Effects of $^3$He impurities on the mass decoupling of $^4$He films

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We carried out quartz crystal microbalance experiments of a 5 MHz AT-cut crystal for superfluid $^3$He films on Grafoil (exfoliated graphite) with a small amount of $^3$He up to 0.40 atoms/nm$^2$. We found that the mass decoupling from oscillating substrate is considerable sensitive even in a small amount of $^3$He doping. In a $^3$He film of 29.3 atoms/nm$^2$, we observed a small drop in resonance frequency at $T_3$ of $\sim$0.4 K for a small amplitude, which is attributed to sticking of $^3$He atoms on the $^4$He solid atomic layer. For a large amplitude, the $^4$He solid layer shows a reentrant mass decoupling at $T_R$ close to $T_3$. This decoupling can be explained by the suppression of the superfluid counterflow due to the adsorption of $^3$He atoms on edge dislocations. As the $^4$He areal density increases, $T_R$ shifts to the lower temperature, and vanishes around a $^4$He film of 39.0 atoms/nm$^2$.

I. INTRODUCTION

It is well known that the surface of graphite is atomically flat and helium film on graphite grows up layer-by-layer to more than five-atom thick film in layers.1,6 Because of both the quantum nature of helium and the ideal two-dimensional system, helium film on graphite has been attracting the attention of many researchers. The adsorbed structure,1,3 the magnetism,4,5 and the superfluidity6,7 are extensively studied experimentally and theoretically.

Lately the nano friction of films, or the mass decoupling of films from oscillation, has been widely discussed.8 Several films on metal substrates show a partial mass decoupling.9 In addition, it is reported that the film takes place the pinning-depinning transition against the driving force of oscillating substrates.10

In response to the study on nano friction, we started to study the mass decoupling of helium films on graphite using the quartz crystal microbalance (QCM) technique. Up to the present, we have reported the following observations above two-atom thick films:11–14

(a) When the oscillation amplitude is large enough, the solid layer of $^4$He films undergoes partial mass decoupling below a certain temperature $T_S$.
(b) This decoupling brings a low-friction metastable state when the overlayer is normal fluid. The solid layer after the reduction in amplitude remains in the low-friction state with a finite life time.
(c) When the overlayer is superfluid, the mass decoupling suddenly vanishes at $T_D$ below $T_S$.
(d) For $^3$He films, the mass decoupling shows a similar behavior up to five-atom-thick films without an abrupt suppression due to superfluid.

The inhomogeneity of films plays an important role in this decoupling. We proposed the following scenario:13 The motion of the edge dislocation in the solid layer is responsible for mass transport. The mass decoupling occurs when the edge dislocation overcomes the potential barriers of the substrate (Peierls potential). This explains the external force threshold for mass decoupling and the low-friction state being metastable. In addition, the sudden vanishment below $T_D$ can be explained by the cancellation of mass transport due to the superfluid counterflow of the overlayer.

The mass decoupling of helium films has shown various interesting behaviors. In the present experiments, we confine ourselves to $^3$He impurity effects of $^4$He films on Grafoil (exfoliated graphite) when the overlayer is superfluid. In this Paper we report a systematic study on the mass decoupling using a MHz range AT-cut crystal.

After a brief explanation on the experimental setup in II, we show in III.1 the $^3$He areal density dependence for a four-atom thick film for various oscillation amplitudes. By adding a small amount of $^3$He, it was found that the mass decoupling appears again at a certain temperature $T_R$ below $T_D$ for a large amplitude. In III.2, we show the $^4$He areal density dependence for a fixed amount of $^3$He. $T_R$ decreases with increasing the $^4$He areal density, and disappears above a certain $^4$He areal density. In addition to these observations, we discuss a possible mechanism of the reentrant mass decoupling at $T_R$.

II. EXPERIMENTAL SETUP

We used the QCM technique with an AT-cut crystal to measure the mass decoupling. In the QCM technique, the coupled mass to the oscillating substrate is obtained from the change in the resonance frequency $\Delta f$ as

$$\Delta f = - \frac{m}{M}$$

where $m$ is the coupled mass of film, $M$ is the oscillating mass of the crystal, and $f$ is the resonance frequency. When the film is decoupled from the oscillation, the coupled mass decreases and the resonance frequency increases.

In the present experiments, the resonator is a 5.0 MHz AT-cut crystal. The crystal was commercially available, and no special treatment was applied to the Ag electrode. At first, Grafoil was baked in a vacuum at 900°C.
for 3 h, and a 300-nm-thick film of Ag was deposited onto it. The crystal and Ag-plated Grafoil were pressed together and were heated in a vacuum at 350°C for 2 h. Then, Grafoil was bonded on both sides of the Ag electrode. After bonding, the excess amount of Grafoil was removed to increase the Q value of the crystal. To keep good thermal contact, the crystal was fixed to the metal holder with electrically conductive adhesive. After these processes, the Q value remained better than 10⁴, and the areal density of Grafoil was 7.30 g/m². After being heated in 2 × 10⁻⁶ Pa at 130°C for 5 h, the crystal was mounted in the sample cell. In the present experiments, the mass loading of ³He is 3.8 Hz·atoms⁻¹·nm².

The resonance frequency was measured using a transmission circuit. In the circuit, the crystal was placed in series with a coaxial line connecting a 50 Ω cw signal generator and a RF lock-in amplifier. The frequency of the signal generator was then controlled in order to keep the inphase output zero, and was locked to the resonance frequency. The quadrature output at this frequency is the resonance amplitude.

In the present experiments, the ³He areal density is at most up to 0.4 atoms/nm², which corresponds to 5% of the areal density of ⁴He one-atomic layer.

III. RESULTS AND DISCUSSION

III.1. ³He areal density dependence

We carried out temperature sweep experiments of a four-atom thick ⁴He film for various oscillation amplitudes by changing the ³He areal density (Run A).

Figure 1 shows the variation in resonance frequency for ⁴He of 29.3 atoms/nm² with several ³He areal densities. The overlay of these films undergoes superfluid at low temperatures. All data were taken during cooling with the oscillation amplitude being fixed at 0.018 nm. In this amplitude, the superfluid onset of a pure ⁴He film was clearly observed at T_C of 0.80 K, although it is hardly seen in the scale of Fig. 1. As the ³He areal density increases, T_C decreases gradually. For a ⁴He film with ³He of 0.30 atoms/nm², T_C is shifted down to 0.75 K. By adding ³He, it was found that a small additional drop in resonance frequency appears at T_3 below T_C. As the ³He areal density increases, this drop becomes clear. However, T_3 does not depend strongly on the ³He areal density above 0.1 atoms/nm².

In the inset, we compare the variation in resonance frequency and Q-value between the pure ⁴He film and the ⁴He film with ³He of 0.20 atoms/nm². For the pure ⁴He film, the superfluid onset is observed at T_C of 0.80 K, accompanied with a small increase in ∆(1/Q). When ³He is added by 0.20 atoms/nm², T_C is slightly shifted down to 0.76 K. The resonance frequency is deviated downwards at T_3 of 0.41 K from the extrapolated curve from high temperatures. The difference from this extrapolated curve increases gradually down to the lowest attainable temperature ~0.1 K, and becomes ~1.5 Hz. On the other hand, the anomaly in ∆(1/Q) was not observed at T_3 within the present accuracy.

It is natural that the drop below T_3 is connected to the addition of ³He. The mass loading of ³He is estimated to be 2.9 Hz·atoms⁻¹·nm² from that of ⁴He. The drop of ~1.5 Hz at low temperature corresponds to ~0.5 atoms/nm² for ³He. This value is about the double of ³He dopant. Thus, it is concluded that the drop below T_3 is caused not only by the sticking of ³He atoms on the ⁴He solid layer, but also by preventing ³He atoms from decoupling. Furthermore, it was found that T_3 does not depend strongly on the ³He areal density above 0.1 atoms/nm², which means that a number of adsorption sites for ³He atoms is on the order of 0.1 nm⁻².

The possible candidate of the adsorption site on the ⁴He solid layer is the edge dislocation core. Because of the adsorption potential of graphite, the first solid atomic layer is about 20% denser than the second one. Due to the density difference between the solid layers, it is naturally assumed that the top solid atomic layer consists of commensurate domains separated by domain walls to the first solid atomic layer. Since domain walls have the same motif as edge dislocations, we here call them edge dislocations. The local areal density of the top solid atomic layer becomes small at the edge dislocation. From the difference in the zero-point energy, it is thought that ³He atom is adsorbed on the edge dislocation core from
the liquid overlayer. Here, it should be noted that the thickness of the liquid overlayer is at most one atomic layer and that $^3$He atoms may not be bounded on the free surface in contrast to bulk $^4$He.[18] In fact, it is revealed that $^3$He atoms are trapped on the dislocation core with the adsorption potential of 0.7 K in the case of $^3$He-$^4$He solids.[17]

Figure 2 shows that the amplitude dependence for a $^4$He film with $^3$He of 0.20 atoms/nm$^2$. All data were taken during cooling. As shown in Fig. 1, for the amplitude of 0.018 nm, the superfluid onset and the drop in frequency are observed at $T_C$ of 0.76 K and $T_S$ of 0.41 K, respectively. As the amplitude increases, the increase in resonance frequency due to the superfluid onset is smeared out. In contrast, for the amplitudes of 0.18, 0.25 and 0.56 nm, the resonance frequency increases clearly at $T_S$, and this increase is terminated abruptly at $T_D$.

As shown in the inset, these behaviors are also observed for a pure $^4$He film, which is attributed to the decoupling and sticking of the $^4$He solid layer. By adding a small amount of $^3$He, a new phenomenon appears. Below $T_D$, the resonance frequency rises up at a certain temperature $T_R$, which means that the $^4$He solid layer undergoes decoupling again. It was found that the reentrant mass decoupling temperature $T_R$ is close to $T_3$, i.e., the temperature where $^3$He atoms are trapped at the adsorption site on the $^4$He solid layer. For the amplitude of 0.56 nm, $T_D$ and $T_R$ disappear and the decoupling of the $^4$He solid layer remains at low temperatures.

To clarify the $^3$He areal density dependence of $T_S$, $T_D$ and $T_R$, we carried out temperature sweep experiments with the amplitude of 0.25 nm for several $^3$He areal densities. Figure 3 shows the variation in resonance frequency. All data were taken during warming. When $^3$He of 0.05 atoms/nm$^2$ is added, the decoupling and stick-
ing behaviors are drastically changed from the pure $^4\text{He}$ film. $T_S$ is lowered down to 0.69 K from 0.74 K of the pure $^4\text{He}$ film. In contrast, $T_D$ of 0.50 K does not change greatly. As the temperature decreases, the resonance frequency increases gradually below 0.4 K, and rises up at $T_R$ of 0.33 K. With further decreasing temperature down to 0.2 K, it decreases gradually again. Above $^3\text{He}$ of 0.10 atoms/nm$^2$, the increase in frequency at $T_R$ becomes sharp. As the $^3\text{He}$ areal density increases, $T_R$ increases gradually and $T_D$ does not change greatly.

The inset shows a phase diagram of decoupling and sticking behaviors. This diagram is divided into four regions. At high temperature, the $^4\text{He}$ solid layer sticks to the oscillating substrate (stick I). As the temperature decreases, this layer undergoes decoupling below $T_S$ (slip I), and sticks suddenly at $T_D$ (stick II), regardless whether or not the film contains $^3\text{He}$. By adding $^3\text{He}$, the reentrant mass decoupling appears below $T_R$ (slip II).

We discuss a possible mechanism of the reentrant mass decoupling. It should be noted that $T_D$ and $T_R$ show up when the overlayer of these films becomes superfluid. For the pure $^4\text{He}$ film, the sticking at $T_D$ can be explained by a mechanism in which the mass transport caused by the motion of edge dislocations is cancelled by the superfluid counterflow of the overlayer. [13]

In developing this scenario, we can explain the reentrant mass decoupling. A cartoon for $^3\text{He}-^4\text{He}$ mixture films is shown in Fig. 4. Since $^3\text{He}$ atoms are dissolved, or are spread over the fluid overlayer at high temperature, the $^4\text{He}$ solid atomic layer shows the decoupling at $T_S$ and the sticking at $T_D$ as the same manner as pure $^4\text{He}$ film (Figs. 4(a) and (b)). As above-mentioned, the sticking at $T_D$ means that the superfluid counterflow between edge dislocations cancels the mass transport. As the temperature decreases, $^3\text{He}$ atoms start to adsorb on the edge dislocation at around $T_3$, and prevent the exchange between liquid and solid $^4\text{He}$ atoms (Fig. 4(c)), i.e., the superfluid counterflow is ceased. Then, the $^4\text{He}$ solid atomic layer undergoes decoupling again at $T_R$.

**III.2. $^4\text{He}$ areal density dependence**

In a different series of experiments from III.1., we carried out temperature sweep experiments for a fixed amount of $^3\text{He}$ by changing the $^4\text{He}$ areal density (Run B). Figure 5 shows the variation in resonance frequency for several $^4\text{He}$ areal densities with $^3\text{He}$ of 0.20 atoms/nm$^2$. The data are shifted vertically. (Run B)

![Graph showing variations in the resonance frequency](image)

**FIG. 5.** Variations in the resonance frequency at the amplitudes of (a) 0.18 nm and (b) 0.018 nm for various $^4\text{He}$ areal densities with $^3\text{He}$ of 0.20 atoms/nm$^2$. The data are shifted vertically. (Run B)

remains. As further increasing the $^4\text{He}$ areal density, $T_R$ shifts to the lower temperature and vanishes at around $^4\text{He}$ of 39.0 atoms/nm$^2$.

In the amplitude of 0.018 nm, all data were taken during cooling. For $^4\text{He}$ of 28.5 atoms/nm$^2$, it is difficult to definitely determine $T_3$, i.e., the sticking temperature of $^3\text{He}$ atoms, while it is observed at $T_3$ of 0.40 K for $^4\text{He}$ of 29.0 atoms/nm$^2$. As the $^4\text{He}$ areal density increases, $T_3$ shifts to the lower temperature. Above $^4\text{He}$ of 35.0 atoms/nm$^2$, it is difficult to determine $T_3$ again. Here, it should be noted that $T_3$ has the same $^4\text{He}$ areal density dependence as $T_R$, although $T_3$ is observed in the limited range. On the other hand, the superfluid onset is also observed for these areal densities. As the $^4\text{He}$ areal density increases, $T_C$ moves to the higher temperature and reaches 1.23 K at $^4\text{He}$ of 39.0 atoms/nm$^2$.

Figure 6 shows the phase diagram of the sticking and decoupling behaviors for $^3\text{He}$ of 0.20 atoms/nm$^2$. $T_S$,
$T_D$ and $T_R$ are obtained from the amplitudes of 0.18 nm, while $T_C$ and $T_3$ at the amplitudes of 0.018 nm. In contrast to the phase diagram of Fig. 3, both regions of Stick I and II and of Slip I and II connect continuously. As mentioned above, $T_3$ is close to $T_R$. This supports strongly the scenario mentioned in III.1., i.e., the adsorption of $^3$He atoms on the edge dislocation causes the reentrant mass decoupling.

Furthermore, the vanishment of $T_R$ at a high $^4$He areal density may be explained by the competition between the adsorption on the edge dislocation and on the free surface. For bulk $^4$He, it is well known that $^3$He atoms are bounded on the free surface at low temperature.\[18\] The bound energy primarily comes from the difference in the zero-point energy between in bulk $^4$He liquid and on the free surface. Thus, we can propose the following scenario: In the case of an atomic-thin overlayer, $^3$He atoms are located on the $^4$He solid layer because of no advantage of the zero-point energy on the free surface. As the $^3$He areal density increases, i.e., the overlayer becomes thick, $^3$He atoms move to the free surface, and the adsorption of $^3$He atoms no longer occurs.

### III.3. The model calculation for $^3$He adsorption

We discuss whether the $^3$He areal density dependence of $T_3$ can be explained by a simple adsorption model. To build the model, we can refer to the previous experiments for $^3$He-$^4$He mixture thin films.\[19, 20\]

Saunders and co-workers have carried out heat capacity experiments of $^3$He above 0.4 atoms/nm$^2$ in a $^4$He film of 33.5 atoms/nm$^2$ on Grafoil. They have reported that $^3$He atoms in a thin $^4$He film behave as the two-dimensional (2D) Fermi gas.\[19\] On the other hand, Hallock and co-workers have carried out NMR experiments for 0.1 monolayer of $^3$He in thin $^4$He films on Nuclepore.\[20\] They have reported that a part of $^3$He atoms are immobile below a critical $^4$He areal density. As the $^3$He areal density increases, $^3$He atoms experience a mobility edge.

The present observations are quite similar to those of Hallock and co-workers’ experiments, i.e., a small amount of $^3$He atoms are localized in thin $^4$He films, and this localization vanishes at a certain $^4$He areal density. Sanders and co-workers concluded that $^3$He atoms in a thin $^4$He film are not adsorbed on Grafoil and are extended. We think, however, that there is a possibility that a small amount of $^3$He atoms are adsorbed because the heat capacity is independent of the areal density of the 2D Fermi gas.

From these considerations, we consider the following model: $^3$He atoms in the overlayer behave as the 2D Fermi gas with the hydrodynamic effective mass $m^*_3$. In addition, there exits a surface binding state with the adsorption site density $N_a$ and the binding energy $\varepsilon_a$ measured from the bottom of the 2D Fermi gas. In this model, the adsorption density $n$ is obtained as

$$ n = N_a e^{-\beta(-\varepsilon_a-\mu)} + \frac{2}{(2\pi)^2} \int_0^{\infty} \frac{2nk \, dk}{e^{\beta(-\varepsilon_a-\mu)} + 1}, $$

where $\beta = 1/k_BT$ is the inverse temperature, $\varepsilon = \hbar^2K^2/2m^*_3$ is the kinetic energy of the Fermi gas, and $\mu$ is the chemical potential which is determined from the $^3$He areal density.

Here, we may adopt $m^*_3/m_3 \sim 1.5$ from heat capacity experiments for a $^4$He film of 33.5 atoms/nm$^2$.\[19\] Although $m^*_3$ in thinner $^4$He films is still unknown. The inset of Fig. 7 shows a typical calculation of $n$ as a
function of temperature for several $^3$He areal densities with $m_3^*/m_3 = 1.5$, $N_a = 0.06$ sites/nm$^2$ and $\varepsilon_a = 1.268$ K. Here, the parameters were chosen where $n = 0.05$ atoms/nm$^2$ at 0.43 K for $^3$He of 0.20 atoms/nm$^2$. For comparison, we plotted a curve of $m_3^*/m_3 = 10$ at 0.20 atoms/nm$^2$.

As seen in the inset, $n$ increases gradually from high temperature and becomes nearly equal to $N_a$ below a certain temperature. As the $^3$He areal density increases, $n$ shifts to the higher temperature. Although it is not clear that which value of $n$ corresponds to $T_3$, it is assumed here that $n_c = 0.05$ atoms/nm$^2$ is $T_3$. We plotted the temperature where $n = 0.05$ atoms/nm$^2$ as a function of $^3$He areal density in Fig. 7. It was found that the calculated lines has a stronger $^3$He areal density dependence than that of the observations. This behavior does not depend strongly on the parameters of $N_a$, $\varepsilon_a$ and $n_c$. Thus, we may conclude that the simple adsorption model does not explain the areal density dependence of $T_3$. Here, we would like to make a comment on the model. As shown in Fig. 7, when we choose $m_3^*/m_3 = 10$, $n$ varies rapidly in a small temperature range and the areal density dependence of $T_3$ becomes weaker, i.e., when the number of density just above the surface binding state is large enough, $T_3$ does not depend strongly on $^3$He areal density. This may suggest that $^3$He atoms in a very thin overlayer are nearly localized on Grafoil. Although it was reported that $m_3^*$ is enhanced with decreasing $^3$He areal density for Nuclepore, this is only speculation for Grafoil. Furthermore, when there exists an attractive interaction between the adsorption sites, $n$ varies more rapidly. These are for future study.

Here, we make a comment on a thicker overlayer. $T_3$ was not observed clearly above $^4$He of 33.0 atoms/nm$^2$. It is, however, natural that $T_3$ is nearly equal to $T_R$. This means that $T_3$ tends to zero around $^4$He of 39.0 atoms/nm$^2$. As mentioned in III.2, $^3$He atoms are bounded on the free surface of bulk $^4$He. The binding energy $\varepsilon_S$ was obtained to be 2.22±0.03 K. This means that $\varepsilon_a$ in the simple adsorption model is smaller than $\varepsilon_S$ if the model explains the $^3$He adsorption.

IV. SUMMARY

We report quartz crystal microbalance experiments using a 5 MHz AT-cut crystal for $^3$He-$^4$He mixture films on Grafoil. In the present experiments, the $^3$He areal density is at most up to 0.4 atoms/nm$^2$. In a four-atom thick $^4$He film of 29.3 atoms/nm$^2$, we observed following behaviors: (a) For a small amplitude of 0.018 nm, a small drop in resonance frequency occurs at $T_3$. (b) For a large amplitude of 0.25 nm, the mass decoupling at $T_3$ and sticking at $T_D$ was observed as the same manner as pure $^3$He films. In addition to $T_S$ and $T_D$, a reentrant mass decoupling occurs at $T_R$ close to $T_3$. Here, it was found that both of $T_3$ and $T_R$ do not depend strongly on the $^3$He areal density above 0.1 atoms/nm$^2$, and are ~0.4 K.

From our previous study for pure $^4$He films, we have proposed the following scenario: the mass decoupling below $T_S$ results from the motion of edge dislocations between the first and second solid layers. The mass sticking at $T_D$ is caused by the cancellation of mass transport due to the superfluid counterflow of the overlayer. As an extension of this scenario, the observed behaviors can be explained as follows. $^3$He atoms which are mobile at high temperature are localized on the edge dislocation at $T_3$. These $^3$He atoms prevent the exchange between liquid and solid $^4$He atoms, and the reentrant mass decoupling occurs by the cease of the superfluid counterflow.

From experiments changing the $^4$He areal density for $^3$He of 0.2 atoms/nm$^2$, it was found that $T_R$ decreases with increasing $^4$He areal density and vanishes above $^3$He of 29.0 atoms/nm$^2$. This behavior can be interpreted by the competition of the adsorption between on the edge dislocation and on the free surface.

The above explanation about the reentrant mass decoupling below $T_R$ naturally leads to the model that $^3$He atoms adsorb on the $^4$He solid layer. However, this model cannot explain a weak $^3$He areal density dependence of $T_R$ using the known hydrodynamic effective mass of $^3$He in the overlayer. This is for future study.

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