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Surface ⁴He Adsorption Level Determination with a Microelectromechanical Oscillator

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A controlled amount of helium-4 is adsorbed onto a microelectromechanical oscillator. The number of ^4He atomic monolayers is extracted from the change of the effective mass of the oscillator by measuring the resonance frequency shift of the oscillator in its shear eigenmode. The method gives a mass resolution of $\approx 7 \times 10^{-17}$ kg, and allows for direct measurement of the ^4He adsorption level with the same device that is used in ^3He experiments.

KEYWORDS: MEMS, Preplate, Helium-4

1. Introduction

Bulk superfluid ⁴He is a macroscopic quantum state that is the first confirmed superfluid, which has undergone comprehensive studies over the past few decades. On the other hand, two-dimensional and quasi-two-dimensional ⁴He systems have also attracted broad interest. They exhibit thickness dependent topological phase transitions [1–3], and are investigated in the search for supersolids [4–6]. Furthermore, the surface scattering of ³He is significantly affected by the specularity of the surface in both the normal fluid and the superfluid [7–10]. The specularity of a surface is commonly enhanced by preplating it with several atomic monolayers of ⁴He. Therefore, it is often necessary to quantify the amount of ⁴He adsorption on a given detector. The conventional method of calculating the adsorption level is by introducing a controlled amount of ⁴He into the experimental cell with a known surface area, either by injecting a given amount of ⁴He in vacuum, or by introducing an amount of ³He-⁴He mixture with known ⁴He concentration level. This method provides an indirect way of measuring the adsorption level, but a direct measurement would be desirable.

Microelectromechanical systems (MEMS) oscillators have been successfully employed to detect quantum turbulence in superfluid ⁴He [11, 12], as well as viscosity in normal fluid ³He [13], and the surface states in superfluid ³He [14–16]. In particular, in the ³He experiments, the surface bound states are profoundly influenced by the boundary condition between the fully diffusive to the specular surface. The modification of the boundary condition is achieved through the controlled amount of ⁴He adsorption. Therefore, the ability to determine the level of coverage on the MEMS device allows a single MEMS device to directly determine the specularity, and to probe the surface states. Since the resonance frequencies of the eigenmodes of a MEMS oscillator depends on the mass of the movable parts, one can achieve a high resolution determination of the coverage using high quality factor micromechanical oscillators. In this work, we present results of using MEMS oscillators to measure ⁴He adsorption on its surface, and discuss the advantages of using this method.

2. Experimental Setup

The MEMS device used in this experiment is fabricated with the PolyMUMPS process. The device is fabricated from polysilicon. The main component of the device is the horizontal center plate with dimensions $1.5 \times 125 \times 125 \,\mu\text{m}^3$ suspended above the substrate by four serpentine springs. The movable parts of the device, which include both the center plate and the attached electrodes, are capacitively driven into the shear eigenmode. The COMSOL simulation of the shear mode is shown in Fig. 1. The direction of oscillation of the movable parts in the shear mode is along the x-direction as indicated by the arrows. The mass of the movable parts is $2.134 \times 10^{-10} \, \text{kg}$. The frequency spectrum

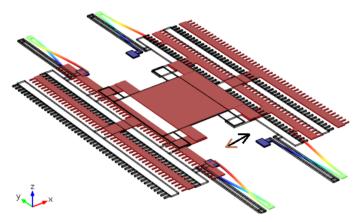


Fig. 1. The direction of the oscillation of the center plate in the shear eigenmode as indicated by the arrows. The black contour represent the equilibrium position of the device, and the colored shapes represent the displaced plate.

of the MEMS device in the shear mode is Lorentzian. At 4 K in vacuum, the measured resonance frequency of the shear mode is 23770.73 Hz, and the quality factor is $\approx 6 \times 10^5$.

The experimental cell contains a silver sinter heat exchanger, and is connected to a standard volume buffer container with volume $72.5 \,\mathrm{cm^3}$ at room temperature. The total surface area of the heat exchanger is $A_{\mathrm{Ag}} = 43 \,\mathrm{m^2}$. Helium-4 is injected into the experimental cell in several shots. Each shot involves first pressurizing the buffer container to about $6-7 \,\mathrm{kPa}$, which constitutes about $2\times10^{-4} \,\mathrm{mol}$ of $^4\mathrm{He}$. The buffer container is then opened to the cell through a fine controlled manual valve, and the $^4\mathrm{He}$ gas is bled into the cell over about $10 \,\mathrm{hr}$. The pressure in the cell is monitored by the MEMS oscillator. When gas is first introduced, the increase of pressure manifests as the reduction of the quality factor of the shear mode. As $^4\mathrm{He}$ is adsorbed, the pressure decreases, and the quality factor recovers to its vacuum value, while the resonance frequency decreases. The resonance frequency of the bare MEMS oscillator can be expressed as

$$f_{0n} = \frac{1}{2\pi} \sqrt{\frac{k}{m_c}},\tag{1}$$

where k is the total effective spring constant of the springs, and m_c is the mass of the movable parts. In the temperature range for preplating, k is temperature independent. After preplating, the shift of the resonance frequency due to the hardening of the spring and the mass loading on the oscillator can be estimated by

$$f_0 \approx f_{0n} \left(1 + \frac{E_4}{E_s} \frac{h}{t_1} - \frac{\rho_4}{2\rho_s} \frac{h}{t_2} \right),$$
 (2)

where $t_1 = 2 \,\mu\text{m}$ and $t_2 = 1.5 \,\mu\text{m}$ are the thicknesses of the spring cantilever structure and the movable parts, respectively; E_s and E_4 are the Young's moduli of silicon and solid ${}^4\text{He}$; ρ_s and ρ_4 are the densities of silicon and ${}^4\text{He}$; h is the thickness of the ${}^4\text{He}$ coating. It is estimated that the moduli term is about 3 orders of magnitude smaller than the mass loading term [17–19]. Therefore, any resonance frequency shift from its natural resonance frequency in vacuum, $\Delta f_0 = f_0 - f_{0n}$, can be attributed to the change of mass:

$$\Delta f_0 = \frac{1}{2\pi} \sqrt{\frac{k}{m_c + \Delta m}} - \frac{1}{2\pi} \sqrt{\frac{k}{m_c}} \approx -\frac{\Delta m}{2m_c} f_{0n}$$
(3)

where $\Delta m \ll m_c$ is the mass of the adsorbed ⁴He. This gives a mass resolution of $\approx 7 \times 10^{-17}$ kg (corresponding to about 0.009 monolayer) as defined by 10% of the full-width-half-maximum of the Lorentzian peak, and allows one to calculate the number of ⁴He monolayers adsorbed using

$$\Delta m = n_4 \sigma A M_4,\tag{4}$$

where n_4 is the number of ⁴He monolayers, σ is the surface molar density of ⁴He coating, A is the surface area of the movable parts, and M_4 is the molar mass of ⁴He. In this experiment, $\sigma = 17.33 \,\mu\text{mol/m}^2$ [7], and $A = 114190 \,\mu\text{m}^2$.

3. Results and Discussion

In this experiment, 6 consecutive shots were performed. The corresponding frequency spectra of the shear mode were measured with the minimum excitation to prevent nonlinear response, and the Δf_0 values were extracted. The resonance spectrum after each shot is shown in Fig. 2. The shots

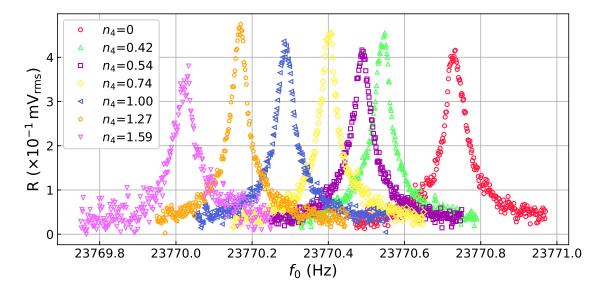


Fig. 2. The resonance spectra of the shear mode after each ⁴He injection. The measurements were done with the minimum excitation to prevent nonlinear response. The red circles represent the non-preplated spectrum. The legend shows the calculated number of ⁴He monolayers, n_4 using Eq. 4.

were performed at progressively lower temperatures in order for more ⁴He to be adsorbed. The first

injection (green triangles) was made at 2.5 K, and the last (lavender inverted triangles) was made at 0.9 K. The number of 4 He monolayers after each shot is shown in the legend. Another set of values of n_4 are obtained from the amount of injected 4 He with the assumption that 4 He uniformly preplates all surfaces of the experimental setup. The total surface area of the experimental setup is estimated by A_{Ag} which is much larger than all the other surface areas combined. The values of n_4 measured through the two methods are plotted against each other in Fig. 3. Both methods show $n_4 \approx 1.6$.

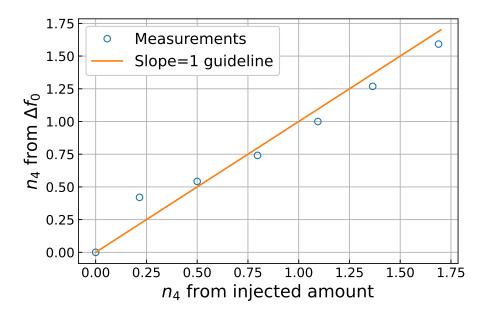


Fig. 3. The values of n_4 obtained from two different methods plotted against each other. The y-axis corresponds to the n_4 calculated from Δf_0 , and the x-axis corresponds to the n_4 calculated from the controlled amount of injected ⁴He. The straight line is the slope=1 guideline.

The straight line is the slope=1 guideline, and the measurements follow the guideline, showing that the two methods yield similar results. However, two interesting phenomena are observed. First, it is discovered that at sufficiently low temperature, the MEMS oscillator observes no pressure increase or mass loading when 4 He is introduced. This suggests that all injected 4 He is captured by the heat exchanger. Furthermore, it is discovered that after preplating the MEMS oscillator, $|\Delta f_0|$ decreases slowly while the quality factor remains the same. The effect is less pronounced at lower temperatures. It can be explained by the evaporation of 4 He adsorbed on the MEMS oscillator which is then preferentially captured by the heat exchanger. In either of the aforementioned scenarios, if one solely relies on the known amount of introduced 4 He, n_4 is overestimated. On the other hand, measuring Δf_0 of the MEMS oscillator should give the true value of n_4 irrespective of the experimental condition. Therefore, MEMS oscillators serve as good preplating monitors.

4. Summary

A MEMS oscillator is preplated by 6 shots of ⁴He. The resonance frequency shift of the MEMS oscillator in the shear mode from its vacuum value is measured. The number of adsorbed ⁴He monolayers on the MEMS oscillator is extracted using two methods: calculated from the resonance frequency shift, and from the controlled amount of injected ⁴He from a standard volume buffer container onto a known surface area estimated by that of the heat exchanger. The two methods give

similar results. It is shown that the preferential adsorption of ⁴He on the heat exchanger can cause overestimation of ⁴He preplating on the device. We propose that MEMS oscillators can be used as preplating monitors which do not suffer from this issue.

5. Acknowledgement

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