Annealing Effect for Supersolid Fraction in $^4\text{He}$

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We report on experimental confirmation of the non-classical rotational inertia (NCRI) in solid helium samples originally reported by Kim and Chan. The onset of NCRI was observed at temperatures below $\approx 400\:\text{mK}$. The active velocity for initiation of the NCRI suppression is estimated to be $\approx 10\:\mu\text{m/sec}$. After an additional annealing of the sample at $T = 1.8\:K$ for 12 hours, $\sim 10\%$ relative increase of NCRI fraction was observed. Then after repeated annealing with the same conditions, the NCRI fraction was saturated. It differs from Reppy's observation on a low pressure solid sample.

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1. INTRODUCTION

Recently the observation of NCRI$^1$ in solid $^4\text{He}$ reported by Kim and Chan$^2$ for samples confined in Vyvor, in porous gold, and also in bulk solid. The existence of NCRI was reported by Shirahama et al.$^3$ and by Rittner and Reppy$^4$ with torsional oscillator (TO) measurements. We report on our independent experiment$^5$ to check the existence of NCRI and related properties. In spite of very large differences of the surface to volume ratios in different samples, the NCRI fraction in low temperature limit was on the order of $1\%$ for all reported cases. This indicates the observed phenomenon is not for a surface related one, but for a uniform system over length scales, 6 nm and larger. Nevertheless, a number of theoretical papers$^6,^7,^8,^9,^{10,11}$ suggest the superfluidity is unlikely to occur in a perfect crystal.

A possibility for Bose Einstein condensation (BEC) of zero point vacancies was suggested by Andreev and Lifshitz$^{12}$. If observed decoupling is a simple consequence of BEC of zero point vacancies, then the supersolid fraction should monotonically decrease as the pressure of solid sample increases from the melting curve and should not strongly depend on the sam-
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ple quality. However the measured\textsuperscript{13} pressure dependence of NCRI fraction was nonmonotonous with a maximum at \( \approx 55 \) bar. The absence of significant influence of annealing to the solid samples was pointed out by Kim and Chan\textsuperscript{13}. Bose condensation of other types of imperfections, for example, interstitial-vacancy pair excitations as elementary excitations have been considered by Galli and Reatto\textsuperscript{14}, and imperfections at crystalline boundaries and dislocations are studied\textsuperscript{5,9}. In the latter case we can expect some influence of sample quality. A recent experimental result supports such an idea that supersolid is preferred in a disordered sample and annealing out such disorder would destroy supersolid state. The complete disappearance of NCRI after annealing was observed by Rittner and Reppy\textsuperscript{4}.

In the present report we confirm independently the existence of the NCRI with TO technique and also show a preliminary result of the annealing influence to the bulk solid \( ^4\text{He} \) samples, which indicate the opposite result to the low pressure sample case reported by Ritter and Reppy\textsuperscript{4}.

2. EXPERIMENTAL DETAILS

We use a TO made of BeCu\textsubscript{25} alloy. The sample bob has a replaceable brass cover screwed and soldered with Wood’s alloy forming a cylindrical sample cell. Two types of cells have been used up to now. The difference between them is a small capacitance pressure gauge similar to described in Ref.\textsuperscript{15} mounted only on the 1st cell. The torsional rod is 2.5 mm outer diameter, 0.8 mm inner diameter and 20 mm long. The TO was mounted on a copper block mechanical isolator which was connected to mixing chamber and was operated at small ac velocities in constant amplitude mode. The 1st and 2nd empty cell have resonant frequencies 1499.6 Hz and 1534.7 Hz, respectively. The filling line was connected to the torsion rod with minimum dead volumes to avoid the influence of the solid helium inside of the torsion rod. The solid sample was 8 mm in diameter and 4 mm in height. Taking into account the volume of the pressure gauge, the sample volume was 160 mm\textsuperscript{3} for the 1st cell and 200 mm\textsuperscript{3} for the 2nd.

The solid samples were prepared from commercially available \( ^4\text{He} \,(\sim 0.3 \) ppm \( ^3\text{He} \) impurity) by blocked capillary method at slow cooling (10-15 mK/min) along the melting curve to avoid appearance of the pressure gradient at crystallization. The sample 1 was annealed at \( T = 1.75 \) K overnight before the measurements. The frequency change due to the increase of inertia moment by the sample is in reasonable agreement with molar volume estimation (see Fig. 1).

In the sample 1 measurement we utilized our pressure gauge, but be-
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Fig. 1. The resonant frequency shifts due to load of solid samples (1 and 2) as a function of temperature.

Table 1. Sample List

| sample number | cell | $f_{res}$, Hz | initial pressure, bar | $T_m$, K | final pressure, bar | $\Delta f$, Hz |
|---------------|------|---------------|-----------------------|--------|---------------------|---------------|
| 1             | 1st  | 1499.6        | 71.7                  | 2.08   | 40                  | 2.96          |
| 2             | 1st  | 1499.6        | 90.5                  | –      | $\approx$ 60        | 3.43          |
| 3             | 2nd  | 1534.7        | 74                    | 2.15   | 43                  | 3.76          |

cause of a large leak, appeared suddenly to the space between gauge electrodes, it started to work as a density gauge with a small sensitivity. We could still observe clear indication of the crystallization finish of the sample and estimate the final pressure of the sample. We could also see that the temperature inside of the cell has the synchronous behavior for empty and filled condition as the reading of a Matsushita carbon resistance thermometer mounted on copper isolator. This thermometer was calibrated against $^3$He melting curve thermometer below 1 K and against calibrated Ge thermometer up to 4.99 K and then checked with SRM768 fixed point device.

The measurements were performed at slow heating sweeps ($\sim$ 1 mK/min) to take a lot of points for statistics because of problems with amplitude stability caused by limited isolation of the vibration. At the end of NCRI measurements series the sample was heated to the melting point slowly and temperature $T_m$, where melting process sets in, was determined. Actually $T_m$ was indicated by a sharp amplitude drop. Such a change is expected
due to dissipation by the nucleation fluctuations of the liquid. A hysteresis was observed between cooling and heating which is typical for the first order transition. Finally the value of $T_m$ was determined with accuracy $\pm 0.01$ K. The results for the different samples are summarized in table 1.

3. RESULTS

We show the results of the NCRI measurements. Fig. 2 shows temperature dependence of the NCRI, resonant frequency at various ac velocities of the cell wall for sample 1 without additional annealing. Below 400 mK clear deviation from empty cell data observed. This indicates sample moment of inertia decoupling and we consider it as NCRI. NCRI fraction for the sample 1 was $\approx 0.1\%$ at 200 mK, that is rather large in comparison with other experimental data. Fig. 3 shows dependence of NCRI fraction at 200 mK on ac wall velocity and its suppression above $\sim 10\mu\text{m/sec}$.

Observed temperature dependence of NCRI fraction has qualitatively the same shape for all the samples. The sample 2 was measured only once at $V_{ac} = 8\ \mu\text{m/sec}$ and also exhibited high onset temperature $\approx 350$ mK and NCRI fraction $\approx 0.14\%$ at $T = 130$ mK with extrapolation of the empty cell resonant frequency down to lower temperatures.

![Fig. 2. The temperature dependence of the resonant frequency shifted to empty cell data for the sample 1 at various ac velocities as indicated on the figure without additional annealing.](image-url)
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Fig. 3. The NCRI fraction vs. $V_{ac}$ for the sample 1 at $T = 200$ mK. The data for 11 $\mu$m/sec were extrapolated to 200 mK.

For the sample 3 which was a bulk cylinder, the effect starts below 250 mK and maximum NCRI fraction was only 0.08% at 75 mK. Such reduction of NCRI for bulk cylinder is in agreement with data by Shirahama et al.\textsuperscript{3}.

The additional annealing was performed for the sample 1 at $T = 1.8$ K for $\approx 12$ hours. After annealing we performed measurements at 8 $\mu$m/sec. We observed relative increase of NCRI fraction up to 10% (see Fig. 3). After the second and third additional annealing no observable change appeared.

4. SUMMARY

The existence of NCRI was confirmed by TO technique. The measured NCRI fraction is in reasonable agreement with Kim and Chan data.\textsuperscript{13} The critical velocity is found to be on the order of 10 $\mu$m/sec. Annealing near the melting curve resulted in increase of NCRI fraction and further annealing made an saturation in it, but NCRI fraction never decreased by annealing as in low pressure samples.

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