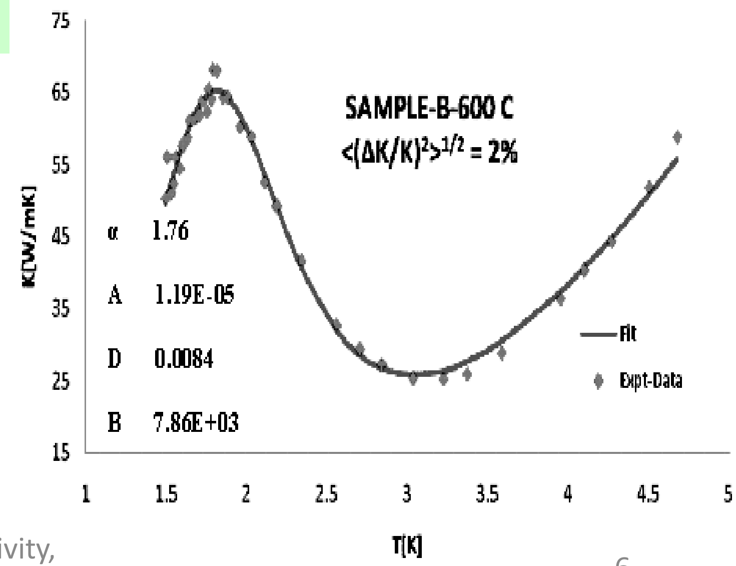
$$R(y) = \frac{k_{es}}{k_{en}}, where\, y = \Delta(T)/K_B T$$

- **Δ(T) BCS gap energy at temperature T.**





$$K_s(T) \cong R(y)\underbrace{\left[\frac{\rho_{295K}}{L \cdot RRR \cdot T} + a \cdot T^2\right]^{-1}}_{\text{Electronic term}} + \underbrace{\left[\frac{1}{\exp(y) D T^2} + \frac{1}{B l T^3}\right]^{-1}}_{\text{Lattice term}}$$

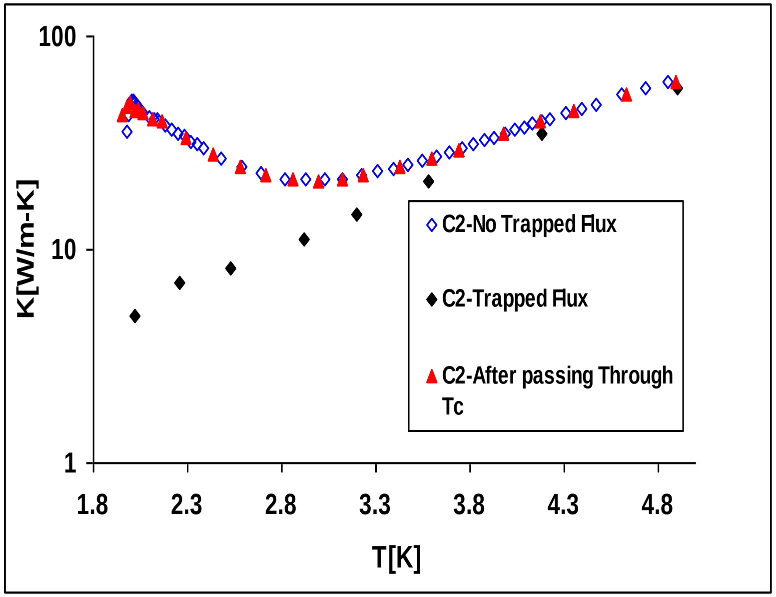


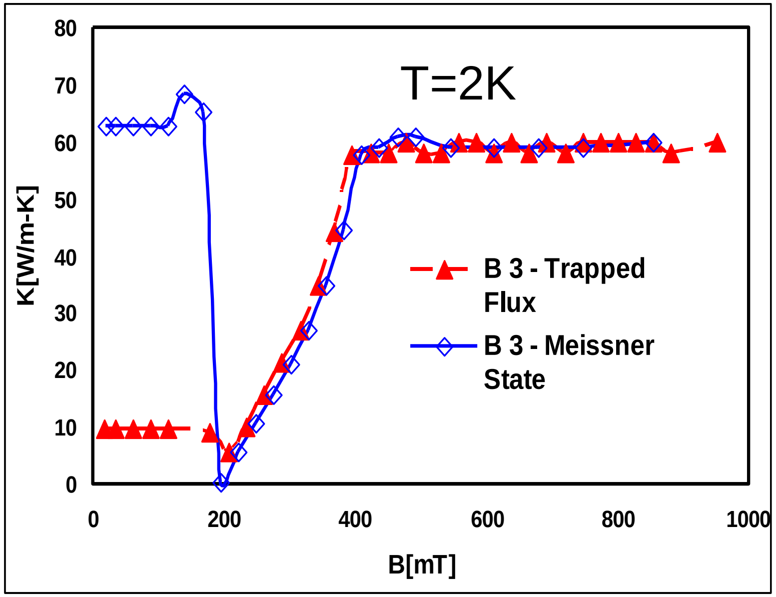
The values of the fitted parameters are in good agreement with theoretical estimates:

$a \approx 2.3\times10^{-5}$ m/(W- K) **$1/D \approx 300$ m/(W- K^3)**

$\alpha \approx 1.76$ **$B \approx 7\times10^3$ W/(m^2 K^4)**

B. Effect of Trapped Vortices on the Thermal Conductivity





- Before applying a magnetic field to the sample shows clearly a phonon peak.

- After cycling the applied magnetic field from zero to above H_{C2} and then back to zero, the thermal conductivity measurements do not show the phonon peak anymore.

- Sample temperature is raised above T_C and then lowered below T_C, with no magnetic field applied, then the phonon peak reappears in the thermal conductivity measurement.

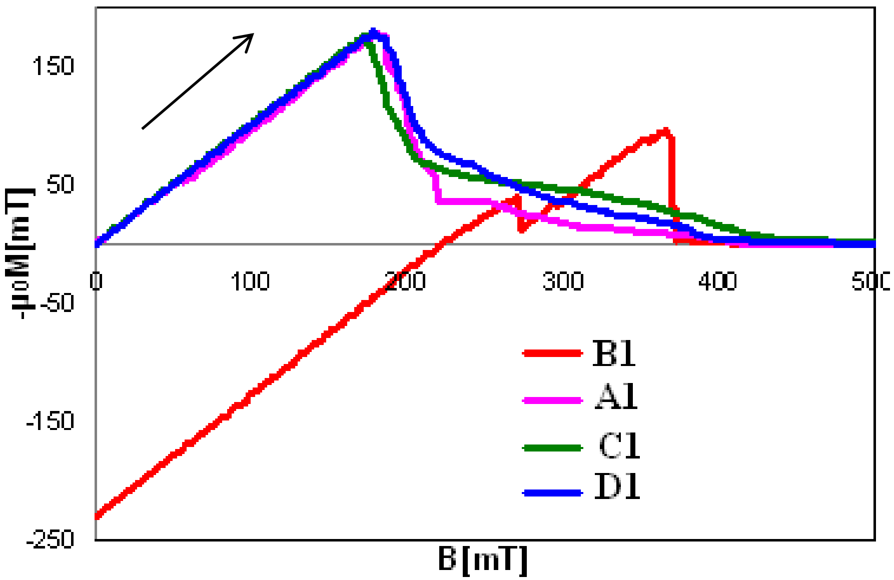
- Blue curve designated as Meissner state is a measurement before applying any magnetic field.

- Red one is with the remnant magnetization, after cycling the applied field above H_{C2}.

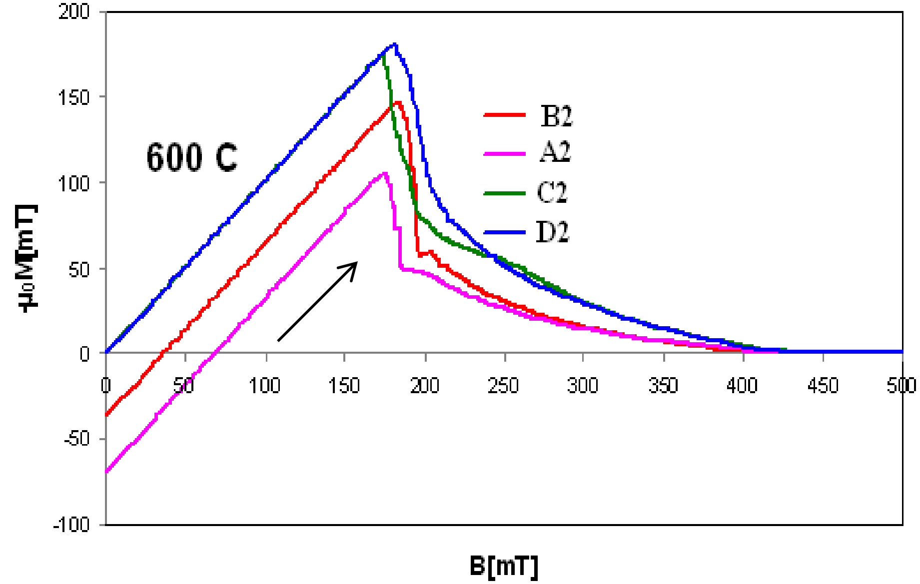
- Figure clearly shows that the thermal conductivity at 2 K is greatly reduced by the additional scattering of the phonons with the vortex cores.

Magnetization Measurement

A. Bulk Magnetization Measurement



Magnetization curves of BCP samples at 2K.



Magnetization curve of 600^{0}C heat treated samples at 2K.

DC magnetization measurement results for 1st and 2nd set of samples.

Sample	B_{C1}(2K) [mT]	B_{C2}(2K) [mT]	B_C(2K) [mT]
A	172	400	184
B	181	388	186
C	175	410	184
D	176	405	189

- H_{C1} and H_{C2} are not affected by the 600^{0}C heat treatment.

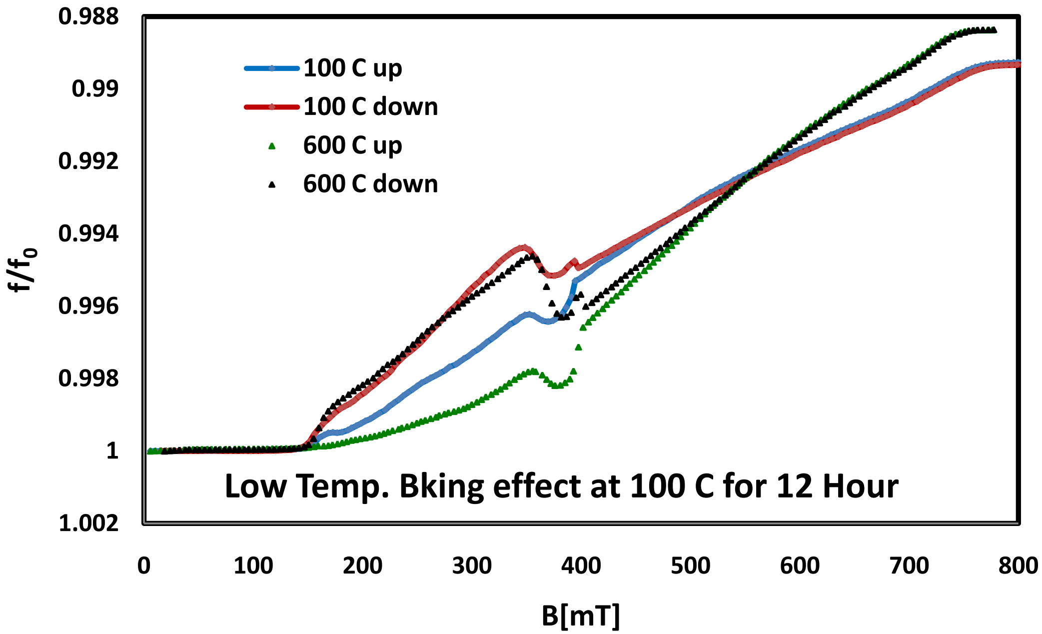
- B1 shows strong paramagnetic characteristics but after 600^{0}C heat treatment the paramagnetic behavior is reduced but still present.
- The paramagnetic behavior of A2 is due to the remnant magnetization from 4.2 K run

B. Surface Pinning Measurements

- By connecting the pick-up coil as part of a L-C oscillator, it is possible to measure the changes of the penetration depth as a function of the applied DC magnetic field by measuring the changes of the oscillator's resonant frequency f_0 (the base frequency is 270 kHz, sampling up to a depth $\sim$10 μm) while slowly ramping up and down the magnetic field.

- This method provides information about surface pinning and allows measurement of the surface critical field B_{c3}.

- The irreversibility of the curve between B_{c1} and B_{c2} is an indication of surface pinning.



1.The value of the surface H_{C1} and H_{C2} remains constant with different surface treatments like BCP 180 µm, 600 C 10 hr. heat treatment and 100 C, 120 C bake for 12 hr. in atmospheric pressure.
2. No change in H_{C3} for BCP 180 µm and 600 C 10 hr. heat treatment, but H_{C3} is higher for 100 C and subsequent baking temperature.
3. This irreversibility arises due to the pinning of fluxoids which are not in equilibrium with the external field.
4. The results show that after low temperature baking the irreversibility reduces by a significant fraction. This can be interpreted as the reduction of impurities near the surface.
5.Similar pinning measurements at higher frequency will allow reducing the sampling depth and it would have higher sensitivity to impurities within the penetration depth.

C. Effect of LTB on Hc3/Hc2 ratio

Theoretical Model

- **SHMIDT's Model**
- The Surface of the superconductor is contaminated by atoms of different types, dislocations and distortions which decreases the mfp.
- So the surface of the bulk superconductor can be represented as a film of thickness d with GL parameter k_1 and the bulk is represented by k_2.
- The basic assumption is that the the surface film and the bulk superconductor has the same T_c and H_c(Thermodynamic critical field)
- The model predicts that the ratio H_{C3}/H_{C2} is enhanced if $d \le \xi_0$

$$\frac{H_{C3}}{H_{C2}} = 1.67\left[1+\left(\frac{k_1-k_2}{k_1}\right)\sqrt{1.7}\,\frac{d}{\xi(T)}\right]$$

- **Correction of SHMIDT's Model for $T \ll T_C$**

•Hu and Korenman (HK) derived the H_{C3}/H_{C2} ratio in the temperature range $0 \le T \le T_C$ using the Gorkov's gap equation.

$$\frac{H_{C3}}{H_{C2}} = 1.69C(t)$$

$$C(t) = 1 + 0.614(1-t) - 0.577(1-t)^{3/2} - 0.007(1-t)^2 + 0.106(1-t)^{5/2}$$

•The temperature dependence of H_{C3}/H_{C2} has two independent sources. One which takes into account the nonlinearity of the microscopic theory and the other consider the effect of the surface film of thickness'd'.

$$R = \frac{H_{C3}}{H_{C2}} = 1.67C(t)\left[1+\left(\frac{k_1-k_2}{k_1}\right)\sqrt{1.7}\,\frac{d}{\xi(T)}\right]$$

•k_2 is calculated using the relation

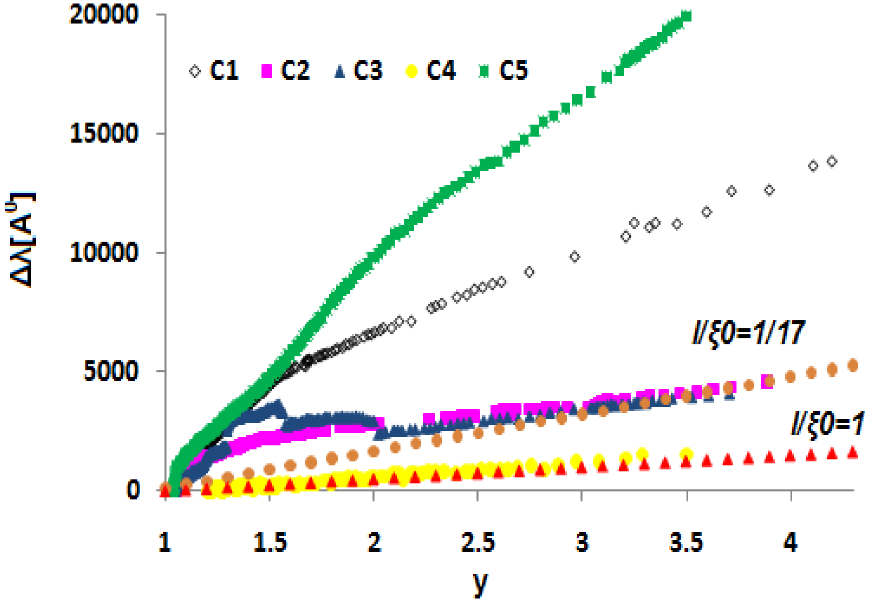
$$H_{C2} = \sqrt{2} k_2 H_c$$

•K_1 is calculated i.e. $H_{C2, surf}$ was Determined by $H_{C2, surf} = H_{C3}/1.67*C(t)$

•Results

- with the increased baking temperature the contaminated layer thickness increases to an average of 5.8 nm, 9.5 nm and 19.6 nm at 100 C, 120 C, and 140 C baking temperature respectively.
- at 140 C both the contaminated layer thickness and bulk H_{C2} increases which may be due to the partial dissociation of the Nb_2O_5 layer as explained in the oxygen diffusion model by G. Ciovati.
- The oxygen diffusion model corresponds to a diffusion depth of 7.6nm, 19nm and 40 nm for a baking at 100 C, 120 C and 140 C for 12 hour duration.

Sample	$R(B_{C3}/B_{C2})$	d_{expt} [nm]
BCP(1:1:1) 180 µm		
A1	2.0	5.61
B1	1.93	7.35
C1	2.0	5.87
D1	1.98	5.46
600 C 10 Hour Degassing at a vacuum < 10-6 Torr then BCP(1:1:2) removing about 24 µm		
A2	2.02	6.84
B2	2.01	8.45
C2	1.98	6.66
D2	2.01	9.44
Low temperature baking at 100 C in a vacuum <5x10-7 Torr for 12 Hour		
A3	2.14	12.5
B3	2.15	13.8
C3	2.15	11.6
D3	2.2	16.7
Low temperature baking at 120 C in a vacuum <5x10-7 Torr for 12 Hour		
A4	2.1	14.5
B4	2.12	18.7
C4	2.1	14.6
D4	2.17	21.4
Low temperature baking at 140 C in a vacuum <5x10-7 Torr for 12 Hour		
A5	2.76	30.1
B5	2.60	28.5
C5	2.60	25.3
D5	2.55	25.8



PENETRATION DEPTH & T_C MEASUREMENT

- ➢ Penetration depth measurement as a function of temperature with different surface treatments provides qualitative information about the changes in the surface.
- ➢ The slope of $\Delta\lambda(y)$ represents the penetration depth at 0 K in the two-fluid model.

• Observations

- ➢ **As the LTB temperature is increased the slope of decreases till 120^0C. At 140^0C baking the slope increases at a faster rate resulting in a dirtier surface with decreased mean free path (mfp).**
- ➢ **In 100 and 120 C the slope continuously decreases meaning that diffusion of surface impurities toward the bulk and dilution of impurity concentration.**
- ➢ **The faster increase of the slope at 140 C might be due to the partial Nb_2O_5 layer dissociation and thereby increasing the impurity concentration at the surface resulting in a lower mfp.**
- ➢ **The surface magnetization measurement further corroborates this fact as the bulk H_{C2} starts increasing at 140 C baking.**

CONCLUSIONS

- Thermal conductivity measurements show a phonon peak at about 1.9 K, except for the sample of highest purity (sample B).

- One important observation is that the phonon peak is eliminated by the presence of trapped magnetic flux, mainly due to the interaction between the phonons and the bound excitations in the vortices' core.

- The bulk properties of the samples such as T_C, B_C, H_{C1} and H_{C2} were essentially unchanged with different surface treatments and independent of RRR.

- Surface pinning measurement shows that the H_{C3} value increases with the increased LTB temperature. It also shows that the surface H_{C1} is lower than the bulk H_{C1} and that the highest surface Hc1 was obtained after baking at 120°C for 12h.

- The irreversibility between Hc1 and Hc2 decreases significantly with the low temperature baking, thus a reduction in the surface pinning centers can be inferred.

- The ratio of H_{C3}/H_{C2} increases with the increased LTB temperature. The enhanced H_{C3}/H_{C2} ratio has been interpreted as the increased contaminated surface layer thickness. This fact is further corroborated by the penetration depth measurement.

Thank You for Your Kind Attention