고체 헬륨의 비고전적 회전관성과 
충밀리기 탄성률

Non-Classical Rotational Inertia and Shear Modulus of Solid Helium

김덕영 (金德英 Kim, Duk Young)
물리학과
Department of Physics

KAIST
2010
고체 헬륨의 비고전적 회전관성과 층밀리기 탄성률

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Advisor : Professor Kim, Eunseong

by

Kim, Duk Young
Department of Physics
KAIST

A thesis submitted to the faculty of the KAIST in partial fulfillment of the requirements for the degree of Doctor of Philosophy in the Department of Physics

Daejeon, Korea
2010. 5. 4.

Approved by

________________________
Professor Kim, Eunseong
Advisor
고체 헬륨의 비교전적 회전관성과 총밀리기 탄성률

김덕영

위 논문은 한국과학기술원 박사학위논문으로 학위논문 심사위원회에서 심사 통과하였음.

2010년 5월 11일

심사위원장 김은성 (인)

심사위원 김동락 (인)

심사위원 김형찬 (인)

심사위원 심홍선 (인)

심사위원 이순철 (인)
Abstract

Supersolid is a state of matter that has the properties of a solid and a superfluid simultaneously. In 1970, non-classical rotational inertia (NCRI) was suggested to be a consequence of supersolidity; this property was detected by Kim and Chan in 2004, who found a drop in the resonant period of a torsional oscillator. However, the exact mechanism of the phenomenon is not clear yet. This dissertation presents three experimental approaches to revealing the origin of the supersolid state. The purpose of the first experiment is to look for a 2-dimensional supersolid. In 2-dimensional helium films, the first few atomic layers, which are called the inert layer, are strongly localized and considered to be solid-like. In order to find the superfluidity in the inert layer helium films, torsional oscillators containing porous Vycor glass were used. However, no signature of NCRI was found in the inert layer films that were adsorbed on Vycor glass. The helium films adsorbed on the Vycor glass are known to be amorphous. This amorphous structure or strong interaction between helium and Vycor might have prohibited the appearance of superfluidity.

Meanwhile, Day and Beamish observed a shear modulus increase in solid helium at low temperatures in 2007. This result brought a lot of interest because it showed remarkable similarities with the NCRI in the torsional oscillator experiments. Although the rotational inertia and the shear modulus are different physical quantities, they showed similarities in temperature dependence, drive amplitude dependence, frequency dependence, $^3$He concentration dependence, hysteresis and dissipation. These similarities indicate that the two phenomena are closely related. It was suggested that the origin of the shear modulus increase was the dislocation pinning to $^3$He impurities.
To clarify the relationship between the NCRI and the shear modulus increase, two experiments have been performed.

In the solid helium inside a narrow pores, it is hard for motions of dislocations to take place. The non-classical response of solid $^4$He confined in porous gold that has pore size of 180 nm was investigated. As a result, large NCRI, from 1.4% to 7%, have been observed. These results confirmed that NCRI can exist when the motions of the dislocations are restricted. The largest NCRI of 7% was acquired by quench cooling the solid. The large surface area of porous gold seems to enhance the cooling efficiency and increase disorder. Extended relaxations of the NCRI were also observed at low temperatures. The long relaxation in the solid confined in small pores should originate from superfluidity.

In order to directly investigate the relationship between the NCRI and the shear modulus increase, both measurements were done simultaneously in a single experimental setup. A pair of piezoelectric transducers were positioned in the center of a torsional oscillator to measure the shear modulus. The temperature dependence and the drive amplitude dependent suppression are major common features in both phenomena. The drive stress and temperature dependence of the shear modulus have been systematically studied and interpreted based on the thermally assisted unpinning process of dislocations from impurities. However, NCRI was found to be reduced with much lower drive stress than the shear modulus. This means that the NCRI can be suppressed even when all the dislocations are pinned. Moreover, no linear relationship between the magnitude of the NCRI and the shear modulus increase was found. In addition, the NCRI shows slower relaxation dynamics than the shear modulus with abrupt temperature change. Based on all these results, the microscopic origin of the NCRI seems to be different from that of shear modulus increase.
Dedicated to my beloved wife, Y. K. Sung.
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1. Introduction

1.1 Supersolid

1.1.1 Early theories

Supersolid is a state of matter characterized by the coexistence of crystallinity and superfluidity. The usual definition of solid is the substance possessing a shear modulus which is a consequence of localization of atoms. On the contrary, in a superfluid state, atoms are indistinguishable and delocalized. This paradoxical phenomenon was proposed in 1969-1970 by a few theorists [1, 2, 3, 4]. Andreev and Lifshitz [2] argued that the defects or vacancies in quantum solid can exchange its position with a neighboring atom by quantum tunneling. If the exchange frequency is large enough, the vacancy become a quantum wave that is delocalized in the whole lattice. Its energy lies inside a certain band and can be negative so that the crystal is invaded by a finite density of vacancies, which are called ‘zero point vacancies’. In case of the crystal lattice consisted of Bose particles, the vacancies are also Bose particles and should undergo a Bose-Einstein condensation at a sufficiently low temperature. Leggett [4] predicted, in the case of the supersolidity, the crystal should exhibit nonclassical rotational inertia (NCRI) properties.

1.1.2 Non-classical Rotational Inertia of Solid Helium

In 2004, Kim and Chan reported anomalous behavior of solid helium in a series of torsional oscillator experiments [5, 6]. The resonant period of the torsional oscillator which contains solid helium showed gradual drop below $T_0 \approx 200$ mK as temperature decreased. This abnormal period drop was suggested to be non-classical rotational inertia (NCRI) and a consequence of superfluidity in solid helium. The resonant
The period of a torsional oscillator is given by $\tau = 2\pi\sqrt{I/K}$ where $I$ is the moment of inertia of the torsion bob and $K$ is the torsional spring constant of the torsion rod.

The relative change in the rotational inertia is called non-classical rotational inertia fraction (NCRIF) and it is identified with the superfluid fraction.

$$NCRIF = \frac{\rho_s(T)}{\rho} = \frac{I(T_0) - I(T)}{I(T_0) - I_{\text{empty}}} = \frac{\tau(T_0) - \tau(T)}{\tau(T_0) - \tau_{\text{empty}}}$$  \hspace{1cm} (1.1)$$

$I_{\text{empty}}$ and $\tau_{\text{empty}}$ mean the inertia and the period of the empty oscillator. The measured value of NCRIF was approximately 1%.

The period drop of the torsional oscillator was accompanied by a peak in the dissipation $Q^{-1}$ of the system at the temperature where the period change was maximum. The existence of a dissipation peak is well known phenomenon in a two-dimensional superfluid (Kosterlitz-Thouless) transition. It is understood to be a consequence of diffusive motions of two-dimensional vortices. However, the origin of the dissipation in solid helium is still in dispute. NCRI showed strong dependence on the driving amplitude of the oscillator. If the oscillating amplitude was increased, the NCRI was suppressed with velocities larger than about 10 $\mu$m/s. This value was interpreted as the critical velocity $v_c$ of the superflow and was comparable to the Onsager-Feynman

![Figure 1.1](image.png)

Figure 1.1: The resonant period of the torsional oscillator containing solid $^4$He and the schematic diagram of the torsional oscillator [5].
quantized circulation velocity, $v_s = n \cdot \hbar / mR$, with $n = 1$ [7]. The temperature ranges where NCRI appeared were shifted depending on the $^3$He concentration of the solid samples. This effect tells us that the impurities and disorder play important role in the NCRI phenomena.

The results of control experiments solidified the supersolid interpretation on the anomaly. In the first control experiment, the torsion bob was replaced with a dummy mass and the fill line in the torsion rod was filled with solid helium. No NCRI in the dummy cell excluded the possibility that the period change had originated from the stiffening of torsion rod. The second control experiment was the measurement on a $^3$He solid. The NCRI was not observed in the solid $^3$He sample. Although it had been bcc $^3$He crystal, hcp $^3$He crystal also turned out later to show no NCRI. The most crucial control experiment was the blocked annulus experiment. If the annular channel for the solid helium was blocked by a barrier, the NCRI was greatly reduced. This result suggests the macroscopic quantum coherence in the sample.

1.1.3 Recent experimental studies

NCRI was reproduced in many groups after the Kim and Chan experiment [8, 9, 10, 11, 12]. The qualitative properties of the measured NCRI were consistent among all groups. However the quantitative properties were observed in vary wide range. The magnitudes of the NCRI fraction (NCRIF) varied from 0.02% up to 20% and the onset temperatures varied between 60 mK and 400 mK. These variations exist not only between groups, but also from sample to sample within same cells. Rittner and Reppy reported significant decrease of NCRI by annealing the samples [8]. And they obtained an NCRIF as large as 20% by quench cooling the solid sample in a narrow cylindrical channel [13]. They also noticed that the NCRIF was increased with the surface to volume ratio of the the sample cell. They claimed that the possibility of greater frozen-in disorders increased as the S/V ratio increased. These sample
dependences of NCRI point out the importance of disorders in NCRI.

Clark and coworkers investigated the effect of the crystal quality of samples on NCRI by growing solid at constant T and constant P [14]. The solid helium samples grown from the superfluid are known to be single crystals [15]. They observed NCRI in the range 0.03~0.3% with an onset temperature $T_0 = 75$ mK for 1 ppb $^3$He samples and $T_0 \simeq 150$ mK for 300 ppb samples. The temperature dependence of NCRI is reproducible and exhibits a sharper onset and agrees with the two-thirds power law, which is expected for a superfluid transition.

By Lin and coworkers in Chan’s group, the temperature dependence of the heat capacity of solid helium was measured [16]. They observed a broad peak centered at 75 mK if the $T^3$ phonon term was subtracted from the data. They claim this peak as the thermodynamic signature of the supersolid. In more recent study, they measured the heat capacity of the sample which consisted of 75% solid in coexistence with liquid and found that a peak existed as well [17]. They suggest that its presence in an essentially stress-free sample implies that NCRI is intrinsic to solid helium and the role of stress and dislocations is only to enhance the effect.

1.1.4 Recent theoretical studies

In theoretical aspect, the activation energy of vacancies is calculated to be as large as $E_{\text{vac}} = 10$ K [18]. As a consequence, not only are there no zero point vacancies, but at temperatures of a few hundred millikelvin the density of thermally activated vacancies is negligible. However, possibilities of superfluidity along the cores of screw dislocations [19], along grain boundaries [20] and along certain types of edge dislocation [21] were reported and the issue is still in controversy.

P. W. Anderson suggested a vortex liquid state as an origin of the phenomena measured by torsional oscillator experiments [22]. According to his argument, the true supersolid transition occurs at a much lower temperature than the typical onset
temperature in the torsional oscillator experiments. The relevant physics observed by the torsional oscillator is that of quantized vortices whose motion can dissipate energy by phase slippage. The dissipation peak is observed at temperatures where the vortex damping rate and the probing frequency coincide. Several experimental results have been explained in terms of vortex liquid but no quantitative description was reported yet.

1.2 Shear modulus of Solid Helium

1.2.1 Day and Beamish experiment

In 2007, Day and Beamish reported shear modulus increase in solid helium at low temperatures [23]. Although the measured physical property is different from the torsional oscillator experiment, the shear modulus stiffening shows similarities with NCRI in temperature dependence, drive amplitude dependence, frequency dependence, hysteretic behavior and dissipation. First of all, the two measurements show transition

![Figure 1.2: Shear modulus of solid $^4$He as a function of temperature. Top curve (open circles, right axes) shows typical NCRI fraction from a torsional oscillator measurement in a 65 bar sample.](image-url)
around 200 mK with broad onsets. Although the transition temperatures deviate from sample to sample by experimental condition, the directions of the temperature shift by $^3$He concentration were same in the shear modulus and NCRI. In both case the temperature ranges shifted to lower, around 60 mK, by making solid from ultra pure, 1 ppb, $^4$He. The both anomalies are suppressed by increasing the drive amplitude. As increasing the driving frequency of the piezoelectric shear transducer, the temperature range where the shear modulus changes broadened and increase to higher temperature. This is consistent with the double frequency torsional oscillator experiment by H. Kojima’s group. Aoki et al. made a double pendulum torsional oscillator that has resonant frequencies of 495.8 Hz and 1172.8 Hz [9]. In this experiment, the NCRI was observed at higher temperatures with the high frequency mode than with the low frequency mode.

1.2.2 Dislocation pinning to $^3$He impurities

The mechanism of the shear modulus increase was suggested to be pinning of dislocation lines to $^3$He impurities based on the Granato-Lücke theory [24]. In this model, the motions of dislocations are described by vibrating strings. The displacement of the dislocation follows the equation

$$A \frac{\partial^2 \xi}{\partial t^2} + B \frac{\partial \xi}{\partial t} - C \frac{\partial^2 \xi}{\partial y^2} = b \sigma$$

(1.2)

$A = \pi \rho b^2$ is the effective mass per unit length, $B = gT^\nu$ is the damping force per unit length and $C = 2\mu b^2/\pi(1 - \nu)$ is effective tension, where $\rho$ is the density of the material and $b$ is the Burgers vector, $\mu$ is the shear modulus and $\nu$ is Poisson’s ratio. The motion of the dislocation lines gives the extra strain in solid.

$$\epsilon = \epsilon_{el} + \epsilon_{dis}$$

(1.3)

$$\epsilon_{dis} = R\Lambda b\bar{\xi}$$

(1.4)
R is an orientation factor depending on the polarization of the elastic wave and the crystal orientation and Λ is the dislocation density. This extra strain caused the decrement and shear modulus change of the solid.

The resonance frequency of a dislocation segment of length L is

\[ \omega_0 = \frac{\pi}{L} \sqrt{\frac{C}{A}} = \frac{1}{L} \sqrt{\frac{2\mu}{(1 - \nu) \rho}} \]  

which is usually MHz in solid helium. Assuming a low frequency static limit, \( \omega \ll \omega_0 \), and a negligible damping constant \( B \), the dislocation part of the strain is given as

\[ \epsilon_{\text{dis}} = \frac{R\Lambda \sigma}{\pi \rho \omega_0^2} \]  

The macroscopic shear modulus that is measured in the experiment is

\[ \mu_m = \frac{\sigma}{\epsilon_{\text{el}} + \epsilon_{\text{dis}}} \]

\[ \mu_m = \frac{\mu}{1 + \frac{R\Lambda}{\pi \rho \omega_0^2}} \approx \mu \left(1 - \frac{R\Lambda L^2 (1 - \nu)}{2\pi}\right) \]  

The shear modulus change can be written as

\[ \frac{\Delta \mu}{\mu} = \frac{(1 - \nu)}{2\pi} R\Lambda L^2 \]  

This result is for the case that all the dislocations have a length of L. If an exponential distribution with average length L is assumed, the shear modulus change is given as below [25].

\[ \frac{\Delta \mu}{\mu} = \frac{8\mu b^2}{\pi^4 C} 6\Lambda L^2 = \frac{24(1 - \nu)}{\pi^3} R\Lambda L^2 \]  

Another suggested mechanism is roughening of the dislocation network [26].

### 1.2.3 The effect of the shear modulus increase on torsional oscillators

The dependence of torsional oscillators on the shear modulus change of solid helium need to be discussed. Clark et al. calculated the quantitative influence of the shear
modulus change on the resonant frequency of their torsional oscillator [27]. For the small changes of shear modulus, \( \mu \), the expected frequency change is almost linear.

\[
 f (\mu + \delta \mu) = f (\mu) + \delta f \approx f (\mu) + \delta \mu \frac{df}{d\mu}
\]  

\[
 \frac{df}{f} \approx \frac{\delta \mu df}{f d\mu} = \left( \frac{d \ln f}{d \ln \mu} \right) \frac{\delta \mu}{\mu}
\]  

The magnitude of \( \frac{d \ln f}{d \ln \mu} \) was \( 5 \times 10^{-7} \) if it was acquired by a simple analytical model, and \( 5 \sim 10 \) parts in \( 10^6 \) if it was obtained by finite element method calculations. Although the inferred temperature dependence of the frequency change is identical to the change of the shear modulus, the magnitude of the change is only between one-fifth and half of the experimental results.

Iwasa applied a dislocation model to analytical solutions for the torsional oscillator containing solid helium and suggested that NCRI could be caused by the variation of the average pinning length of dislocations [28]. The equation of motion for a torsional oscillator (TO) is given by

\[
 I_{TO} \frac{d^2 \phi}{dt^2} + \kappa \phi = \tau
\]

where \( I_{TO} \) is the rotational inertia of the TO, \( \phi \) is the torsional angle, \( \kappa \) is the torsional constant and \( \tau \) is the torque exerted by solid helium on the wall of the TO. If a cylindrical TO with radius \( R \) and height \( H \) is assumed and the effect of the top and bottom plates are neglected, the torque is

\[
 \tau = -2\pi R^2 H \sigma (R)
\]

Using a theory of elasticity, the shear stress can be calculated.

\[
 \sigma_{\theta \theta} = -\mu u_0 k_r \frac{J_2 (k_r R)}{J_1 (k_r R)} e^{-i\omega t}
\]

where \( J_1, J_2 \) denote Bessel’s function and \( k_r \) is the wave number for shear wave in the radial direction which satisfies the dispersion relation

\[
 \mu k_r^2 = \rho \omega^2
\]
\( \mu \) and \( \rho \) are the shear modulus and the density of solid helium, respectively.

The first order approximation on Eq. 1.15 gives the resonant period of TO as

\[
p = 2\pi \sqrt{I_{TO} + I_{He} \frac{\kappa}{\kappa}}
\]  

(1.17)

, in which the period is independent of the shear modulus.

The second order approximation gives the resonant period as

\[
p = 2\pi \sqrt{I_{TO} + I_{He} \left(1 + \frac{\rho \omega^2 R^2}{24\mu}\right) \kappa}
\]  

(1.18)

This result is same as that of analytic derivation by Clark et al. [27].

Iwasa claim that the vibration of dislocation causes the wave number of the shear wave in solid helium to increase. Considering the dislocation part of the strain is given as in Eq. 1.6, the dispersion relation, Eq. 1.16, should be replaced with

\[
\mu_{el} k'^2 = \rho \omega^2 \left(1 + \frac{\mu_{el} \Omega \Lambda}{\pi \rho \omega_0^2}\right)
\]  

(1.19)

The first order approximation on the equation of the shear stress gives the resonant period of TO as

\[
p = 2\pi \sqrt{I_{TO} + I_{He} \left(1 + \frac{\mu_{el} \Omega \Lambda}{\pi \rho \omega_0^2}\right) \kappa}
\]  

(1.20)

This result suggests that the magnitude of mimicked NCRIF by the dislocation motion is same as the amount of the shear modulus change.

\[
NCRIF = \frac{\Omega \Lambda \sigma}{\pi \rho \omega_0^2} = \frac{(1 - \nu)}{2\pi} R \Lambda L^2
\]  

(1.21)

However, my experimental results didn’t satisfied this quantitative prediction.

### 1.2.4 Shear modulus increase in \(^3\)He solid

In 2009, Beamish and Chan collaborated and measured the shear modulus and NCRI of solid \(^3\)He [29]. As a result, the low temperature increase of the shear modulus was observed in hcp \(^3\)He though it was not observed in bcc \(^3\)He. As for the NCRI, they
couldn’t see the anomaly in hcp $^3$He as well as in bcc $^3$He. From the fact that the shear modulus increase occur only in hcp not in bcc crystals, we can conjecture that the shear modulus increase is crystal structure dependent. But the NCRI occur only in $^4$He solid not in $^3$He solid. Thus, the NCRI could be quantum statistics dependent and occur only in a stiffened Bose solid.

Figure 1.3: (A) Shear modulus of hcp $^4$He, hcp $^3$He and bcc $^3$He. (B) The period and (C) dissipation of the torsional oscillator containing hcp $^4$He, hcp $^3$He and bcc $^3$He.
1.3 2-dimensional helium films

1.3.1 Kosterlitz-Thouless transition in helium films

The superfluidity in helium films is a realization of the Kosterlitz-Thouless (KT) transition predicted for 2-dimensional systems with a broken continuous symmetry [30, 31]. Unlike 3-dimensional or bulk helium, the “ordered” or low-temperature phase does not exhibit true long-range order in 2D helium films. Instead, it shows a power-law decay of the order-parameter correlation length. According to the KT theory, vortices are topological excitations in the 2D XY model and the 2D superfluid is populated by vortices and antivortices. Binding of vortex-antivortex pairs at low temperatures restores phase coherence. As the temperature increased above $T_{KT}$, the transition temperature, the vortex-antivortex pairs are dissociated by thermal fluctuations. The superfluid density $\rho_s$, the order parameter, is directly proportional to $T_{KT}$ at a temperature just below the transition.

$$\rho_s = \frac{2k_B m^2}{\pi \hbar^2} T_{KT}$$ (1.22)

where $k_B$ is Boltzmann’s constant and $m$ is the mass of the $^4$He atom.

1.3.2 The inert layer in 2-dimensional helium films

Even though the superfluid transition of two dimensional helium films shows good agreement with Kosterlitz-Thouless transition, the KT theory does not account for the $^4$He-substrate interaction. An ideal 2D film would be a free-standing helium layer. However, the experimental realization of 2D film is usually accomplished by adsorbing helium atoms to an substrate with large surface area. Due to disorders and the van der Waals interaction with the substrate, the first few atomic layers of a helium film do not participate in superflow and effectively localized. This strongly adsorbed helium film is called the inert layer.

If we assume the average interatomic distance in a helium film is same as in bulk,
one monolayer $^4$He film corresponds to the coverage of 12.8 $\mu$mol/m$^2$. Then, the coverage of the inert layers in conventional substrates like Vycor glass [32], Mylar [33], Aerogel [34] and porous gold [35] is approximately two monolayers. This non-superfluid $^4$He layer shields away all irregularities of the substrate and forms an ideal substrate for the superfluid above it. Since the inert layer and the superfluid layer are considered independent, the zero temperature superfluid density $\rho_{s0}$ can be obtained by subtracting the inert layer coverage $n_0$ from the total $^4$He coverage $n_4$.

$$\rho_{s0} = n_4 - n_0 \quad (1.23)$$

Combining this relation with the KT prediction, Eq. 1.22, gives

$$T_c \propto n_4 - n_0 \quad (1.24)$$

The superfluid onset temperature $T_c$ increases as the surface coverage increases and the linear dependence extrapolate to $n_4 = n_0$. The substrate can be modified by pre-plating it with other materials. Preplating with Ne greatly reduced the inert coverage to 13 $\mu$mol/m$^2$ [36] and with H$_2$ the inert coverage was only half of a $^4$He monolayer [35]. Csáthy and coworkers systematically studied the substrate dependence of the inert coverage and found that the inert coverage showed monotonic dependence on the well depth of the $^4$He-substrate potential and $^4$He-substrate binding energy [37].

The well depth $D$ is given in the interaction potential

$$V(z) = \frac{4C_3^3}{27D^2z^9} - \frac{C_3}{z^3} \quad (1.25)$$

where $C_3$ is the van der Waals constant determining the interaction at long length scales [38].

### 1.3.3 Search for superfluidity in the inert layer helium films

The localized $^4$He atoms in the inert layer are considered as if they are solid. Sarfatt suggested in 1969 that adsorbed solid helium films, at coverages below the value for
onset of conventional superfluid film behavior, constitute possible candidate for a supersolid system [39]. If the superfluidity in the inert layer helium films exists, it might be related with supersolid.

Crowell and Reppy’s work suggests the possibility of the superfluidity in the inert layer helium film. They observed a reentrant superfluid phase in the second layer of helium film adsorbed on graphite [40, 41]. Figure 1.4 shows the temperature dependence of the reentrant superfluidity. Unlike most KT transitions in superfluid helium films, the reentrant superfluidity shows broad transition that do not approach an asymptotic value as the temperature is decreased toward zero. And the period shift varies linearly with the logarithm of the temperature. The coverage at which this reentrant superfluidity appears is slight below the completion of the second layer. No superfluidity was found at the completion of the second layer and the conventional 2-dimensional superfluidity appeared just above the second layer completion. Motivated by this reentrant superfluidity, we started the torsional oscillator experiment to find the superfluidity in the inert layer helium films adsorbed on Vycor glass.

1.4 Superfluid helium in Porous Media

The superfluidity of $^{4}\text{He}$ has been studied inside porous media in numerous experiments. Not only for 2-dimensional films but also for the bulk superfluid the porous materials have been used as hosting substrates. Vycor glass and porous gold are useful and interesting porous materials made through chemical etching process.

1.4.1 Superfluid helium in Vycor glass

The pores of Vycor glass have interconnected network of worm-hole like structure and average diameter of 7 nm. The porosity is approximately 30% of the total volume. In the first experiment using Vycor, the onset temperature for superfluid liquid $^{4}\text{He}$ was reduced several hundreds mK below the bulk transition temperture, $T_{\lambda}=2.172$ K
The most interesting results on the superfluidity in Vycor is that the superfluid density of $^4$He shows a power law temperature dependence near $T_c$, almost identical to that of bulk $^4$He \cite{43}. The superfluid $^4$He in Vycor is considered to behave at the phase transition as a 3-dimensional system that belongs to the same universality class as that of bulk $^4$He \cite{44}. Although the pore size of the Vycor is very small, the open channels seem to be sufficiently interconnected to allow the superfluid to behave as a 3-dimensional system. The superfluid $^4$He in Vycor might become a diluted homogeneous 3-dimensional system since the correlation length of superfluid $^4$He in Vycor is larger than the characteristic length of the pore structure in Vycor.

Also in the superfluid film adsorbed on the Vycor, the superfluid density follows a power law in reduced temperature similar to that of bulk $^4$He \cite{45}. The 3-dimensional character of the substrate seems to produce a dilute 3-dimensional surface gas in contrast to other experiments in 2-dimensional geometry.

![Figure 1.4: The period shift data for the second layer helium films adsorbed on the graphite substrate \cite{41}](image)
1.4.2 Superfluid helium in porous gold

The porous gold is gold substrate which has multiply connected uniform pore structures. The structure of porous gold is an interpenetrating solid-void composite similar to that of Vycor. The pore diameter can be tuned to be as small as 24 nm and as large as a few microns depending on the manufacturing environment and heat treatment. The porosity of porous gold is about 70%. The superfluid density exponent is found to be identical to that of bulk $^4$He meaning that it belongs to the same universality class as that of bulk $^4$He and $^4$He in Vycor [46].

The solid helium in porous substrates also have been investigated [5, 47]. The complicated porous structure prohibits any elastic motion of line defects larger than a characteristic pore diameter. Therefore, NCRI in porous media cannot be understood in terms of a stiffening of solid helium induced by the impurity pinning of the dislocation network.

![SEM image of porous gold](image)

Figure 1.5: SEM image of porous gold [48]
2. Experimental Methods

2.1 Dilution refrigerator

Using a dilution refrigerator is the most effective way to have temperatures below 100 mK nowadays. We can simply understand the cooling mechanism of a dilution refrigerator as the evaporation of $^3\text{He}$ atoms into $^4\text{He}$ phase in $^3\text{He}/^4\text{He}$ mixtures. $^3\text{He}$ atoms are more strongly bound to $^4\text{He}$ than among each other because the $^4\text{He}$ atoms have a lower zero-point energy due to their larger mass [49]. The $^3\text{He}$ atoms obey the Fermi statistics and thus their kinetic energy increases with their number density. Because of this, the effective binding energy is reduced. At a $^3\text{He}$ concentration of 6.5% in $^4\text{He}$, the effective binding energy vanishes as temperature goes to zero, and no further $^3\text{He}$ atoms can be dissolved in the $^4\text{He}$. Two phases are more favorable when the average $^3\text{He}$ concentration is greater than 6.5%. A light, $^3\text{He}$-rich phase and a heavy phase mainly consisting of $^4\text{He}$ atoms are formed. The maximum solubility of the $^3\text{He}$ atoms in the heavy phase depends on temperature and pressure.

Figure 2.1 shows the phase diagram of liquid $^3\text{He}$-$^4\text{He}$ mixtures at saturated vapor pressure. At high temperatures, the $^3\text{He}$ concentration is constant and leads into a common curve at very low temperatures. This curve reflects the maximal solubility of $^3\text{He}$ in $^4\text{He}$ under normal pressure as a function of temperature. Below this curve, a miscibility gap opens up and two separate phases with different $^3\text{He}$ concentrations coexist at the same temperature. The $^3\text{He}$ atoms in the $^3\text{He}$-rich phase have a lower entropy than the $^3\text{He}$ atoms in the $^4\text{He}$-rich phase. The cooling process of a dilution refrigerator takes place in the so-called mixing chamber and consists of the transfer of $^3\text{He}$ atoms from the $^3\text{He}$-rich phase into the dilute phase. In analogy to the evaporation of gases, one sometimes refers to this process as an evaporation into the
quasivacuum of the superfluid $^4\text{He}$. In order to use this cooling mechanism for continuous operation we have to remove $^3\text{He}$ from the dilute phase in the mixing chamber and feed it back into the $^3\text{He}$-rich phase.

The centerpiece of a dilution refrigerator is the cold part of the $^3\text{He}/^4\text{He}$ circuit that is shown schematically in Figure 2.2. This part is located in a vacuum chamber that is immersed into a $^4\text{He}$ bath. It essentially consists of the mixing chamber, the still and a counterflow heat exchanger. The relatively complex $^3\text{He}/^4\text{He}$ circuit shown in Figure 2.2 is necessary to realize a sufficiently high circulation of $^3\text{He}$, while maintaining a low heat load at the mixing chamber. The circulation of the $^3\text{He}/^4\text{He}$ mixture is driven by pumping the still. The still is heated to about $0.7$ K to increase the efficiency of the pumping. Because of its higher vapor pressure, $^3\text{He}$ is predominantly evaporated from the liquid, although the $^3\text{He}$ concentration in the liquid in the still is only about $1\%$. Once it has been pumped, the $^3\text{He}$ is cleaned outside the

![Figure 2.1: Phase diagram of liquid $^3\text{He}$–$^4\text{He}$ mixtures at saturated vapor pressure](image)

Figure 2.1: Phase diagram of liquid $^3\text{He}$–$^4\text{He}$ mixtures at saturated vapor pressure [50]
cryostat in cold traps before being returned to the cryostat. Following this step, the $^3$He enters the vacuum chamber in a capillary and is precooled at the 1 K pot. The pressure of the $^3$He is maintained sufficiently high by using a flow impedance before the still so that it condenses. After the still, the $^3$He is led into the counterflow heat exchanger that consists, in most systems, of two different types of heat exchangers. The first one is called a continuous heat exchanger and is normally made of two tubes that are arranged with one inside the other in a rather complicated manner so that the interface between the two is as large as possible. The second heat exchanger consists of several chambers each of which has a dividing wall with sintered silver attached to it in order to increase the thermal contact area. This type of heat exchanger is called a step heat exchanger. After passing through the heat exchangers the $^3$He enters the mixing chamber. The return line to the still starts in the mixing chamber below the phase boundary in the $^4$He-rich phase. On the way back to the still, the cold mixture again flows through the heat exchangers and in this way precools the incoming $^3$He. Pumping the still results in a concentration gradient and, in turn, to an osmotic pressure that causes $^3$He to flow from the mixing chamber to the still. This is, of course, only possible if $^3$He atoms cross the phase boundary in the mixing chamber, which leads to cooling. With this method, typically base temperatures of about 5 mK can be produced.
Figure 2.2: Schematic diagram of a dilution refrigerator
2.2 Torsional Oscillator

The torsional oscillator is a highly sensitive micro-balance which has been widely used in superfluid research. The resonant period of the mechanical oscillator is given by

$$2\pi\sqrt{I/K}$$

where $I$ is the rotational inertia of the torsion bob and $K$ is the torsional spring constant of the torsion rod [51].

2.2.1 Electrical operation

The torsional oscillator is driven and maintained at resonance by a pair of electrodes which are capacitively coupled to the wings of the oscillator. On the drive side the electric force is induced by applying a voltage between the wing and the drive electrode. The applied voltage is composed of two components. One is a DC voltage, $V_0$, obtained from biasing the wings by a series of batteries and the other is an AC voltage, $\Delta V e^{i\omega t} (\Delta V \ll V_0)$, that is supplied by the lock-in reference output. The amplitude of oscillation can be varied by the modulation of the AC driving voltage. The core of the torsional oscillator technique is adjusting the driving frequency at the resonance frequency by feeding back the detecting signal to the reference of the lock-in amplifier. On the detection side the capacitance is given by $C = A\varepsilon_0/d$, where $d$ is the gap between the wing and the detection electrode and $A$ is the area of the electrode. With the motion of the oscillator, the gap $d$ is oscillating and hence the capacitance, $C$, oscillates. As a result, a tiny AC current is induced between the wing and the electrode as below.

$$I = \frac{dQ}{dt} = d(CV_0) = V_0\frac{d}{dt}\left(\frac{A\varepsilon_0}{d_0 + \Delta de^{i\omega t}}\right) \approx -i\omega V_0\frac{A\varepsilon_0}{d_0^2} \Delta d e^{i\omega t} \quad (2.1)$$

The induced current is sent to lock-in amplifier through a current pre-amplifier. The maximum displacement and velocity of oscillation are given respectively as

$$\Delta d = \frac{IA\varepsilon_0}{V_0C_0^2\omega} \quad (2.2)$$
\[ v = \omega \Delta d = \frac{IA\varepsilon_0}{V_0 C_0^2} \]  

(2.3)

where \( C_0 \) is the capacitance between the wing and the detecting electrode. The DC bias voltage is usually \( V_0 = 100 \sim 300 \) V. The gap between the wing and the electrode is typically \( 200 \sim 300 \) \( \mu \)m and the corresponding capacitance is approximately 2 pF. Another voltage preamp is used in connecting the detecting signal to the reference input of a lock-in amplifier.

### 2.2.2 Mechanical setup

Although the torsion rod can be made of various metals, BeCu is used most widely. In case of BeCu, the heat treatment is known to increase the quality factor of the oscillator. The optimal condition of precipitation hardening for BeCu is the temperature of 315 °C and duration of 3 hour.

In order to get high quality factor, the vibration isolation is most important because torsional oscillators usually have resonant frequencies around 1 kHz, which correspond to acoustic sound wave. A torsional oscillator should be attached on a heavy mounting stage and the stage should be connected to a mixing chamber by a vibration isolator. The vibration isolator is just a thin copper rod with which the mounting stage should have very low resonant frequency. The vibrational noise with frequencies higher than the resonant frequency of the isolator are reflected and cannot penetrate to the torsional oscillator. I learned by trial and error that a torsional oscillator should be positioned at the center of a heavy mounting stage. And any accessories on the mounting stage should not have resonant frequencies relevant to that of the torsional oscillator. For example, thin posts for supporting wires or capillaries can have similar resonant frequencies with the oscillator and interfere with having high quality factors. Eigen frequency analysis on the hole experimental setup using a commercial software can be helpful.
2.3 Shear modulus measurement

2.3.1 Piezoelectric shear transducers

When a piezoelectric crystal is mechanically strained, or when the crystal is deformed by the application of an external stress, electric charges appear on the crystal surface. When the direction of the strain reverses, the polarity of the electric charge is reversed. This is called the direct piezoelectric effect. Conversely, when a piezoelectric crystal is placed in an electric field, or when charges are applied by external means to its faces, the crystal exhibits strain, i.e. the dimensions of the crystal change. When the direction of the applied electric field is reversed, the direction of the resulting strain is reversed. This is called the converse piezoelectric effect. The direction in which tension or compression develops polarization parallel to the strain is called the piezoelectric axis. From different combinations of the direction of the applied field and orientation of the crystal it is possible to produce various stresses and strains in the crystal. For example, an electric field applied perpendicular to the piezoelectric axis will produce elongation along the axis. If, however, the electric field is applied parallel to the piezoelectric axis, a shear motion is induced. The piezoelectric constant which relates the mechanical strain produced by an applied electric field are termed the strain constant $d_{ij}$. For an applied voltage $V_{in}$, $d_{11}$ will determine the resultant thickness change

$$\Delta x = d_{11} V_{in}$$

(2.4)

To determine the resultant voltage for the direct piezoelectric effect, two different piezoelectric constants are used. The piezoelectric deformation constant $h_{ij}$ is used to relate the resultant voltage to a given deformation. In this case the thickness change $\Delta x$, produces an output voltage according to

$$V_{out} = h_{11} \Delta x$$

(2.5)
A second constant, the piezoelectric pressure constant $g_{ij}$ is used to relate the resultant electric field to a given applied stress $\sigma$, the resultant voltage $V_{out}$ is given by

$$V_{out} = g_{11}\sigma t \quad (2.6)$$

where $t$ is the thickness of a transducer.

The capacitance is a quantity which depends both on the type of material and its dimensions whereas the relative dielectric constant is strictly a material property. At frequencies far below resonance, piezoelectric ceramic transducers are fundamentally capacitors. Consequently, the voltage coefficients $g_{ij}$ are related to the charge coefficients $d_{ij}$ by the dielectric constant $K_i$ (as in a capacitor the voltage is related to the charge by the capacitance).

$$d_{ij} = K_i\varepsilon_0 g_{ij} \quad (2.7)$$

### 2.3.2 Measurement principles

In the shear modulus measurement, almost same method as that of Day and Beamish was used [25]. The shear transducers used in the measurement were also same type (PZT-5A) as those of Day and Beamish. The subscripts in $d_{15}$ indicate that the voltage is applied to the electrodes which are at right angles to the original poling electrodes and that the applied mechanical stress is shear. With a applied voltage $V$, the shear displacement of $\delta x = d_{15}V$ is produced. The voltage generated across the detecting transducer is

$$V = Et = g_{15}\sigma t \quad (2.8)$$

The capacitance of the piezo-ceramic is given by

$$C = \frac{K_1\varepsilon_0 A}{t} \quad (2.9)$$

The charge generated on the face of the stressed electrode is $q = CV$ and if we consider the relation Eq. 2.7,

$$q = d_{15}\sigma A \quad (2.10)$$
The oscillation of the charge is measured as a current $I$

$$I = \omega q = 2\pi f q = 2\pi f d_{15} \sigma A$$  \hspace{1cm} (2.11)

where $f$ is the driving frequency. The stress $\sigma$ is related to the strain $\epsilon$ by the shear modulus $\mu$.

$$\mu = \frac{\sigma}{\epsilon}$$  \hspace{1cm} (2.12)

The strain of the helium in the gap $D$ between the two transducers is

$$\epsilon = \frac{\delta x}{D} = \frac{d_{15} V}{D}$$  \hspace{1cm} (2.13)

The output current from the detecting shear transducer equal to

$$I = 2\pi f \frac{A}{D} d_{15}^2 \mu V$$  \hspace{1cm} (2.14)

If the piezoelectric constant is assumed to be constant, the shear modulus $\mu$ is proportional to $I/fV$

$$\mu = \frac{D}{2\pi d_{15}^2 A} \frac{I}{fV}$$  \hspace{1cm} (2.15)
Using a typical value of the shear modulus in solid helium ($\mu \approx 1.5 \times 10^7$ Pa) at 35 bar, the low temperature value of $d_{15}$ is obtained as $9.8 \times 10^{-11}$ m/V.

### 2.3.3 Measurement Details

Since the gap between the driving and detecting transducers is very narrow and the metal electrodes are facing each other, the capacitive coupling between them can give electrical crosstalk. This problem can be avoided if we ground the two facing electrodes and connect wires on the opposite electrodes as in Figure 2.3. Since the electrodes on the PZT transducer are evaporated thin metal films, attaching electrical wire to them is a very finicky job. Soldering with indium is effective because it requires lower temperature than other solders like tin or lead.

Variable driving frequency is possible in the shear modulus measurement. Because the amplitude of the detecting signal varies with the driving frequency, the sensitivity of the current preamplifier should be adjusted depending on the driving frequency. The higher sensitivity gives the lower noise level. However, the bandwidth of the amplifier decreases as the sensitivity setting goes higher. Figure 2.4 shows the bandwidth and noise level of a current amplifier depending on the frequency and the sensitivity.

![Figure 2.4: The bandwidth(A) and noise level(B) of the current pre-amplifier SR570 in the high bandwidth mode.](image-url)
setting. Using a sensitivity as high as possible within the limit of the bandwidth is the best choice.
3. Search for 2-dimensional supersolid

3.1 Experimental details

Two experimental cells were used to measure the superfluidity of helium films. Both of them were torsional oscillators containing Vycor glass inside the torsion bob. The first cell was the torsional oscillator used in the first Kim and Chan experiment [5]. The Vycor glass disk has a diameter of 15 mm and thickness of 4 mm. The surface area of the Vycor glass was $102 \, \text{m}^2$ and open volume was $0.2 \, \text{cc}$. The second cell was designed to improve the surface area and the mass sensitivity for helium films to measure the superfluidity more accurately. Cylindrical Vycor glass tube was fit into a torsional oscillator and the both ends of the tube were glued strongly to the torsional oscillator. The Vycor glass cylinder has a outer diameter of 15 mm, inner diameter of 12.2 mm and height of 22.7 mm. The surface area of the Vycor glass was $190 \, \text{m}^2$.

3.2 Measurements in the Vycor disk cell

The period and amplitude of a empty torsional oscillator have its own temperature dependence. The period of a empty oscillator usually shows linear temperature dependence and is subtracted from the measured data for liquid and solid helium samples. Figure 3.1 shows the period and amplitude of the torsional oscillator with the Vycor glass disk. The period has almost linear temperature dependence above 200 mK and the slope is slightly changed below 200 mK. The amplitude increases and the period shows upturn below 50 mK. This abnormal behavior is known to originate from the property of beryllium copper.

Table 3.1 shows the specification of helium films adsorbed on the Vycor glass
substrate. Total 8 samples of different thickness have been prepared and measured. The coverage means the amount of adsorbed helium atoms on the substrate divided by the total surface area of the substrate. The layer was calculated based on the fact that the single layer of close packed helium atoms have 12.8 $\mu$mol/m$^2$ density. The period of the torsional oscillator was increased linearly with the adsorbed amount of helium. Samples which have thickness larger than two layer showed superfluidity of liquid helium films. Figure 3.2 ~ 3.7 show the period and amplitude data for the inert layer helium films, which have lower coverages than the film that shows KT-like superfluid transition. The period data were almost identical to the period of the empty cell. Although the periods seem to be slightly lowered at low temperatures, the quantities were too small to discriminate whether they were due to superfluidity. The period difference of $0.1 \sim 0.2$ ns was nearly the measurement limit.

For the 1.7 layer helium film, the temperature scans were done with two different oscillating amplitudes. The amount of period decrease was smaller in case of the high

![Figure 3.1: Empty cell background of the torsional oscillator that contains Vycor glass disk](image)
driving amplitude. In order to interpret this result as a suppression of superfluidity by the high oscillating velocity, more precise measurement on the empty cell period is needed. Unfortunately, the empty cell measurement was done with only one driving amplitude.

The period change for the inert layer helium films were summarized in Figure 3.8. Though the period change increases as the temperature was lowered, the amount was within the measurement error and no coverage dependent behavior was observed. The period change by loading of helium films was 458 ns per layer. The period change of 0.1 ns corresponds to approximately 0.02% of the total helium films. Although a few solid helium sample showed 0.02% NCRIF, more accurate experimental setups were needed to discriminate the superfluidity in the inert layer films.

<table>
<thead>
<tr>
<th>Sample#</th>
<th>Coverage (µmol/m²)</th>
<th>Layer</th>
<th>∆Period (ns)</th>
<th>Tc (mK)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>13.0</td>
<td>1.02</td>
<td>384.3</td>
<td>-</td>
</tr>
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<td>2</td>
<td>19.3</td>
<td>1.51</td>
<td>659.3</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>20.4</td>
<td>1.59</td>
<td>731.5</td>
<td>-</td>
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<td>2.14</td>
<td>984.1</td>
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</tr>
</tbody>
</table>

Table 3.1: Specification of helium film samples adsorbed in the Vycor disk cell
Figure 3.2: Temperature scan on the monolayer helium film adsorbed on the Vycor glass

Figure 3.3: Temperature scan on the 1.5 layer helium film adsorbed on the Vycor glass
Figure 3.4: Temperature scan on the 1.6 layer helium film adsorbed on the Vycor glass. The abnormal peak might be due to external noise or malfunctioning of instruments.

Figure 3.5: Temperature scan on the 1.7 layer helium film adsorbed on the Vycor glass
Figure 3.6: Temperature scan on the 1.7 layer helium film with different drive amplitudes

Figure 3.7: Temperature scan on the 1.8 layer helium film adsorbed on the Vycor glass
Figure 3.8: Period changes of the inert layer helium films
With the coverage higher than 24.6 $\mu$mol/m$^2$, the superfluid transition of liquid films started to appear. As increasing the coverage, the superfluid transition temperatures were increased. However, the third sound resonance in the film with 26.2 $\mu$mol/m$^2$ coverage prohibited the normal measurements. The divergence of the period and the significant amplitude suppression in Figure 3.10 are the indication of the third sound resonance in the helium film. In order to eliminate the third sound resonance, the coverage of film was reduced to 25.2 $\mu$mol/m$^2$ and the drive amplitude dependent superfluidity was investigated. Figure 3.12 shows the period data of the temperature scan with various driving amplitudes. If a portion of period drop was related with the superfluidity in the inert layer, the drive dependent suppression of the period drop would be expected. However, no recognizable suppression was observed in the measurements. Figure 3.13 summarize the measurements on helium films with coverage larger than 2 layer. The shape of the transition is that of conventional KT transition and the transition temperature increased as the thickness of the film increased. The transition temperature of each films were plotted in Figure 3.14. The inert layer coverage of 25.16 $\mu$mol/m$^2$ was calculated from the linear dependence of the transition temperatures on coverages.
Figure 3.9: Temperature scan on the 2 layer helium film adsorbed on the Vycor glass

Figure 3.10: Temperature scan on the 2.2 layer helium film adsorbed on the Vycor glass
Figure 3.11: Temperature scan on the 2.1 layer helium film adsorbed on the Vycor glass

Figure 3.12: Temperature scan on the 2.1 layer helium film with a few different drive amplitudes
Figure 3.13: Period change by the superfluid transition in helium films

Figure 3.14: Period change by the superfluid transition in helium films
3.3 Measurements in the Vycor cylinder cell

The second torsional oscillator with Vycor glass had an improved mass sensitivity compared to the first one. The period increase by loading helium films was 874 ns per layer. Table 3.2 shows the specification of helium films adsorbed on the Vycor glass substrate. Total 9 samples have been prepared with increasing the thickness of films. Same as in the first Vycor cell, the period drop was investigated in the inert layer helium films and the drive amplitude dependence was measured in the superfluid films. The temperature scans on the empty cell were done with two different driving amplitudes. The low temperature value of the period showed a little difference in two cases. Figure 3.16 shows the temperature dependence of the empty cell. Deviation from the linear temperature dependence of periods appeared below 100 mK. The relatively large period upturn disrupted the accurate analysis on the data since the

Figure 3.15: 3D drawing of the torsional oscillator containing a cylindrical Vycor glass tube

amplitudes. The low temperature value of the period showed a little difference in two cases. Figure 3.16 shows the temperature dependence of the empty cell. Deviation from the linear temperature dependence of periods appeared below 100 mK. The relatively large period upturn disrupted the accurate analysis on the data since the
period changes of the inert layer films were very small or absent. Moreover, the temperature range of the period upturn overlap with the temperature at which we expect the superfluidity.

Figure 3.17 ~ 3.22 show the period and amplitude data for the inert layer helium films, which have lower coverages than the film that shows superfluid transition. And the period change from the value at 300 mK for all the inert layer samples were plotted in Figure 3.23. No apparent period drop compared to the empty cell background was observed in all the measured samples.

<table>
<thead>
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<th>Sample#</th>
<th>Adsorbed He (µmol)</th>
<th>Coverage (µmol/m²)</th>
<th>ΔPeriod (ns)</th>
<th>Layer</th>
<th>Tc (mK)</th>
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Table 3.2: Specification of helium film samples adsorbed in the Vycor cylinder cell
Figure 3.16: Empty cell background of the torsional oscillator which contains Vycor glass cylinder

Figure 3.17: Temperature scan on the helium film of 9.5 µmol/m² adsorbed on the Vycor glass cylinder
Figure 3.18: Temperature scan on the helium film of 13.1 $\mu$mol/m$^2$ with a few different driving amplitudes

Figure 3.19: Temperature scan on the helium film of 14.2 $\mu$mol/m$^2$ adsorbed on the Vycor glass cylinder
Figure 3.20: Temperature scan on the helium film of 16.6 µmol/m² adsorbed on the Vycor glass cylinder

Figure 3.21: Temperature scan on the helium film of 21.3 µmol/m² adsorbed on the Vycor glass cylinder
Figure 3.22: Temperature scan on the helium film of 23.1 $\mu$mol/m$^2$ adsorbed on the Vycor glass cylinder

Figure 3.23: Period change of the inert layer helium films
Figure 3.24 ∼ 3.26 show the superfluid transition of helium films that have thickness larger than two monolayer. In all the superfluid transition, the dissipation peak was not observed. This is consistent with the previous results in Vycor glass [32, 45]. Temperature dependences were measured with three different drive amplitudes for each films. Contrary to expectations, the measured period data were almost exactly same for all different drive amplitudes. We tentatively concluded that the superfluidity does not exist in the inert layer helium films adsorbed on Vycor glass.

Figure 3.24: Temperature scan on the helium film of 26.0 µmol/m² adsorbed on the Vycor glass cylinder
Figure 3.25: Temperature scan on the helium film of 27.8 $\mu$mol/m$^2$ adsorbed on the Vycor glass cylinder

Figure 3.26: Temperature scan on the helium film of 29.6 $\mu$mol/m$^2$ adsorbed on the Vycor glass cylinder
3.4 2-dimensional helium film on the hydrogen substrate

In order to weaken the van der Waals interaction between the helium film and the substrate, the Vycor glass was preplated with hydrogen molecules. Hydrogen was known to have a small potential well depth compared to other materials. The inert layer of helium film on the hydrogen substrate is only 0.5 monolayer.

Total 3 layers of hydrogen was adsorbed on the Vycor and the background scan was performed. In Figure 3.27, the period change of TO with hydrogen is almost similar to that of the empty cell. After the background scan, a helium film sample was prepared by adsorbing 0.4 layer of helium atoms. Figure 3.28 shows the resonant period change for the sample. Below 300 mK, period drop with broad onset temperature was observed. Total period change is approximately 5 ns, which corresponds to 1.9% of the total helium film. Whether the dissipation at 200 mK is real signal or just

![Graph showing period change with temperature](image)

Figure 3.27: Empty cell background of the bare Vycor glass and the hydrogen plated Vycor glass.
Figure 3.28: Temperature scan on the helium film of 0.4 layer adsorbed on the hydrogen substrate.

Figure 3.29: Temperature scan on the helium film adsorbed on the hydrogen substrate with different driving amplitudes.
a fluke is not clear. Temperature dependences are tested with two more driving amplitudes. Unlike solid helium, the period change was not suppressed with high drive amplitudes although the velocity of the TO was 44 mm/s. The period change can be due to conventional 2-dimensional superfluid films which might have inhomogeneous film thickness according to narrow and disordered pore structure. The broad onset could be caused by the inhomogeneous film thickness or inhomogeneous tortuosity.

3.5 Conclusion

Superfluidity in the inert layer helium films adsorbed on Vycor glass was thoroughly investigated. However, no signature of NCRI in the inert layer films was observed within our experimental limit. The helium films on Vycor glass are known to be amorphous. The inert layer helium films in amorphous structure may not show superfluidity or the interaction between helium and Vycor might be strong enough to localize all the helium atoms. More studies on the films with hydrogen or other substrates are demanding. Figure 3.30 shows the result of another experiment conducted by S. Kwon, who is my lab colleague. He adsorbed 1.1 layer of helium film on the hydrogen preplated Vycor glass. Besides the conventional KT transition and dissipation peak, he found an amplitude dependent period drop and extra dissipation. This result seems to be related with the inert layer film and the research is still in progress.
Figure 3.30: Temperature dependence of the periods and amplitudes for the 1.1 layer helium film adsorbed on hydrogen substrate, which was measured by S. Kwon.
4. Solid helium in porous gold

The NCRI of solid helium inside porous material is important since the complicated porous structure prohibits any elastic motion of line defects larger than a characteristic pore diameter. The non-classical response of solid $^4\text{He}$ confined in porous gold was investigated in the torsional oscillator. Regarding the observed NCRI in porous gold, two features were investigated. First, the solid helium enclosed in porous gold is expected to have high density of disorders since the narrow and complex pore structure gives the structural confinement. Additional disorders can be introduced by a quench-cooling technique during solidification. Thus, NCRI in a sample with structurally induced disorders can be compared to a case with quenched disorders. Second, the dynamic response of the torsional oscillator containing a highly disordered solid was investigated.

4.1 Experimental details

A torsional oscillator which contains porous gold was prepared and the solid helium was grown inside the porous gold. The resonant frequency of the torsional oscillator was approximately 948 Hz and the mechanical $Q$ factor measured by a conventional ring-down time constant was $3 \times 10^5$. The physical dimension of the porous gold disk was 10 mm in diameter and 0.6 mm in height. The disk had a surface area of 0.66 m$^2$ and porosity of 67% according to oxygen vapor isotherm measurements. The pore diameter was about 180 nm assuming mono-disperse cylindrical pores. The empty space was deliberately removed by filling this space with Stycast 2850FT epoxy.

The solid helium samples were grown by the blocked capillary method. It was possible to finish the entire freezing process in few seconds via quench-cooling. During
the solidification, a minute amount of latent heat (0.01 µJ) was generated due to the small open volume (0.032 cc) to helium and was drained efficiently via the complicated network of gold strands. The pressure in the torsion cell was monitored using a resistive strain gauge that measures the deflection of the torsion cell due to high pressure with a resolution of approximately 0.5 bar. The dynamic response of the TO containing a highly disordered solid was also measured to understand the function of low temperature excitations.

![Torsional oscillator containing porous gold](image)

**Figure 4.1: Torsional oscillator containing porous gold**

### 4.2 NCRI of the solid helium in porous gold

Figure 4.2 shows the temperature scan on the first sample that has 25 bar pressure. Below 0.2 K the resonant period reveals the onset of NCRI that shows qualitatively identical temperature and velocity dependence to those of previous measurements.

The mass loading by the solid helium was 163 ns and the period drop at low temperatures was 8.7 ns, which was 5.3% in NCRIF.

The presence of porous matrix in a TO also provides a simple reasoning to test
Figure 4.2: The period and amplitude of the torsional oscillator with solid helium inside the porous gold

the feasibility of the proposed non-supersolid scenarios. For instance, NCRI in porous media cannot be understood in terms of a stiffening of solid helium induced by the impurity pinning of the dislocation network. Given that the length scale of a free vibrating dislocation cannot exceed the pore diameter, the characteristic dislocation length in porous gold can be expected to be much shorter than that in bulk helium. Thus, the increase in the shear modulus due to pinning dislocation networks in porous gold is expected at substantially lower temperatures (or a higher 3He impurity concentration) compared to the bulk value. Similarly, glassy behavior of solid helium as a possible origin of the period reduction is strongly limited as well. The motion of liquid or solid confined in porous media is suppressed by the complicated multiply connected tortuous geometry. With oscillation frequency less than a characteristic frequency of $\omega = 2\eta/\rho\delta^2$, a viscous liquid is immovable and localized to the pore walls. Here, $\eta$ is viscosity, $\rho$ is density, and $\delta$ is viscosity penetration depth and should be less than the pore diameter. If the frequency is smaller than a characteristic frequency, no
decoupling of liquid is expected unless superfluid is present.

4.3 The effect of quench cooling on NCRI

Figure 4.3 shows the NCRI fraction and dissipation of the TO enclosing solid $^4$He sample at 48 bar as a function of temperature. The large NCRI of about 7% was observed at an oscillating speed of about 200 $\mu$m/s. For each set of a specific rim speed $v_{\text{max}}$, a broad minimum in the amplitude of the oscillation, a signature of dissipation, is detected at the temperature where the period shift shows the maximum variation. Solid helium was subsequently grown rather slowly so that the dwelling time on the melting curve was about 90 min. The final pressure of the sample was tuned to be roughly equal to the pressure of the highly disordered sample. The onset was quite similar to the quenched sample but a smaller magnitude of NCRI (about 1.4%) with broader dissipation was observed.

![Figure 4.3: Non-classical rotational inertia fraction and dissipation of the quench-cooled solid $^4$He in porous gold.](image)

A large NCRIF was also reported in highly disordered solids grown by quench-
cooling methods in thin annular channels [13, 52]. The NCRIF was enhanced further by confining a solid helium sample into narrower annular channel with higher surface to volume ratio. However, a small NCRIF of less than a few % has also been obtained in even more severely restrained solid samples [5, 53], deviated from the reported confinement effect. This is probably because the large thermal mass of helium combined with poor thermal conductivity of glass likely reduces the solidification speed and, consequently, hinders the appearance of the quench effects. The 7% of large NCRIF was obtained since the quenched-cooling was possible due to small sample volume. These results support the explanation that a large NCRI primarily originates from the quench-cooled frozen disorders [54].

![Non-classical rotational inertia fraction and dissipation of the slowly grown solid ⁴He in porous gold.](image)

Figure 4.4: Non-classical rotational inertia fraction and dissipation of the slowly grown solid ⁴He in porous gold.
4.4 Relaxation dynamics of the solid helium

The relaxation dynamics of the resonant period was investigated when the driving voltage changes discretely at various temperatures. A solid sample was first cooled to target temperatures with a minimum drive. The driving voltage was then increased in sequential steps and then decreased in the reverse order. Below the onset of NCRI, extended relaxation in the TO period appears in the drive-up scans, while fast relaxation occurs in drive-down scans.

The discrepancy in the relaxation leads to hysteretic behavior during a drive sweep at low temperatures. The hysteresis is more pronounced below 60 mK, as the relaxation is lengthened progressively with decreasing temperature. Figure 4.5 shows the period and amplitude of TO with the drive sweep. The relaxation was so long that the equilibrium could not be reached within a few hours. The apparent drive hysteresis and long relaxation were reported with robust NCRI upon increasing drive, while NCRI was suppressed when the drive decreased [27, 9]. The authors discussed the

![Figure 4.5: The relaxation of the resonant period and velocity of the torsional oscillator according to driving volatage change.](image-url)
hysteresis in connection with the severe pinning of vortices or dislocation network. The severe pinning at low temperatures was only observed at the minimum driving voltage in the current experiment. The discrepancy is possibly related to the large NCRIF in our measurements.

![Graph showing relaxation of NCRIF and velocity of the torsional oscillator](image)

Figure 4.6: The relaxation of the NCRI and velocity of the torsional oscillator by increasing drive voltage at 20 mK.

Figure 4.10 shows time evolution data of the resonant period change during the drive-up scans. For a very short time scale, the relaxation of the TO period shows a simple exponential form that is solely attributed to the high mechanical $Q$ factor regardless of the drive-up or drive-down scans. An exponential evolution with a time constant larger than the mechanical relaxation of the TO arises in an intermediate time scale. The relaxation is further extended as the temperature decreases. Finally, logarithmic relaxation appears in a long time scale at low temperatures.

The origin of logarithmic time evolution is not clearly understood, but it has been found in various relaxation phenomena. Logarithmic relaxation was observed where disorders function as energy barriers and pin down thermal excitations [55, 56].
The pinned excitations can overcome the barriers with thermal activation assisted by the driving force such as the Magnus force and the Lorentz force in this framework. The difference between the average pinning potential barrier and the driving
kinetic energy ($\Delta U$) gives an empirical unpinning probability that is proportional to $\exp(-\Delta U/k_B T)$. During the drive-up scan, weakly trapped excitations can be released first, exhibiting additional dissipation and a period increase. This process emerges with an exponential relaxation in which a temperature-dependent characteristic time constant (i.e. the relaxation rate) is connected to the unpinning probability. As shown in Figure 4.10(b), the extended relaxation rate of the unpinning process is proportional to the inverse temperature. The relaxation rate shows a weak dependence on a stress field; higher drive agitation leads to faster relaxation as expected. Once most of the weakly localized excitations are removed, the unpinning probability decreases substantially. In this limit, the unpinning of strongly pinned excitations results in logarithmic time dependence at low temperatures, which is consistent with the observed relaxation at the long time scale.

The motion of the liquid or solid confined in porous media is limited by the complicated multiply connected tortuous geometry. Therefore, the extended relaxation in porous gold torsional oscillator should have different origin rather than glassy solid-
Figure 4.10: (A) The normalized NCRI when the drive voltage increases from 2 mV to 5 mV is plotted as a function of the elapsed time. The inset shows logarithmic time evolution (20 mK) at a long time scale. (B) The relaxation in an intermediate time scale as a function of the inverse temperature for various drive voltages. Induced dissipation. The extended relaxation upon sudden increase in the driving voltage is likely to be related with the low temperature excitation in solid helium.
4.5 Conclusions

Although the motion of a dislocation or a glassy solid is restricted in the entangled narrow pores, a large amount of NCRI was observed. The magnitude of the non-classical response of solid helium confined in porous gold is strongly dependent on the cooling history of the sample. Substantial enhancement of NCRI in a quench-cooled sample and the absence of the confinement effect in porous glasses suggest that the cooling history play a more significant role in producing large NCRI compared to that by geometrical confinement. The extended relaxation behavior of the resonant period was also observed in a severely constrained solid helium. The long relaxation time is probably not caused by the motion of an elastic solid, but it is possibly connected to the pinning of low-energy excitations at low temperatures.
5. Simultaneous measurement of the shear modulus and NCRI of solid helium

From what have been observed in experiments so far, shear modulus seems closely related to NCRI but the exact mechanism is unclear yet. In order to investigate the relation between NCRI and shear modulus further, we simultaneously measured the both quantities in a single experimental cell. The basic concept was measuring shear modulus inside a torsional oscillator.

5.1 Experimental details

The schematic of the torsional oscillator cell is shown in Figure 5.2. The inner structures were inserted to make an annular channel and a center channel. The physical dimension of entire channel is 400 µm in width and 11 mm in height. The outer diameter of the annular channel is 16.1 mm. The piezoelectric transducers are located in the center of the TO cell. The piezoelectric shear transducers are epoxied onto the inner structures that are rigidly mounted onto the body of the TO. The body of the TO, including the torsional rod, are made out of a single piece of beryllium-copper. The outer structure, a hurricane-shaped Al cap, is designed to increase the rigidity of the TO cell. A fill line, 0.3 mm OD with 0.05 mm thick CuNi capillary, is placed on top of the TO cell. The resonant frequency of the TO was approximately 911 Hz and Q factor was $6 \times 10^5$ below 1 K.

Total 11 samples were prepared by the blocked capillary method in a standardized manner. The temperature of the mixing chamber was increased above 3 K and the sample cell was pressurized with 0.3 ppm commercial $^4$He to a target pressure. Then the dilution refrigerator was cooled by pumping out the mixture at still without
operating 1 K pot. The usual cooldown time to 1.4 K was 2~3 hour. 1 K pot was operated when the cell temperature was lower than 1.4 K and pumping on the still is stopped. Samples were annealed for more than 3 hours at 0.1 K below the temperature at which the solidification completed. Although the locations where the NCRI and the shear modulus are different in the cell, the overall crystal quality is not expected to be different based on three facts. First, the samples were grown along the same thermodynamic path. And the $^3$He concentration over whole sample would be same because the diffusion coefficient of $^3$He atoms in $^4$He solid is very large. Finally, in order to have similar S/V ratio, the whole channel width was designed as 400 µm.

Various measurement conditions were tested in order to avoid the interference between the two measurements. The two measurements were independent only if the driving amplitudes were not extremely high. Thus, the usual temperature dependences of NCRI and shear modulus could be measured simultaneously and comparing the relaxation dynamics between two by a temperature change was possible. All the samples were characterized by the temperature scan with a same condition in which the velocity of the torsional oscillator was approximately 10 µm/s and the shear transducer was driven with 1 kHz frequency and a strain of $10^{-8}$. 
Figure 5.1: 3D drawing of the experimental cell

Figure 5.2: Schematic diagram of the experimental cell
5.2 Temperature Dependence

5.2.1 Temperature scans

Figure 5.3 represents the result of a typical temperature scan on a 34 bar sample. NCRI and the shear modulus increase were observed in similar temperature range. However, the temperature ranges in which the anomalies occurred did not coincide exactly and the difference varied in each samples. Figure 5.4 summarize the characteristic temperatures of the whole measured samples. Txx% means the temperature where the transition reaches the given fraction from the high temperature value. The discrepancy in the temperature dependence may be due to local differences in qualities and orientations of helium crystals.

Table 5.1 summarize the characteristics of each samples that were determined by the standardized temperature scans. For the hole 11 samples, the average NCRI is 1.65% and the average shear modulus increase is 27%. The average T_{50%} of NCRI is 75.9 mK and that of shear modulus increase is 68.6 mK.

5.2.2 Frequency dependence of the shear modulus measurement

Although the torsional oscillator has only one operating frequency, the shear modulus can be measured with various driving frequency. Figure 5.5 shows the result of temperature scans with different frequency of the shear transducer on the same sample as Figure 5.3. The temperature range lowered as the driving frequency decreased from 1 kHz to 10 Hz. However the transition didn’t go to higher temperature with increasing drive frequency higher than 1 kHz.

The frequency dependence might be related with relaxation time, \( \tau \), of the modulus. According to Syshchenko and coworkers [57], the transition from unrelaxed modulus to relaxed modulus occurs at temperature where \( \omega \tau = 1 \). Since the relaxation time increases as temperature decreases, low frequency is needed to probe the
slow dynamics.

Figure 5.3: Temperature dependence of the shear modulus and the NCRI of a 34 bar solid helium.
<table>
<thead>
<tr>
<th>#</th>
<th>P load (bar)</th>
<th>Mass (ns)</th>
<th>NCRI (%)</th>
<th>$\Delta Q^{-1}$ $\times 10^{-6}$</th>
<th>$T_{50%}$ NCRI (mK)</th>
<th>SM 0.5 K ($\frac{pA}{HzV}$) (%)</th>
<th>$\Delta$ SM (%)</th>
<th>$\Delta\phi$ (deg.)</th>
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Table 5.1: Specification of solid helium samples for the simultaneous measurement
Figure 5.4: Transition temperature of the shear modulus and the NCRI
Figure 5.5: Temperature dependence of the shear modulus with various driving frequency and the NCRI of solid helium (upper). The lower part shows the dissipation of the two measurements.
5.3 Drive amplitude Dependence

The drive dependence of NCRI has been discussed from the first observation of NCRI. NCRI was suppressed with high driving amplitude of the torsional oscillator. The critical velocity with which the suppression started was approximately $10 \mu m/s$ and varied with each experimental setup. In the following studies, drive dependent phenomena was known to be hysteretic at low temperatures [9, 27]. In the Day and Beamish experiment [23], the shear modulus increase also showed drive dependent behavior. The critical drive amplitude in the experiment was $2.2 \times 10^{-8}$ in strain and $0.3$ Pa in stress. The corresponding velocity for the strain, $\epsilon = 2.2 \times 10^{-8}$, at 2000 Hz was $50$ nm/s, which was much smaller than the critical velocity in torsional oscillator experiments. The hysteretic behavior similar to the torsional oscillator experiments was also observed in the shear modulus measurement.

5.3.1 Drive dependence of the shear modulus

![Figure 5.6: The shear modulus with the drive-down and drive-up scans at 20 mK](image)
Figure 5.6 shows the result of the drive sweeps at 20 mK. The sample was cooled with the highest driving amplitude from the temperature higher than 300 mK. The highest drive was with the driving voltage of 2 V, which corresponds to the strain of $5 \times 10^{-7}$ and the stress of 7.3 Pa. With the highest drive, the shear modulus stayed almost unchanged even though the samples were cool down to 20 mK. Then the shear modulus was measured with decreasing the drive amplitude. If the drive was reduced at low temperature, the shear modulus was increased. The drive amplitude was reduced logarithmically to the lowest value with which the strain was approximately $1 \times 10^{-9}$ and the stress was 0.01 Pa. Then the drive amplitude was increased again to the highest value. The shear modulus did not reduced again with the high drive amplitudes at low temperatures. The shear modulus according to the drive down and up scan showed hysteresis.

The whole drive down and up scans were repeated with a few different driving frequencies. The critical drive amplitudes where the shear modulus increased to the highest values were independent of driving frequencies. This result means that the critical stress or strain, not velocity, is meaningful value in the shear modulus measurement [58]. In case of the 10 kHz driving frequency, too high driving amplitudes induced the heating of samples. The data points were omitted in the graph for a few high drive amplitudes with which the temperature differences between the sample and the mixing chamber were larger than 10 mK.

### 5.3.2 Drive dependence of NCRI and shear modulus

The drive down and up scans were also performed in the torsional oscillator measurements. Similar hysteretic behaviors were observed in NCRIF at low temperatures, but the critical stresses of two measurements were different. Figure 5.7 displays the change of the NCRI and the shear modulus as a function of stress by decreasing and increasing the drive after cooling with a high drive amplitude. NCRI had a two orders
of magnitude smaller critical stress. The critical stress for NCRI was 0.002 Pa while that for the shear modulus increase was approximately 0.4 Pa. With the stress level between the two critical stresses, the NCRI showed suppression although the shear modulus is in increased state. Increased shear modulus means all the dislocations are pinned to impurities. However, the suppression of NCRI with the pinned dislocations suggest that another underlying mechanism is relevant to NCRI.

### 5.3.3 Interference between the two measurements

Measuring the two properties simultaneously, mutual disturbance had been investigated carefully. NCRI was not effected by the shear modulus measurements regardless of whatever the conditions of the PZT transducers are. The shear modulus was also not effected by the motion of the torsional oscillator in low amplitudes. However, the shear modulus increase was prohibited by extremely high drive amplitudes of the torsional oscillator. Figure 5.8 shows the NCRI and shear modulus with various drive...
amplitudes of the torsional oscillator. The shear modulus was measured with a low, 0.18 Pa, drive amplitude, which was smaller than the critical drive amplitude. As increasing the drive of the torsional oscillator, the shear modulus anomaly start to be suppressed with drives higher than 600 μm/s, with which the NCRI was completely suppressed. This result supports the speculation that the NCRI is originated from a different low-temperature excitation which can be suppressed much easily.
5.4 Thermally assisted breakaway of dislocations

5.4.1 Teutonico-Granato-Lücke Theory

The shear modulus increase can be explained by the pinning of the dislocation lines on impurities. Since the original “vibrating string” model only considers the unpinning of dislocations by stress field, it is strictly a zero temperature theory. The theory of thermally assisted unpinning of dislocation can extend our understanding on several characteristic aspects of the shear modulus measurements [59, 60, 61]. The interaction energy between a dislocation line and a impurity pinning point is given by the Cottrell potential and the relevant Cottrell force is a attractive force [62]. If we assume a system that consists of a dislocation line and a impurity atom at the center of the line as in Figure 5.9, the potential energy of the system is composed of three terms; the pin-dislocation interaction energy, a dislocation line energy and the strain energy [59]. Whereas the strain energy by applied stress favors the unpinned state, the pin-dislocation interaction energy and the line energy due to the tension of the dislocation line lead the system to the pinned state. The total potential energy of the system is given like Figure 5.10 with two minima as a function of the distance between the impurity and the dislocation line; a pinned state at a short distance and an unpinned state at a longer distance. The transition between the two minimum states can be assisted by thermal energy. Because the potential energy is strongly dependent on

![Figure 5.9: Dislocation double loop pinned at the middle](image-url)

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the applied stress, the entire characteristic shape of the potential energy changes dramatically due to the applied stress. At low stress, only one energy minimum appears as a strongly pinned state, and at high stresses only the unpinned state is energetically stable. In the intermediate range of stresses, two energy minima appeared. The heights of the potential barriers are given as below [60].

\[
U_1 = r_1 U_0 (1 - \sigma / \sigma_1)^2
\]  
(5.1)

\[
U_2 = r_2 U_0 (\sigma / \sigma_2 - 1)^2
\]  
(5.2)

where \( \sigma_1 \) is the mechanical breakaway stress, above which the first minimum disappears and \( \sigma_2 \) is the mechanical repinning stress, below which the second minimum disappears. \( U_0 \) is the height of the Cottrell potential. \( r_1 \) and \( r_2 \) are the constants which are related with material properties. Then the probability that a pinned dislocation will be unpinned thermally is

\[
Q_1 = \nu_1 \exp (-U_1 / kT)
\]  
(5.3)

The probability that an unpinned dislocation will be pinned thermally is

\[
Q_2 = \nu_2 \exp (-U_2 / kT)
\]  
(5.4)
where \( \nu_1, \nu_2 \) are frequency factors \([63]\). The status of breakaway is described by the fraction \( f \) of dislocation that is in second minimum and its rate equation is

\[
\frac{df}{dt} = (1 - f) Q_1 - f Q_2
\]

(5.5)

with \( P = Q_1 + Q_2 \)

\[
\frac{df}{dt} + P f = Q_1
\]

(5.6)

For a constant stress \( \sigma \), we can obtain a solution.

\[
f(t) = \left( \frac{Q_1}{P} \right) - \left( \frac{Q_1}{P} - f(0) \right) e^{-Pt}
\]

(5.7)

The solution is time dependent and the relaxation time is \( 1/P \). The equilibrium value at \( t = \infty \) is

\[
f_\infty = \frac{Q_1}{P} = \frac{1}{1 + (\nu_1/\nu_2) \exp \left[ \frac{(U_1 - U_2)}{kT} \right]}
\]

(5.8)

Meanwhile, the shear strain created by the motion of dislocation is given by

\[
\epsilon_{\text{dis}} = R\Lambda b \left[ \bar{y}_{\text{unpin}} f + \bar{y}_{\text{pin}} (1 - f) \right]
\]

(5.9)

where \( \bar{y}_{\text{unpin}} \) and \( \bar{y}_{\text{pin}} \) are the average displacements of the dislocation loop in the unpinned and pinned state. In case of the double loop dislocation, they are given as

\[
\bar{y}_{\text{pin}} = \frac{\bar{y}_{\text{unpin}}}{4} = \frac{L^2 \sigma}{24 b \mu}
\]

(5.10)

Then the strain is

\[
\epsilon_{\text{dis}} \approx \left( R\Lambda L^2 / 24 \right) (1 + 3 f) \sigma / \mu
\]

(5.11)

In the low frequency static limit, we have similar form with Eq.1.10 for the shear modulus change by the motion of dislocations

\[
\frac{\Delta \epsilon}{\epsilon} = \frac{\Delta \mu}{\mu} = \frac{(1 + 3 f_\infty)}{24} R\Lambda L^2
\]

(5.12)

In previous arguments, the temperature dependence of the shear modulus change was explained as a shortening of loop length by condensation of \( ^3\text{He} \) impurities to
the dislocations. In this model, the temperature dependence is given by $f$. But the dynamics is more complicated since the $f$ is also stress dependent. Another difference with the previous arguments is that the shear modulus change due to dislocations can exist even at zero temperature. This is because this model considers the dislocation strain in its pinned state. This suggests that the configuration of dislocations could influence the intrinsic shear modulus at low temperatures.

### 5.4.2 Drive dependent hysteresis of shear modulus

The hysteresis of the shear modulus in the drive sweep in Figure 5.6 can be understood in the framework of the thermally assisted breakaway of dislocations. If the sample is driven with a high applied stress, $U_2$ is large and $U_1$ is small or zero. Since the unpinned states are more stable than the pinned states, the dislocations can move unpinned from the impurities and the shear modulus does not increase. The shear modulus can remain small by the high stresses even at low temperatures by two reasons. First, the repinning activation energy $U_2$ is larger than the unpinning activation energy $U_1$. We need to notice that the pinning as well as the unpinning requires activation by thermal energy. As the higher stress is applied, the transitions from unpinned states to pinned states would be more difficult at low temperatures. One more reason that dislocations can be in the unpinned states at low temperature is the long relaxation time. The relaxation time of the thermal breakaway process in the pin-dislocation system is given by $1/P = (\nu_1 e^{-U_1/kT} + \nu_2 e^{-U_2/kT})^{-1}$ in Eq. 5.7. It is temperature dependent and increases substantially at low temperatures. Since the relaxation time would be larger than the time scale of the oscillating stress at low temperatures, the transitions between unpinned states and pinned states are not favorable in the short time scale. By Oleksandr et al. the shear modulus stiffening was discussed in terms of the relaxation process. They argued that the relaxation time $\tau$ in the thermally activated Debye process increases as the temperature decreases and
a crossover occurs where $2\pi f \tau = 1$, in which $f$ is the probing frequency [57].

As the stress decreases to zero in Figure 5.11, $U_2$ decreases to zero and $U_1$ increases. Since no unpinned states can exist due to the negligible repinning activation energy, the system progressively shifts to the pinned state. Conversely, if the stress is increased again at low temperatures, $U_2$ increases again and the pinned states become metastable states. However, the system cannot easily transit to unpinned state because the thermal energy is insufficient to overcome the potential barrier $U_1$ at low temperatures. Either increasing the temperature where the thermal energy is comparable with $U_1$ or increasing the stress with which $U_1$ is small enough can unpin the dislocations and decrease the shear modulus. At high temperatures above 60 mK, hysteresis is not observed since the sufficient thermal energy allows the dislocations to transfer between pinned and unpinned states.

Figure 5.11: Shear modulus changes by drive-down and drive-up scans at various temperatures
Figure 5.12: Temperature and drive stress dependence of shear modulus obtained by two different methods. The solid line was measured by temperature scans with constant stresses (CS). The solid circles with dotted lines were taken from the drive scans at constant temperatures (CT) in Figure 5.11.
5.4.3 Stress-Temperature phase diagram of shear modulus

Shear modulus is dependent on both the temperature and the drive stress. And it is also dependent on the history of the stress and temperature. For a specific set of a stress and temperature, the shear modulus values can differ depending on the path with which the stress and temperature are acquired. This history dependent behavior is obvious in Figure 5.12. The solid lines in the graph show the results of temperature scans with constant stresses (CS). After setting the driving stress at 500 mK, the solid helium sample was cooled down to the base temperature. Then the shear modulus has a finite value at low temperatures depending on the amplitude of the driving stress. After waiting about an hour at low temperatures, the shear modulus was measured with warming scan. We will refer to the results as the CS shear modulus.

The solid circles with dotted line in the graph were obtained by reconstructing the results of the drive-up scans at constant temperatures (CT) in Figure 5.11. The data that correspond to each drive stresses and temperatures were collected and plotted in the temperature-shear modulus plane. These traces are labeled as the CT shear modulus. The CS and CT shear modulus are almost identical for the low drive stresses. However, the high stress results show discrepancies in two cases. Below a specific temperature, the CT shear modulus is always larger than the CS shear modulus. The specific temperature below which the difference start to appear is called the repinning temperature $T_r$ since it might be related with the repinning activation energy $U_2$.

Above $T_r$, both unpinning and repinning are possible assisted by the relatively high thermal energy compared to $U_1$ and $U_2$. Since the transitions in both directions are possible, the equilibrium ratio between the two states would be always same regardless of the history. However, the transition from the unpinned state to pinned state is difficult below $T_r$ because the thermal energy is insufficient to overcome $U_2$. Since the transition from unpinned state to pinned state is not favorable, the configuration
of the dislocations are affected by the history. The observation that $T_r$ increases with increasing drive stress is consistent with that $U_2$ increases as drive stress increases.

The characteristic temperature that should be related with the unpinning activation energy $U_1$ is the pinning temperature $T_p$. $T_p$ is the temperature above which the unpinning begins and below which all the dislocations are pinned. The $T_p$ was picked where the CT shear modulus start to differ from the shear modulus with the lowest stress in Figure 5.12. As $T_p$ was lower than the base temperature for the stress higher than 0.9 Pa, the low T data were extrapolated to estimate the temperatures. The fact that the $T_p$ decreases with higher stress agrees with that $U_1$ is decreased as the stress increases.

Based on three characteristic temperatures, the stress-temperature phase diagram was constructed in Figure 5.13. The onset temperature $T_o$ is the temperature below which shear modulus starts to increase. In the ‘pinned dislocations’ region below $T_p$, 

![Figure 5.13: Stress-Temperature phase diagram of the shear modulus change](image-url)
all the dislocations are strongly pinned because thermal energy is smaller than $U_1$ and $U_2$ is almost zero due to the small stress. In the ‘fully unpinned dislocations’ region above $T_o$, dislocations move freely unpinned from the impurities because the thermal energy overwhelm the potential barriers. In between the two regions, the ‘partially unpinned dislocations’ region, some of dislocations are unpinned and the others are in pinned states. The fraction of the pinned and unpinned dislocations would give a corresponding shear modulus value.

The ‘partially unpinned dislocations’ region can be divided into two regions by the characteristic temperature $T_r$. Owing to the reason described before, the shear modulus is independent of the history of the stress and temperature in the ‘repinning allowed’ region and is dependent on the history in the ‘repinning forbidden’ region. If a specific point in the repinning forbidden region is reached with the constant stress (CS) procedure, the maximum amount of dislocation would remain in unpinned states since all the dislocations were unpinned initially. If the point is reached with the constant temperature (CT) procedure, a lot of dislocation would remain in the pinned states since the most of dislocations were in pinned states initially. From this discrepancy, we can understand why the CT shear modulus is always larger than the CS shear modulus value.

Another distinct feature in the ‘repinning forbidden’ region is the slow dynamics. With increasing drive stresses, the shear modulus changes very slowly in the low temperature and high stress region when it equilibrates to a new value. In the ‘repinning allowed’ region with high temperature and low stress, the response to the drive change is very fast. Although the time dependence of the shear modulus change is even logarithmic in the ‘repinning forbidden’ region, fitting with exponentially decaying function is enough to reveal the difference between the two regions. Figure 5.14 shows the equilibrium time constants of the shear modulus changes for the drive-up scans in Figure 5.11. The long time constants in the ‘repinning forbidden’ region can
Figure 5.14: Equilibrium time constants of the shear modulus changes during drive-up scans in Figure 5.11

be due to the creep to unpinned states from pinned states. The pinned state is the metastable state since $U_2$ is larger than $U_1$ in case of the high stress.

I should address that all the crossovers in the phase diagram are in fact not real phase transition since we understand them as thermally assisted mechanical processes in pin-dislocation systems. The dynamics in real crystal would be more complicated because the impurity pinning point and dislocations would have wide range in their distributions. The dislocations with two or more pinning points have been discussed in detail [64, 65, 60]. The basic features are similar to that of the single pin-dislocation although more metastable states exist in the multiple pin system.
5.5 The effect of the shear modulus increase on the torsional oscillator

If the NCRI is just a consequence of the shear modulus increase, the resonance frequency shift $\delta f$ of the torsional oscillator and the shear modulus change $\delta \mu$ should have a almost linear relation as mentioned earlier. The experimental cell was simulated using the finite element method (FEM) to determine how the change of the shear modulus of solid helium influences to the resonance frequency of the TO. Initially, the eigen frequencies of the empty cell and of the cell that contains the solid helium with a typical shear modulus of $1.5 \times 10^7$ Pa were calculated. The change of eigen frequency due to the change of the shear modulus is calculated. As a result, $\delta f/f$ and $\delta \mu/\mu$ exhibit a nearly linear relationship within the range of our concern and the proportional constants are equal to $4.6 \times 10^{-6}$. According to the simulation, an only 0.056% increase in NCRI is expected with 27% increase of shear modulus. This is quite small compared to the average NCRI value of 1.65%. This result confirms that the shear modulus increase alone cannot explain the NCRI and the underlying principle of NCRI can be different from the dislocation pinning.
Figure 5.15: Displacement of the experimental cell in torsional resonance mode calculated from the FEM method.

Figure 5.16: The mimicked NCRIF by the shear modulus change of the solid helium in the TO, which is calculated from the FEM method.
5.6 Correlation between NCRI and shear modulus

For the measured quantities for 11 samples including Table 5.1, dependencies between the quantities were investigated. The relation between the magnitude of NCRI and the shear modulus increase got most attention. However, the NCRIF and the shear modulus increase in each samples showed no correlation, as shown in Figure 5.17. The absence of direct correlation between the NCRI and the shear modulus increase may suggest that the microscopic origins of the two phenomena are not compatible.

Figure 5.17: Correlation between the NCRIF and the shear modulus increase
In order to find a meaningful relation between NCRI and the shear modulus, correlation coefficients between all the characterized quantity in samples were calculated. Spearman’s rho (Eq. 5.13) is a non-parametric measure of statistical dependence between two variables. It assesses how well the relationship between two variables can be described using a monotonic function. The \(n\) raw scores \(X_i, Y_i\) are converted to ranks \(x_i, y_i\) and the differences \(d_i = x_i - y_i\) between the ranks of each observation on the two variables are calculated.

\[
\rho = 1 - \frac{6 \sum d_i^2}{n(n^2 - 1)} \tag{5.13}
\]

If tied ranks exist, Pearson’s correlation coefficient between ranks should be used for the calculation.

\[
\rho = \frac{\sum_i (x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum_i (x_i - \bar{x})^2 \sum_i (y_i - \bar{y})^2}} \tag{5.14}
\]

The pairs having meaningful correlation with 5% significance level are displayed in Figure 5.18–5.23. The most interesting result is that the intrinsic shear modulus (shear modulus at 20 mK and 500 mK) have negative correlation with the NCRI and the dissipation of TO. This correlation seems to be associated with the crystal quality of the samples but exact causality is unclear.
Figure 5.18: Correlation between the NCRIF and the magnitude of shear modulus at 20 mK

Figure 5.19: Correlation between the NCRIF and the magnitude of shear modulus at 500 mK
Figure 5.20: Correlation between the dissipation of the TO and the magnitude of shear modulus at 20 mK

Figure 5.21: Correlation between the dissipation of the TO and the magnitude of shear modulus at 500 mK
Figure 5.22: Correlation between the sample pressure and the transition temperature of shear modulus

\[ \rho = 0.80 \]

Figure 5.23: Correlation between the mass loading of the TO and the dissipation in the shear modulus measurements

\[ \rho = -0.65 \]
5.7 Relaxation dynamics of NCRI and shear modulus

The most interesting difference between NCRI and shear modulus anomaly is the relaxation dynamics of them. Figure 5.24 and 5.25 show the dynamic responses of TO and the shear modulus measurement when they are subjected to abrupt temperature change from 40 mK to 50 mK. Even though the torsional oscillator has a long time constant of approximately 200 s, NCRI shows much slower relaxation. Following the method of Cornell group [12], the time dependent Davidson-Cole plots of NCRI and shear modulus were constructed in Figure 5.26 and 5.27. The same color represent the same elapsed time after the temperature is changed. The Davidson-Cole plot shows the characteristics of the dynamic susceptibility. A typical Debye solid is known to show a semi-circular D-C plot. Whereas the shear modulus finish the relaxation after few hundreds seconds showing semi-circular shape, NCRI shows long relaxation to skewed form. From this result, we can assume that there is another underlying mechanism in NCRI, which is different from that of the shear modulus anomaly.
Figure 5.24: The relaxation of NCRI and shear modulus when the temperature is increased from 40 mK to 50 mK.

Figure 5.25: The relaxation of the amplitude of the TO and the dissipation of the shear modulus measurement when the temperature is increased from 40 mK to 50 mK.
Figure 5.26: Time dependent Davidson-Cole plot for the NCRI in the torsional oscillator

Figure 5.27: Time dependent Davidson-Cole plot for the shear modulus anomaly
5.8 Acoustic resonance measurement

The experimental cell has a lot of acoustic resonances above 40 kHz. Therefore, the direct measurement of shear modulus was impossible above 40 kHz. Instead, the shear modulus can be measured indirectly by measuring the resonance frequency shift. The sound velocity of shear wave is given as

\[ v = \sqrt{\frac{\mu}{\rho}} \]  

(5.15)

where \( \mu \) is shear modulus and \( \rho \) is the density of solid. The resonance frequency is expected as

\[ f = \frac{v}{2L} \]  

(5.16)

where \( L \) is the length scale relevant to a resonant mode.

Figure 5.28 shows the shift of the acoustic resonance peak near 45 kHz. In Figure 5.29, the resonant peak position was plotted as a function of temperature. The temperature dependence of the resonance frequency change was almost identical to the measured shear modulus of the sample. According to the relation given by Eq. 5.15 and 5.16, the magnitude of \( \frac{\Delta f}{f} \) should be comparable with \( \frac{\Delta \mu}{\mu} \). The value of \( \frac{\Delta f}{f} \) may be small because the resonance is not relevant only to solid helium. The resonance peak in the empty cell that has the lowest frequency is the one at 40 kHz. The frequency shift by solid helium is about 4 kHz and the frequency change by the stiffening of the solid helium corresponds to 6% of it. This ratio may be reminiscent of NCRI. But the resonant frequency of the torsional oscillator is decreased by loading solid helium while the acoustic resonance frequency is increased by the solid helium.
Figure 5.28: Acoustic resonances of a 34 bar sample near 45 kHz

Figure 5.29: Acoustic resonance peak position and shear modulus of a 34 bar sample
5.9 Conclusions

The relation between NCRI and shear modulus increase was investigated by measuring both quantities in a single cell. The characteristics regarding shear modulus can be understood within the framework of dislocation pinning to impurities. The model of thermally assisted unpinning of dislocations can broaden our understanding of the phenomena. The drive-temperature dependent phase diagrams for the NCRI and the shear modulus are similar and the slow dynamics in the high drive amplitude and low temperature range are common in two measurements [66]. These similarities suggest that the dislocation pinning should be involved in the NCRI in some way.

However, important discrepancies are found by simultaneous measurements. The quantitative difference in the drive stress dependent behavior suggests the two phenomena could be caused by other kinds of excitations. Furthermore, the apparent difference in relaxation dynamics demonstrates the underlying mechanisms would be different. According to Balibar, the fluctuating mass currents by mobile dislocations induce phase fluctuations and eliminate the quantum coherence for supersolidity [67]. Pinning of dislocation lines may be a necessary precursor to the appearance of NCRI. The possible origin of the NCRI could be vortex fluid [22] or superfluidity in dislocations [19].
요 약 문

고체 헬륨의 비고전적 회전관성과 충밀리기
탄성률

초고체(supersolid)는 고체의 성질과 초유체(superfluid)의 성질을 동시에 가지는 역설적인 물질의 상태를 말한다. 1970년도에 그 가능성에 관한 논의가 제시되었는데, 2004년 Kim & Chan 실험에 의하여 고체 헬륨에서의 비고전적 회전관성(Non-classical Rotational Inertia)이 측정되면서 그 실제에 대한 논의가 활발히 진행되고 있다. 비고전적 회전관성이란 초유체의 성질을 가지는 고체 일부가 회전에 참여하지 않게 되면서 전체 고체의 회전관성이 줄어드는 현상을 말한다. 이러한 회전관성의 변화는 비틀림 진동자(torsional oscillator)에서 2π√(I/k)로 추출되는 진동주기를 정확히 측정함으로써 관측된다.

본 연구의 첫 번째 실험은 2차원 초고체에 관한 것이다. 2차원 헬륨 박막(film)은 Kosterliz-Thouless 이론을 따르는 초유체 현상을 보인다는 사실이 잘 알려졌다. 2차원 헬륨 박막은 다공성 기판(substrate) 위에 헬륨 원자를 흡착시켜서 실험적으로 구현하게 되는데, 기판과 헬륨 원자 간의 상호작용으로 때문에 기판 위 두 개의 층은 일반적으로 초유동성을 보이지 않는다. 이렇게 국소화 되어 있는 층을 불활성층(inert layer)이라고 하며 이 불활성층은 고체에 가까운 상태로 여겨져 왔다. 1993년에 흑연(graphite) 위에 흡착된 불활성층에서 기존의 2차원 초유동성과는 다른 압력의 초유동성이 관찰된 결과가 있는데, 이렇게 불활성층에서 나타나는 초유동성은 초고체 현상과 관련이 있을 것으로 예상하였다. 이에 Vycor라는 다공성 유리 기판 위에 헬륨 원자를 흡착시키고 불활성층에서의 초유동성을 조사하였다. Vycor 유리 기판을 넣은 비틀림진동자를 제작하였고 흡착시키는 헬륨의 양을 변화시키면서 온도에 따른 비틀림 진동자의 공명 진동수를 측정하였다. 다양한 두께의 헬륨 박막에
대해 측정을 하였으나 실험 장치의 분해능 이내에서 의미 있는 비고전적 회전관성 을 관찰할 수 없었다. Vycor 위에서는 헬륨 박막이 비결정적으로 흡착되는 것으로 알려졌는데 이러한 불활성층에서는 초유동성이 관찰되지 않는 것으로 생각된다. 헬 린원자와 기판 간의 상호작용을 약하게 하기 위하여 수소를 이용하여 기판을 덮은 다음 헬륨 박막을 흡착시켰다. 수소 위에 흡착된 헬륨 박막은 불활성층이 4분의 1로 줄어드는 것으로 알려져 있다. 실험 결과 많은 온도 영역에서 일어나는 초유동성이 관찰되었으나 초고체 현상과 달리 진동 속도를 증가시켜도 초유동성이 억제되지 않았다. 관찰된 현상은 불균일하게 분포된 액체 헬륨에서 나타나는 초유동성 때문일 수 있지만 수소 기판 위에 흡착된 불활성 헬륨에 대해서는 연구가 더 필요하다.

한편, 2007년에 Day와 Beamish에 의하여 고체 헬륨의 충실리기 반성률(shear modulus)이 저온에서 증가하는 현상이 관찰되었는데 그 양상이 기존의 비트림 진 동자에서 측정되는 초고체 현상과 매우 비슷하였다. 회전관성과 충실리기 반성률은 다른 물리량에서도 두 실험에서 보이는 온도 의존성이나 주파수 의존성, 구동 방향에 따른 의존성, 3He 농도의 의존성, 흡어지기(dissipation) 현상, 허스테리시스 등이 매우 비슷하였다. 충실리기 반성률의 증가는 어긋나기(dislocation)들이 3He 불순물에 피 닝(pinning)되면서 나타나는 현상으로 설명되었다. 높은 온도에서는 어긋나기들이 피닝(pinning) 되지 않고 움직이기 때문에 충실리기 반성률이 작게 나타난다는 것이 다. 충밀리기 반성률의 증가와 비고전적 회전관성과의 관계를 명확히 밝히기 위하여 두 가지 실험을 진행하였다.

다공성 기판 안에 체워진 고체 헬륨에서는 어긋나기들의 움직임이 나타나기 어 립다. 어긋나기의 움직임이 제한된 상태에서 비고전적 회전관성의 특성을 조사하기 위하여 180 nm의 기공크기를 가지는 다공성 금(porous gold) 안에 고체 헬륨을 키 우고 비틀림 진동자의 진동 주기를 측정하였다. 실험결과 1.4%에서 7%에 해당하는 다소 큰 값의 비고전적회전관성이 측정되었다. 이로서 어긋나기의 움직임이 없는 경우에도 비고전적 회전관성은 존재할 수 있음을 밝혀내었다. 또한, 고체를 만들 때 급랭(quenching) 방법을 사용하게 되면 비고전적 회전관성의 크기가 매우 크게 나타나는 것을 관찰하였는데, 다공성 금의 넓은 표면적 덕분에 효과적인 냉각이 일
어나면서 고체 내의 무질서(disorder) 양이 증가한 때문으로 생각된다. 저온에서 긴 완화(relaxation)을 보이는 현상도 관찰되었는데, 작은 기공 내에서 일어나는 완화는 어긋나기 때문이나 유타리(glassy) 고체의 특성 때문에 나타나다고 볼 수 없으므로 그 의미가 크다고 할 수 있다.

고체 헬륨의 충밀리기 탄성률과 비고전적 회전관성을 직접적으로 비교하기 위하여 두 물리량을 하나의 실험장치에서 동시에 측정하는 실험을 진행하였다. 실험 장치는 비틀림 진동자 내부에 충밀리기 탄성률을 측정할 수 있는 압전소자(piezoelectric shear transducer)를 넣어 제작하였다. 총 11개의 고체 시료가 정적방법(constant volume)을 통하여 만들어졌고, 온도, 구동 세기, 주파수 등 다양한 측정 조건을 조합하여 두 측정 사이의 상관관계를 조사하였다. 실험결과 두 물리량이 매우 유사한 온도 의존성을 보임을 확인할 수 있었다. 충밀리기 탄성률의 구동에 대한 의존성과 온도에 대한 의존성을 체계적으로 분석하여 열적으로 언피닝(unpinning)되는 어긋나기 이론에 의해 설명될 수 있음을 보였다. 11개의 고체 헬름 시료에 대하여 측정한 결과 충밀리기 탄성률은 저온에서 평균 27% 증가하였으며 비고전적 회전관성은 전체 시료의 1.65%에 해당하는 것으로 측정되었다. 충밀리기 탄성률 증가만으로 비고전적 회전관성과 같은 효과가 나타날 수 있는지 알아보기 위하여 유함요소법을 사용하여 전체 실험장치를 모사하였다. 그 결과 27% 충밀리기 탄성률 증가에 의하여 변하는 공명주기는 0.05% 이하인 것으로 나타났다. 따라서 충밀리기 탄성률의 증가 자체만으로는 비고전적 회전관성을 설명할 수 없음을 증명하였다. 각 시료에서 측정된 두 측정 사이의 상관관계를 분석한 결과, 충밀리기 탄성률의 증가량과 비고전적 회전관성 사이에는 선형관계가 없는 것으로 나타났다. 대신에 충밀리기 탄성률의 크기와 비고전적 회전관성 사이에 비바례 관계가 있는 것을 발견하였다. 충밀리기 탄성률 값이 큰 고체 헬름 시료일수록 비고전적 회전관성이 작게 나타난다는 것이다. 두 측정에서 구동 압력의 크기에 따른 의존성을 정량적으로 분석한 결과 비고전적 회전관성의 경우가 훨씬 낮은 압력 값을 갖는다는 사실을 확인하였다. 이는 충밀리기 탄성률이 증가한 상태에서도 비고전적 회전관성은 억제될 수 있음을 말해 주는 중요한 결과이다. 또 한 가지 늘라온 결과로 두 측정이 다른 동역학적 특성을
보인다는 점을 발견하였다. 갑작스러운 온도 변화에 의하여 충밀리기 턱성률은 빠르게 평형값을 찾아가는 데 비하여 비고전적 회전관성은 느린 완화과정을 보였다.

전체적인 실험결과로부터 비고전적 회전관성이 어긋나기의 움직임과는 다른 에너지 들뜸(excitation)과 관계되었을 것으로 예상할 수 있다. 이 에너지 들뜸의 실제로는 어긋나기(dislocation) 선을 따라 일어나는 초유동성(superfluidity)이나 소용돌이 유체(vortex fluid) 등이 될 수 있을 것이다.
References


**Academic Activities**


3. DUK YOUNG KIM, SANG-IL KWON, HYOUNGSOON CHOI, EUNSEONG KIM, *Torsional oscillator measurements on solid helium in a partially blocked annular channel*, APS March Meeting, (Mar 2009)


