3He Polarization & Transport

Steven Williamson

- Overview of the 3He Services for nEDM@SNS
- Challenges for 3He Services
- R&D Example: Superfluid helium film control
Polarized $^3\text{He}$ in nEDM@SNS

- Polarized $^3\text{He}$ provides:
  - Comagnetometer measure of B field seen by neutrons
  - Mechanism for detecting neutron spin precession

- The experiment relies on efficient…
  - injection of polarized $^3\text{He}$ into isotopically purified $^4\text{He}$
  - transport of the polarized $^3\text{He}$ to the measurement volumes
  - maintenance of polarization during the measurement
  - removal of unpolarized $^3\text{He}$ when a precession measurement is complete.

### Table of Operations

<table>
<thead>
<tr>
<th>Time</th>
<th>Neutrons</th>
<th>$^3\text{He}$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Measurement complete</td>
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<tr>
<td>200 s</td>
<td>Remove from cell to holding</td>
<td>Remove from cell to holding</td>
</tr>
<tr>
<td></td>
<td>volume 2</td>
<td>volume 2</td>
</tr>
<tr>
<td>100 s</td>
<td>Move to cell</td>
<td>Move to cell</td>
</tr>
<tr>
<td>700 s</td>
<td>Accumulate in cell</td>
<td>Move to dump</td>
</tr>
<tr>
<td>700 s</td>
<td>Measure</td>
<td>Inject $^3\text{He}$ into He II</td>
</tr>
<tr>
<td></td>
<td>Move to holding volume 1</td>
<td>Move to holding volume 1</td>
</tr>
</tbody>
</table>
3He Services

Injection and Measurement components must be "3He Friendly" contains polarized 3He

Injection

Measurement & common

Purification

Cold Pure LHe Storage

IV2 Valve

Volume Displace/Pressurizer

IV1 Heater

Injection Volume

Intermediate Volume 1

IV2 Heater

IV1 Pressurizer

Intermediate Volume 2

Purifier "T" Valve

Injection "T" Valve

Cold Storage Valve

SV Pressurizer

Dump Valve

Dump

To Dump Vacuum Pump

Pure 4He from Internal McClintock Purifier

Cell Isolation Valves

Measurement Cell A

Measurement Cell B

Cell "T" Valve
• Step 1: Inject Polarized $^3$He from ABS
  • Existing Equipment
  • $2 \times 10^{14}$ atoms per sec
  • 99.6% Polarization
  • 30 s
• **Step 1:** Inject Polarized $^3$He from ABS
• **Step 2:** $^3$He Diffuses to IV1
  - Low (~zero) pressure
  - Low temperature (< 0.35 K)
  - 2 sec
• **Step 3:** Pressurize IV1
  - Raise pressure to 1 atm.
  - $\Delta V/V = 1.19\%$, $\Delta V = 19$ cc
• **Step 4:** Heat flush to Cells
  - 5 mW supplied at IV1
  - 30 s
Purification

- Step 1: Flush from cells to IV2
  - 5 mW extracted at IV2
  - 350 s

- Step 2: Heat flush from IV2 to SV
  - 8 mW extracted at SV
  - 150 s
Purification

- **Step 1:** Flush from cells to IV2
- **Step 2:** Heat flush from IV2 to SV
- **Step 3:** Dump the SV
  - Depressurize the SV first
  - Evaporate and pump away depolarized $^3$He
  - 85 s
- **Step 4:** Replace $^4$He
  - Pure $^4$He from internal McClintock Purifier
  - Pure storage provides reservoir
  - Re-pressurize SV after filling
  - 60 s

Diagram:
- **To Dump Vacuum Pump**
- **Pure $^4$He from Internal McClintock Purifier**
- **Cold Pure LHe Storage**
- **SV Pressurizer**
- **Dump Valve**
- **Cold Storage Valve**
- **IV2 Heater**
- **Volume Displace/Pressurizer**
- **Intermediate Volume 2**
- **Sequestration Volume**
- **4 He from Internal McClintock Purifier**
- **Pure Storage Provides Reservoir**
- **Re-pressurize SV after filling**
- **60 s**

ORNL nEDM Experimental Techniques Workshop, October 10-12, 2012
Challenges for $^3$He Services

- **Heat Budget**
  - Thermal isolation (e.g. of valves)
  - Adjustable thermal links

- **Valves**
  - Superfluid tight
  - Large diameter
  - $^3$He friendly
  - etc.

- **Depolarization**
  - Wall depolarization
  - Gradient Depolarization

- **$^3$He Transport**
  - Diffusion at $T \leq 300$ mK
  - Heat flush for $T \geq 400$ mK

- **Thermal contraction of long plumbing runs**
- **Many joints and flange-seals**
- **Film control in the injection beam line**
Challenges for $^3$He Services

- Thermal transport and heat budget
- $^3$He transport
- Film Control in injection beam line
- Valves
- Wall depolarization
- Gradient Depolarization

... Not unrelated
Challenges for $^3$He Services

- Heat Budget
  - Thermal isolation (e.g. of valves)
  - Adjustable thermal links
- Valves
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  - etc.
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  - Gradient Depolarization
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  - Heat flush for $T \geq 400$ mK
- Thermal contraction of long plumbing runs
- Many joints and flange-seals
- Film control in the injection beam line
4He Film Control

- A 4He film will be generated on the walls of the beam line immediately above the bulk LHe.
- Evaporation of film into beam line must be prevented.
  - Produces large heat load to injection volume (HeVAC)
  - 4He gas can block the beam (3He-4He atomic scattering)

- Our approach to film control: a “Film Burner”
  - Evaporate the film with a heater. Then re-condense it where cooling is efficient and gas conductance to beam line is low.

- Film burner advantages:
  - Proven technique
  - Robust and reliable (once it works).
  - No contamination (compared to Cs ring).
Film Control

- Film burner challenges:
  - Efficient cooling is required to remove the heat supplied by the evaporator.
  - The conductance of vapor from evaporated film to the beam line must be small.
  - Operation depends on the details of the heat load, the condenser cooling, and the geometry of the evaporator/condenser.

- Difficult to model ⇒ a test is needed to prove a given design.
Film Burner Design

Heater Power Estimate

\[ v_f \cdot t_f = 1.2 \times 10^{-4} \text{ cm}^2 / \text{s} \]

\[ t_f = \text{film thickness (depends on surface)} \]
\[ \approx 30 \cdot h^{1/3} \text{ nm (with h in cm)} \text{ (10-12 nm)} \]
\[ v_f = \text{film velocity (of order 50 cm/s)} \]

\[ P = v_f \cdot t_f \cdot 2\pi R_{\text{min}} \cdot L \approx 4.5 \text{ mW} \]

\[ L = \text{heat of vaporization} = 3.02 \text{ J/cm}^3 \]
\[ R_{\text{min}} = \text{minimum (upward) radius} = 2.0 \text{ cm} \]

1. Typical temperature required to vaporize film is 0.7 to 1 K
2. To insure full condensation, condenser surface should be at < 0.35 K
3. Cooling supplied to outer jacket of condenser by DR Mixer via LHe thermal link
4. To overcome Kapitza resistance, sinter is needed on both inner and outer surface of condenser.
5. Limiting radius defined at top of film-burner where beam is smallest.
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\[ v_f \cdot t_f = 1.2 \times 10^{-4} \text{ cm}^2 / s \]

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- To insure full condensation, condenser surface should be at < 0.35 K
- Cooling supplied to outer jacket of condenser by DR Mixer via LHe thermal link
- To overcome Kapitza resistance, sinter is needed on both inner and outer surface of condenser.
- Limiting radius defined at top of film-burner where beam is smallest.
- Diameter is determined by the projected size of the collimated beam
A “Simple” Test of the Film-burner

- Initial test plan (a.k.a. Phase 1):
  - Using DR in Ike Silvera’s Harvard lab (~8 mW @300 mK)
  - Use existing (but untested) Duke film-burner
    - Cooled with copper thermal links
    - $R_{\text{min}} = 1.33 \text{ cm (2/3 of real FB)}$
    - $P = 3 \text{ mW}$
  - Success: elimination of film above film-burner.
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- Progress so far…
  - Commissioning of the DR 1/12 to 3/12
  - Test cool-downs, 6/12 and 8/12
  - Successful test run, 9/12
Helium Film Sensor

- Use a surface-mount RuO₂ resistor as uncalibrated temperature sensor.
  - 10 kΩ at room temperature
  - > 100 kΩ below 50 mK
  - < 1 cent each (we bought 6000!)
- Suspend from fine wires to thermally isolate sensor.
- Two film sensors:
  - On inj. vol. – always covered with film (when LHe is present)
  - On top flange assembly – should not be covered if film burner is working.

A typical ROX temperature response (NOT the one pictured at left).
Detecting Superfluid Film

- **Procedure for detecting film**
  - Abruptly initiate constant current through the ROX
  - Digitize amplified voltage across ROX vs time.
  - **No film:**
    - If “self-heating” is small, ROX voltage is constant
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![Diagram of experimental setup](image)

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**Graphs:**

- **Excitation Voltage (V):**
  - Time (ms) vs Voltage (V)

- **ROX Temperature (mK):**
  - Time (ms) vs Temperature (mK)

- **ROX Voltage (V):**
  - Time (ms) vs Voltage (V)

---

ROX remains cold until film evaporates.
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  - With film: ROX remains cold until film evaporates.
  - If a small offset is applied, the “re-filling” of the film can be observed.

**Diagram:**
- Constant voltage source
- Current Limit Resistor (R0 >> R)
- (Optional) SRS 560 Pre-amplifier
- ROX Sensor
- ROX cools rapidly when film is present.
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![Diagram of ROX setup with current limit resistor, voltage sensor, and pre-amplifier.](image)

**FB Heater Power = 5 mW**

**Upper Film Sensor**

![Graph showing pulse height (V) vs pulse time (sec)](image)
Detecting Superfluid Film

- Procedure for detecting film
  - Abruptly initiate constant current through the ROX
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  - No film:
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![Diagram showing the setup for detecting superfluid film](image)
The “Phase 1” Film burner works.

...or does it?

- We can stop the film, but require ~70% more heater power than expected.
  - Surface quality can affect film thickness.
  - Radial “knife edge” may not be smooth
- We don’t know that all vapor is recondensed on condensor surface.
  - Test by measuring density of vapor within tube by transmission of $^4$He atoms
  - Generate a pulsed “beam” of atomic $^4$He
    - Method pioneered* in Ike Silvera’s lab
    - Apply a short heat pulse to small surface (Bolometer) coated with $^4$He film to evaporate the film
    - Velocities in vapor pulse follow M-B distribution of 0.65-0.8 K
    - Pulse durations from 30-60 µs
- Pass beam through residual vapor in the “beam line”
  - Flight path ≥ 4 inches
- Detect unscattered beam
  - Measure temperature rise of a neutron transmutation doped (NTD) germanium bolometer chip
  - Measure versus time to get beam velocity dependence
  - Response time is ~40 µs

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Beyond Phase 1: Phase 2

- Realistic dimensions: $R_{\text{min}}$ 50% larger
- Realistic materials: plastic injection volume
- Realistic cooling:
  - LHe thermal link to MC heat exchanger
  - Cooling jacket and 2-sided sinter on condenser
- Change geometry to reduce heat load:
  - Etched Silicon “film thinner”
  - A Series of VERY sharp edges: film thickness reduced due to large surface tension energy at small radius.
  - Edge must be “atomically sharp” (< 10 nm radius).
  - Currently under study at NCSU

Etched 4” wafer at NCSU (D. Haase)  
SEM Image of etched wafer (D. Haase)


Backup Slides
Beam Transmission

\[ \frac{I_T}{I_0} = \exp \left[ -\frac{L}{\lambda_v(T_0)} \right] = \text{Transmission} \]

- \( I_0 \) = Unattenuated intensity
- \( I_T \) = Intensity attenuated by gas at \( T_0 \)
- \( L \) = Length of scattering region

\[ \sigma_{\text{eff}} = \frac{1}{n \cdot \lambda_v(T_0)} \]

- \( n \) = Target gas density
- \( \lambda_v(T_0) \) = Mean free path of atom with velocity \( v \)
- \( T_0 \) = Target Temperature

- Most scattering occurs at the low-temperature end of the beam line.
  - Cross section is highest for low target temperature.
  - Density of gas in the beam line goes as \( 1/T \) (to first order) so is highest at low temperature
- A realistic simulation of the scattering is underway.

Tests and Performance of $^3$He-$^4$He Scattering
Eric Stefan Meyer, PhD
Dissertation, Harvard University, 1993

1K Source (simulated)
Tests and Performance of $^3$He Atomic Beam Polarizer, Steve Lamoreaux, December 2, 2004
Time-of-flight Resolution

- Want pulse duration $\approx 0.1 \times$ shortest flight time for 10% speed resolution
  - Film evaporates in 30-60 $\mu$s
  - Bolometer response is about 40 $\mu$s
  - Speed resolution is about 60 $\mu$s
  - Lowest flight time should be $\sim 600$ $\mu$s

- Conclusion: 4” flight path seems OK (lower temp. evaporator and longer flight path would be better)
Phase 1 with NTD Ge Cold Finger
Detecting Superfluid Film

Top ROX, with LHe, 90 mK, 45 kOhm, 4 V pulses

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Detecting Superfluid Film

Top ROX, with LHe, 90 mK, 45 kOHm, 4 V pulses
Detecting Superfluid Film

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Top ROX, with LHe, 90 mK, 45 kOhm, 4 V pulses
Detecting Superfluid Film
Detecting Superfluid Film

Top ROX, with LHe, 90 mK, 45 kOHm, 4 V pulses
## Heat Budget

<table>
<thead>
<tr>
<th>Subsystem†</th>
<th>Source</th>
<th>Amount (mW)</th>
<th>Basis of Estimate</th>
</tr>
</thead>
<tbody>
<tr>
<td>General</td>
<td>Radiative heating to insulation volume and $^3\text{He}/^4\text{He}$ volumes</td>
<td>0.2</td>
<td>Calculation, emissivity = 1</td>
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<tr>
<td></td>
<td>Mechanical support to 1.5 K</td>
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<td>Calculation x 5</td>
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<td>Conduction through Electrical Leads</td>
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<td>Calculation x 5</td>
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<td>$^3\text{He}$ Heat Flush</td>
<td>Heater currents</td>
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<td>Flow calculations from 3He services group</td>
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<tr>
<td>Purifier</td>
<td>Film Burner</td>
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<td>Calculation x 2</td>
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<tr>
<td></td>
<td>Valves</td>
<td>1.5</td>
<td>2 @ 0.75 mW</td>
</tr>
<tr>
<td></td>
<td>Bellows</td>
<td>1.5</td>
<td>1 @ 1.5 mW</td>
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<tr>
<td>Injector</td>
<td>Radiation through injection pipe</td>
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<td>Calculation x 10</td>
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<tr>
<td></td>
<td>Film burner</td>
<td>6</td>
<td>Calculation x 2</td>
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<tr>
<td>Light Guides and PMTs</td>
<td>Radiative heating from 8 K</td>
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<td>Calculation (includes 15 K PMT’s) x 2</td>
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<tr>
<td>Valves</td>
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<td>10</td>
<td>10 valves @ 1.0 mW</td>
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<tr>
<td>Bellows</td>
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<td>2 bellows @ 3 mW</td>
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<td>Magnets</td>
<td>I$^2$R Losses, eddy currents</td>
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<td>Contingency</td>
<td>Project management</td>
<td>20</td>
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<tr>
<td><strong>Total Heat Load to DR at 0.35 - 0.40 K</strong></td>
<td></td>
<td><strong>88.2</strong></td>
<td></td>
</tr>
</tbody>
</table>

- Heat flush transport is one of the largest sources of heat load.
- A thermal model‡ of the apparatus and measurement cycle indicates:
  - Closed valves should have low thermal conductance (< 1 mW/K)
  - Adjustable thermal links (ATLs) will reduce the overall heat load

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† D. Haase, Heat Budgets and Other Items, a presentation at the June 17-19, 2009 nEDM Collaboration.
‡ D. Kendellen and D. Haase, An Updated Thermal Model for Cooling the nEDM $^3\text{He}$ Services, a Technical Feasibility, February 23, 2010.
Adjustable Thermal Links (and Valves)

- Bolted joints can have poor thermal conductance (unless care is taken)
- Helium is still a pretty good thermal conductor at 0.45 K
  - Kapitza resistance is not so bad at this temperature
  - Offers the possibility of adjustability (adjustable thermal links)

- ATLs are useful for:
  - Reducing the heat load on DR when components are warmed during heat flush…
  - While allowing rapid re-cooling when the heat flush is over.
ATL Principle of Operation

- Heat transport is via phonons for T<0.6 K
- If mean free path of phonons, $\lambda_{ph}$, is geometrically restricted, the effective conductivity is reduced.
  - Need typical dimension $<< \lambda_{ph}$
  - $\lambda_{ph} \approx 0.2$ cm at 0.45K

- Create restriction with adjustable-length annular gap
  - Restriction (w) must be $\sim$ a few mils ($\sim$50X smaller than $\lambda_{ph}$)
  - Wall and plug material must have low conductivity compared to LHe
Implementation in the CAD Model

In the Injection Module

To actuator

Multiple bellows (~2” motion)

In the Purifier Module

IV2 ATL

SV/evap. purifier ATL

IV1 ATL

Plug

To DR mixer heat exchanger

Q
Thermal Conductance of an Annular Gap

- Proof of principle: geometrically restrict phonon mean free path to reduce thermal conductance of LHe for $T < 0.6K$
- Measure for:
  - a single length (1”) -- no actuation
  - a single gap width (0.001”)
  - a range of temperatures ($\sim 0.4 – 0.6$ K)
- Design as simple modification to wall-depolarization apparatus
- Predict behavior following Casimir, Physica (Utrecht) 5 (1938) 495.
  - Similar to Greywall, PRB 23 (1981) 2152: for a cylinder (capillary tube)
Annulus Thermal Isolation Test

1" Plug (note bottom pin)

Kapton strips to set gap width

Nichrome heater at bottom of cell

Stainless He fill line

Anchor to $^3$He pot

Test cell

Heater and "warm" temp. sensor

"Cold" temp. sensors
Annulus Thermal Conductance

- Good agreement with prediction (dotted curves)
  - Small discrepancy may be due to sensor calibration error
- Senior thesis project of Andrew (Ian) Chen (now at MIT)
3He Wall Depolarization

- The total longitudinal relaxation time, $T_1$

$$\frac{1}{T_1} = \frac{1}{T_D} + \frac{1}{T_B} + \frac{1}{T_S}$$

- $T_D$ = dipolar interaction between 3He atoms -- not an issue at nEDM concentration
- $T_B$ = B-field gradient effect -- can be controlled with careful design
- $T_S$ = Surface (wall) effect

- For measurement cell (0.45 K, dTPB in dPS), $T_S > 1000$ s ("R&D" goal)

- Two separate projects were launched to make the measurements at Duke/NCSU and UIUC:
  - Critical to SNS nEDM method
  - Difficult measurement
  - Involved PhD students at early stage in experiment
    - Qiang (Alan) Ye, Duke
    - Jacob Yoder, UIUC
Measuring $^3$He Wall Depolarization

- The Method:
  - Cool test cell
    - UIUC: with $^3$He evaporation fridge.
    - Duke/NCSU: with DR
  - Fill test cell with LHe to some level
  - Polarize $^3$He at room temperature
    - UIUC: Metastability Exchange Optical Pumping
    - Duke/NCSU: Rubidium Spin-exchange Optical Pumping
  - Open isolation valve
  - $^3$He to diffuses (is drawn) into LHe
  - Measure polarization vs. time with NMR
  - Repeat for other levels of LHe, concentrations of $^3$He and Temperatures
- Extrapolate to geometry of nEDM
Measuring $^3$He Wall Depolarization

- **The Method:**
  - Cool test cell
    - UIUC: with $^3$He evaporation fridge.
    - Duke/NCSU: with DR
  - Fill test cell with LHe to some level
  - Polarize $^3$He at room temperature
    - UIUC: Metastability Exchange Optical Pumping
    - Duke/NCSU: Rubidium Spin-exchange Optical Pumping
  - Open isolation valve
  - $^3$He to diffuses (is drawn) into LHe
  - Measure polarization vs. time with NMR
  - Repeat for other levels of LHe, concentrations of $^3$He and Temperatures

- **Extrapolate to geometry of nEDM**
Extrapolation to nEDM Geometry

1. If $P_d$ is the probability per wall bounce of depolarization, can write:

$$N_\uparrow(t + \Delta t) = N_\uparrow(t) - P_d \cdot \varphi \cdot S \cdot \Delta t$$

$$\varphi = \frac{1}{4} \cdot \rho_\uparrow(t) \cdot \langle v \rangle = \text{flux of polarized atoms hitting unit area per unit time}$$

$$\rho_\uparrow(t) = \frac{N_\uparrow(t)}{V} = \text{density of polarized atoms}$$

$$\frac{dN_\uparrow}{dt} = -P_d \cdot \frac{1}{4} \cdot \langle v \rangle \cdot \frac{S}{V} \cdot N_\uparrow(t)$$

$$N_\uparrow(t) = \exp\left(-\frac{t}{T_s}\right)$$

$$\frac{1}{T_s} = P_d \cdot \frac{1}{4} \cdot \langle v \rangle \cdot \frac{S}{V}$$

2. Knowing $P_d$, can extrapolate from $S/V$ of test cell to larger nEDM measurement cell.
$^3$He Relaxation Rate Data

J. Yoder Thesis

$T_s = 2700 \pm 140 \text{ s}, \frac{S}{V} = 2.96 \text{ cm}^{-1}$
$P_d < 1.32 \times 10^{-7}$
$T_s^{nEDM} > 16000 \text{ s (at 0.45 K)}$


$T_s = 6100 \text{ s}, \frac{S}{V} = 2.1 \text{ cm}^{-1}$
$P_d = 1.0 \times 10^{-7}$
$T_s^{nEDM} = 21,000 \text{ s (scaled to 0.45 K)}$

$T_s > 20,000 \text{ s}$
Other Materials

- Improved apparatus to quickly measure $T_s$ on a variety of materials.
  - Use “detachable cell” with low $P_d$
  - Introduce sample
  - This geometry permits measurements with conductive samples (e.g. Be-Cu)
- Analysis: subtraction method
  - Measure empty cell to find $T_s^{dTPB-dPS}$
  - Measure with sample to find $T_s^{Total}$

$$\frac{1}{T_s^{Sample}} = \frac{1}{T_s^{Total}} - \frac{1}{T_s^{dTPB-dPS}}$$

<table>
<thead>
<tr>
<th>Material</th>
<th>$P_d$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acrylic coated with dTPB-dPS</td>
<td>$1.0 \times 10^{-7}$</td>
</tr>
<tr>
<td>Bare Torlon 4203</td>
<td>$(1.01 \pm 0.08) \times 10^{-6}$</td>
</tr>
<tr>
<td>BeCu coated with Polyimide</td>
<td>$(7.9 \pm 0.3) \times 10^{-7}$</td>
</tr>
<tr>
<td>Torlon coated with Polyimide</td>
<td>$(2.5 \pm 0.1) \times 10^{-7}$</td>
</tr>
</tbody>
</table>
Valves

- There are lots of valves
- The valves (in the most demanding case) must be:
  - Large (1” aperture)
  - Minimal LHe open/closed volume change
  - Non-magnetic (and non-superconducting)
  - “3He friendly” (low wall depolarization probability)
  - Superfluid tight
  - Able to stand off 1 atm
  - Thermally isolating
  - Reliable (> 10,000 cycle lifetime)

E.g. plastic, ceramic
Valve Development

- Demonstration of a working valve is required.
- Our solution:
  - Self-cleaning “Cork-in-bottle seal”
  - High-strength Plastics (Torlon, Vespel)
  - Polyimide-coated BeCu Bellows – hidden by another seal
  - “Australian” V-groove flange seals with Kapton gaskets
  - Low conductance annular gap for thermal isolation
- Test plan:
  - Verify seal design
  - Fabricate a full-sized prototype valve
    - Include all essential features
  - Use reasonable materials
  - Test under realistic conditions
    - LHe II inside at ~1.7 K
    - Vacuum outside
- Tested Successfully
  - 10,000 Cycles
Valve Designs

Section view of the current “T” valve design (in closed position)

1” Torlon-Body Valve

1/2” Valve (IV2-to-SV)

Mini Valve

Injection Volume Valve

Open seal

Annular gap for thermal isolation

Closed seal

Small conductance channel to bellows

40 cm

SNS nEDM NSAC Review, April 1-2, 2011