Minimizing Pressure In-homogeneities for Large Samples in High Pressure Neutron Scattering Measurements

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Minimizing Pressure In-homogeneities for Large Samples in High Pressure Neutron Scattering Measurements

- Reasoning
- Apparatus
- Results
- Technique
Reasoning

A. Freezing ($V_{\text{constant}}$)
B. Cooling to Freezing Point ($P_{\text{constant}}$)
C. Freezing and Cooling ($VP_{\text{constant}}$)

W.F. Sherman, A.A. Stadtmuller. *Exp. Tech. in H. Pressure Research*

- Pressurize above $P_xT$ curve
- Cool slowly under $P_{\text{constant}}$ down to the freezing point
- Hope for the best down to base

Apparatus

$P_{\text{max}} = 7.0$ kbar

Working Pressure = 6.5 kbar

Al 7075-T6 Construction

1.5 cm$^3$ sample volume

69% Neutron transmission at 2Å

Harwood Eng., Inc. 2-Stage Intensifier
i. Pressurize above PₓT curve
ii. Cool slowly under VP\textsubscript{constant} down to the freezing point
iii. Hope for the best down to base

\[ P = \left( \frac{\beta_o}{\beta'} \right) \left[ \left( \frac{r}{r_o} \right)^{-\beta'} - 1 \right] \]

From 1-D Analog to the Murnaghan Equation

\[ \beta' = 10.8(9) \]

\[ \beta_o^{-1} = -\left( \frac{d \ln r}{dP} \right)_{P=0} = 373^{-1} \text{ kbar} \]

Freezing and Cooling ($VP_{constant}$) for neutron elastic measurements of HOPG ($c/c_0$)

Results

Freezing and Cooling (\(V P_{\text{constant}}\)) \(\rightarrow\) \(\Delta P \approx 25\%\)
Results

Cooling to Freezing Point ($P_{\text{constant}}$)

$\Delta P_{AV} \approx 5\%$

When systematically ensuring that the pressure vessel is completely full of solid He
Results

Cooling to Freezing Point ($P_{\text{constant}}$)

$\Delta P_{\text{AV}} \approx 5\%$

When systematically ensuring that the pressure vessel is completely full of solid He

5mm x10mm HOPG Xtals $\sim 10^\circ$ offset
Results

- Freezing and Cooling ($V_{\text{constant}}$)
  \[ \rightarrow \Delta P \sim 25\% \]

- Freezing and Cooling ($P_{\text{constant}}$) for neutron elastic measurements of HOPG ($c/c_o$)
  \[ \rightarrow \Delta P_{AV} \sim 5.5\% \]

A. Freezing ($V_{\text{constant}}$)
B. Cooling to Freezing Point ($P_{\text{constant}}$)
C. Freezing and Cooling ($V_P_{\text{constant}}$)

Freezing and Cooling ($P_{\text{constant}}$) for neutron elastic measurements of HOPG ($c/c_o$)

Freezing and Cooling ($P_{\text{constant}}$) $\rightarrow \Delta P_{AV} \sim 5\%$

Results

Freezing and Cooling \( (P_{\text{constant}}) \rightarrow \Delta P \sim 5\% \)

When systematically ensuring that the pressure vessel is completely full of solid He

Bonus: Sample pressure in-homogeneities are minimized
Results

Sample Pressure in-homogeneities ($P_i$) Comparison

Freezing and Cooling ($VP_{\text{constant}}$) $\rightarrow \Delta P \sim 25\%$

Burp Freezing and Cooling ($P_{\text{constant}}$) $\rightarrow \Delta P \sim 5\%$
Technique

- Use He as pressure media
- Monitor pressure line temperature
- Control line temperature well above desired PₓT
- Cool down to a few Kelvin of PₓT curve
- Apply pressure while heating line
- Begin ramp-cooling through the PₓT curve
- Systematically heat line upon transducer pressure “freeze” providing enough power to counteract the cooling of the cell as noted in the sample stick sensor
- Once transducer pressure “melts” reduce line heater power to once again “freeze” the transducer reading
- Repeat until \( P_{\text{Freeze}} \sim P_{\text{Melt}} \)
- Continue cooling to base while still heating line
- Begin reducing line heater power when the cryostat cooling bottoms out
- Turn off line heater when \( T_{\text{Line}} < T_{\text{Freeze}} \)
Minimizing $\Delta P_{\text{Freeze}} P_{\text{Melt}}$

$\Delta P_F P_M(i) < \Delta P_F P_M(ii) < \Delta P_F P_M(iii)$

$\text{(P x T)}_{\text{Helium}}$
Results

Freezing and Cooling ($P_{\text{constant}}$) → $\Delta P \sim 5\%$

When systematically ensuring that the pressure vessel is completely full of solid He

Bonus:
Sample pressure in-homogeneities are minimized
Results

Sample Pressure In-homogeneities ($P_i$) Comparison

Freezing and Cooling ($P_{\text{constant}}$) $\rightarrow \Delta P \sim 25\%$

Burp Freezing and Cooling ($P_{\text{constant}}$) $\rightarrow \Delta P \sim 5\%$
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✓ Reasoning
✓ Apparatus
✓ Results
✓ Technique
Thank You!