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Adsorption of ^3He on ^4He crystal surfaces

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Abstract

We have compared the surface properties of crystals respectively grown from normal ^4He containing 130 ppb of ^3He and from ultrapure ^4He (0.4 ppb). Below 0.4 K, ^3He impurities are found to decrease both the surface tension and the step energy. Our results are consistent with the existence of two-dimensional bound states for ^3He atoms at the solid–liquid interface. Quantitative agreement with the data is found with a binding energy $\varepsilon_s \approx 4.3$ K and a ^3He density saturating around 0.4 monolayer. The presence of steps is found to increase the binding energy by about 10 mK.

1. Introduction

The existence of bound states for ^3He atoms at the liquid–vapor interface of ^4He – the so-called Andreev states – has received strong experimental evidence, both for bulk liquid [1] and for films [2]. Up to a coverage of half a monolayer, it has been shown that ^3He atoms adsorbed on bulk liquid ^4He behave as an almost ideal two-dimensional (2D) Fermi gas. Until very recently, much less interest had been devoted to the solid–liquid interface. Furthermore, the very possibility of bound states near a solid substrate could seem counterintuitive, since the localization of ^3He atoms increases their zero point kinetic energy. Nevertheless, Treiner has been able to evaluate the delicate energy balance between kinetic energy and van der Waals attraction by the substrate [3]. He finds that a bound state exists in the first oscillation of the substrate potential, with a binding energy $\varepsilon_s = 2.9$ K with respect to a bulk liquid state, and an effective mass of 2.3 times the bare mass. As this calculation is restricted

to a one atom situation, the density at saturation is not computed, but it is expected to be smaller than one monolayer. Beyond the analogy between the two interfaces, ^3He adsorbed on solid ^4He could display some interesting new behavior: depending on the orientation of the surface with respect to crystallographic directions, it can be either faceted, stepped or rough [4]. The binding energy, and maybe the interaction between fermions could thus depend on the structure of the surface. In the case of stepped surfaces, the regular array of steps modulates the substrate potential, thus leading to an anisotropic 2D Fermi gas.

There are very few experiments on the adsorption of ^3He on ^4He crystals. Carmi et al. [5] were the first to observe an effect of ^3He on the faceting temperature T_R of ^4He crystals. They related a lowering of T_R to the lowering of the surface stiffness γ which they estimated to be 15% at $T = 1$ K. Moreover, this shift was found to be independent of the bulk concentration X_{3b} of ^3He in the range 0.8–150 ppm. They deduced a binding energy ε_s of the order of 10 K. This value seems rather high, and it is difficult to understand why $\Delta\gamma$ does not depend on X_{3b} . A more direct evidence of adsorption was found by Wang and Agnolet [6] who measured the surface stiffness γ at $T \approx 0.1$ K for ^3He concentration ranging from 4.5 to

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50 ppb. They found that γ decreases with increasing concentration, which is consistent with an increasing coverage by impurities. They deduced $\varepsilon_s = 3.4$ K, in reasonable agreement with Treiner's calculation. A surprising feature of their results is the value of γ for the lowest concentration ($X = 4.5$ ppb): they find $\gamma \approx 0.06$ erg/cm², while the value for pure ⁴He is about 0.2 erg/cm². If attributed to ³He adsorption, this variation of γ would mean that the coverage is 1.2 monolayer, higher than expected by Treiner [3] (we define one atomic layer to have a number per unit area equal to $(n_3)^{2/3} = 8.1 \times 10^{14}$ cm⁻², where n_3 is the number density for pure liquid ³He at 25 bar). Also surprising is the fact that Wang and Agnolet do not report any variation of γ with temperature, although ³He is expected to dissolve in the liquid above 0.3 K.

The existing experimental data have left many questions open. In this context, we have performed new measurements of the surface stiffness of ⁴He crystals. They provide us with additional information but, as we shall see, the problem of ³He adsorption is not yet totally clarified. For rough surfaces, we have found that ³He impurities decrease the surface tension by several per cent as long as the temperature is less than 0.4 K where this effect vanishes. For stepped surfaces, we measured the step energy and found that it is substantially lowered in the presence of ³He. This variation of the step energy is found to be independent of temperature below 0.2 K. Our new data strongly support the existence of ³He bound states at the liquid–solid interface. We show that quantitative agreement can be found with a binding energy $\varepsilon_s = 4.3$ K and a ³He surface density saturating around 0.4 monolayer. We also find that the effect of a step is to increase ε_s by about 10 mK. Nevertheless, our analysis relies on several hypotheses which would need further experimental check.

2. Experimental results

The experimental cell was built to study the surface stiffness of HCP ⁴He crystals, and has been described in more detail in a previous article [11]. We recall here only the main features of the experiment. The volume of liquid in the cell is roughly 300 cm³, while the volume of the crystal is only about 10 cm³. The difference in ³He concentration between both phases [7] has a negligible consequence on the molar fraction X_{3b} of ³He in bulk liquid. Experiments have been made with both ultrapure ⁴He ($X_{3b} = 0.4$ ppb) and ⁴He with its natural purity ($X_{3b} = 130$ ppb). The cell is attached to the mixing chamber of a dilution refrigerator, and thermal contact is provided by a large sintered silver sponge made of 400 Å silver powder. Its surface area is about 300 m².

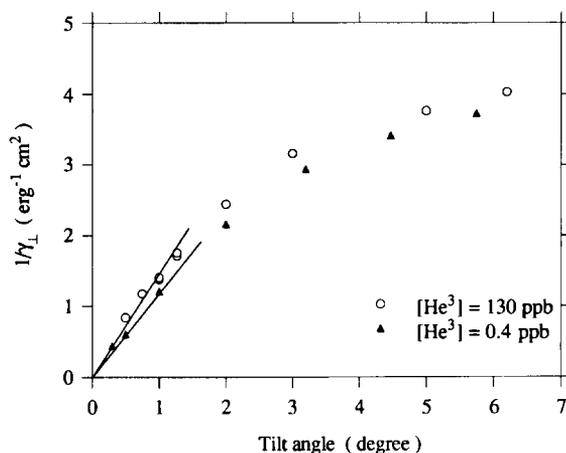


Fig. 1. Measurements of the component γ_{\perp} of the surface stiffness as a function of the orientation at $T = 0.2$ K. The circles correspond to regular helium 4 ($[^3\text{He}] = 130$ ppb), and the triangles to ultrapure helium 4 ($[^3\text{He}] = 0.4$ ppb).

Crystals are grown in a box which can be rotated around two perpendicular axes. The liquid–solid interface (≈ 10 cm²) can thus be oriented with respect to the sixfold symmetry axis (the c axis). At the interface, we propagate melting–freezing waves, as first done by Keshishev et al. [8]. The wave profile is scanned using an optical technique. We measure the wavelength and use the dispersion relation to calculate the surface stiffness. Depending on whether the direction of the wave vector is parallel or perpendicular to the projection of the c axis onto the surface, we obtain the component γ_{\parallel} or γ_{\perp} of the surface stiffness.

The experiment is performed below 0.5 K, so that the c orientation is faceted [9]. Let us call ϕ the tilt angle between the surface normal and the c axis. Fig. 1 shows the measured values of the component γ_{\perp} as a function of ϕ , at $T = 0.2$ K. Depending on the tilt angle, we distinguish between two regimes.

(i) For small tilt angles ($\phi < 1^\circ$), the surface can be described as an array of steps, whose mean distance is $a/\tan \phi$ (a is the step height). Then the component γ_{\perp} has the simple expression

$$\gamma_{\perp} = (\beta/a)(1/\phi),$$

where β is the step free energy per unit length. We checked that $1/\gamma_{\perp}$ vanishes linearly with ϕ (see Fig. 1). We can then compute the value of β/a , which we find to be $(11 \pm 1) \times 10^{-3}$ erg/cm² for the 130 ppb sample, and $(14 \pm 0.5) \times 10^{-3}$ erg/cm² for the ultrapure sample. The step energy does not depend on temperature between 0.1

and 0.2 K. The effect of impurities is measured by

$$\begin{aligned}\Delta(\beta/a) &= (\beta/a)_{130 \text{ ppb}} - (\beta/a)_{0.4 \text{ ppb}} \\ &= -(3 \pm 1.5) \times 10^{-3} \text{ erg/cm}^2.\end{aligned}$$

The systematic uncertainty is large, but the uncertainty on the temperature dependence is much smaller and does not exceed $0.5 \times 10^{-3} \text{ erg/cm}^2$. Indeed, for a given crystal, there is no observable temperature dependence of β .

(ii) At large tilt angle ($\phi > 5^\circ$), the distance between steps is smaller than their intrinsic width, so that steps overlap. The interface is rough and almost isotropic: we find approximately the same value for both component γ_{\parallel} and γ_{\perp} , and their angular dependence is small. In this case, the surface stiffness is close to the surface tension α . In Fig. 2, we plot the quantity $\Delta\alpha(T) = \gamma(130 \text{ ppb}, T) - \gamma(0.4 \text{ ppb}, T)$ as a function of temperature. The data have been taken from three different runs, corresponding to different crystals and different orientations. The change in the surface tension, $\Delta\alpha$, seems to increase from $-15 \times 10^{-3} \text{ erg/cm}^2$ at 0.2 K to zero above 0.4 K.

At first sight, the amount of ^3He in the ultrapure sample is not necessarily negligible. Indeed, if all ^3He atoms were adsorbed at the interface, one would obtain a coverage of 0.3 monolayer. However, we have not observed the temperature dependence of $\gamma(0.4 \text{ ppb}, T)$ which one would expect from the desorption of this very small amount of impurities: there is no systematic effect of temperature between 0.1 and 0.5 K, within the experimental scatter in the data which is of the order of 10^{-3} erg/cm^2 . In our opinion, this means that the remaining 0.4 ppb impurities are not bound to the interface, but rather trapped somewhere else in the cell, most probably on vortices. In the following analysis, we will make the assumption that with the 0.4 ppb sample, no ^3He is adsorbed on the liquid–solid interface.

The trapping of ^3He on vortices is also consistent with our observation that ^3He impurities have no effect on the growth rate of our crystals. Our experimental technique allows us to measure the growth resistance, which is proportional to the damping of the melting–freezing waves. A complete description and interpretation of the results will be published elsewhere [11]. For our present purpose, it is enough to mention that we do not see any effect of impurities on the growth rate, up to a tilt angle $\phi = 6^\circ$ and up to a temperature $T = 0.4 \text{ K}$. On the contrary, Wang and Agnolet (WA) [10] made systematic studies of the growth resistance of rough crystal surfaces as a function of the impurity concentration. They found an excess growth resistance in the presence of ^3He , and attributed it to the diffusion of impurities in the solid phase close to the interface. When extrapolating their model to our experimental parameters, we estimated an

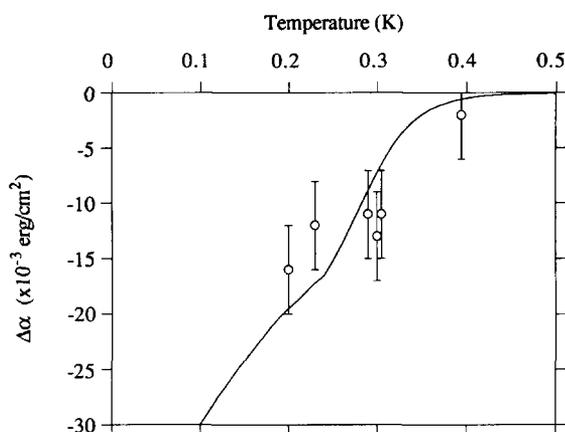


Fig. 2. Temperature dependence of the shift in the surface tension $\Delta\alpha = \alpha(130 \text{ ppb}, T) - \alpha(0.4 \text{ ppb}, T)$ due to ^3He impurities. The circles are the experimental data and the solid line the best theoretical fit, which was obtained with a binding energy equal to 4.3 K. The small kink in the curve $\Delta\alpha(T)$ is a consequence of the crude way in which our simple model treats the completion of the adsorbed layer. This kink is expected to disappear if interactions between adsorbed atoms are taken into account.

excess growth resistance which should be ten times larger than our measurement, even if uncertainties are taken into account. Of course, one could argue that a 6° tilt angle is not large enough to consider the surface as being completely rough as long as dynamical properties are concerned [11]. Furthermore, we use frequencies which are larger by an order of magnitude than WA. It is possible that their model is not robust enough to be used with our set of parameters. However, we think that the following interpretation is more likely.

Our measurements may be reconciled with those obtained by WA if the bulk concentration in the liquid is much smaller than 130 ppb. According to Varoquaux [12], this is possible: our cell is filled below the lambda point, and a large amount of vorticity can be created in the pores of the sinter. A large quantity of ^3He can then be trapped in the vortex cores, as ^3He atoms are bound to vortices with a large binding energy $\epsilon_v \approx 3 \text{ K}$ [13]. Assuming that there is one vortex per pore, and that each vortex is 400 \AA long, we estimate the total length of vortices to be of the order of $2 \times 10^{10} \text{ m}$, while it would be 10^4 times smaller in the WA experiment. We thus believe that, in our experiment and at low temperature, most of the ^3He atoms are trapped on vortices inside the sintered silver. The effective concentration of ^3He in bulk phase is very small and its contribution to the growth resistance is well beyond our experimental accuracy.

Our results also show that the existence of an adsorbate layer does not increase the growth resistance either.

In our opinion, this means that the adsorbed ^3He atoms always keep in equilibrium with the moving interface: the relaxation time of ^3He concentration in the adsorbate layer is presumably very short compared to the period of the capillary wave.

3. Discussion

The change in surface tension due to ^3He is given by elementary thermodynamics [14]:

$$\Delta\alpha = - \int_{-\infty}^{\mu} n_s(\mu') d\mu'. \quad (1)$$

Here μ is the chemical potential of ^3He , fixed by some reservoir, and n_s is the net density of ^3He atoms adsorbed on the surface. If that density saturates to a finite value n_{sat} , then $\Delta\alpha$ keeps increasing as a function of μ once saturation is reached,

$$\frac{\partial\Delta\alpha}{\partial\mu} = -n_{\text{sat}}. \quad (2)$$

Consider now a vicinal surface with a step density $n = \phi/a$. Each step provides a pinning potential for ^3He atoms: the adsorption is modified, thereby reacting on $\Delta\alpha$. The step energy is the change in surface tension per step, $\partial\alpha/\partial n$. Hence the change in β due to ^3He is given by

$$\Delta\beta = - \int_{-\infty}^{\mu} \eta(\mu') d\mu', \quad (3)$$

where $\eta(\mu')$ is the change per step in adsorption due to the inclination ϕ . As long as the interface is unsaturated, pinning on the step enhances n_s and hence $\eta(\mu') > 0$. On the other hand pinning lowers existing states, but does not produce new ones: steps do not increase n_{sat} . With this assumption, we expect that, in contrast to $\Delta\alpha$, $\Delta\beta$ should remain constant once saturation is reached.

In practice, what is monitored is the temperature T , not the chemical potential μ . In order to calculate $\Delta\alpha$ and $\Delta\beta$, we must:

(i) first identify the relevant reservoir of ^3He , so as to define the law $\mu(T)$;

(ii) make sure that the genuine *direct* temperature dependence of α ,

$$d\alpha = -S_s dT,$$

(where S_s is the surface entropy) is negligible as compared to indirect temperature dependence via $d\mu$.

Let us first show that the latter condition is met in practice. The measurements are done at a fixed density n_r

of ^3He in the reservoir, so

$$d\alpha = -S_s dT - n_s d\mu = S_s + n_s \left(\frac{\partial\mu}{\partial T} \right)_{n_r} dT.$$

As we are interested only in the variation of S_s due to ^3He , we have $S_s - S_{s0} = n_s (\partial S_s / \partial n_s)_T$. Using Maxwell's relations, one finds $(\partial S_s / \partial n_s)_T = -(\partial\mu / \partial T)_{n_r}$. Thus

$$d\Delta\alpha = -n_s \left[\left(\frac{\partial\mu}{\partial T} \right)_{n_r} - \left(\frac{\partial\mu}{\partial T} \right)_{n_s} \right] dT.$$

As we shall see, the binding energy is much larger than the temperature, so that the adsorbed ^3He gas is much denser than the one in the reservoir. As a consequence, we are allowed to neglect the second partial derivative in the above expression.

We are left with question (i), a question we must answer with the proviso that the surface is saturated when $T < 0.2$ K, as shown by the evolution of $\Delta\beta$.

3.1. The change in the surface tension $\Delta\alpha$

Let us assume that the adsorbed atoms behave as an ideal 2D Fermi gas on rough surfaces. Their energy spectrum is

$$\varepsilon = -\varepsilon_s^* + \frac{q^2}{2M}, \quad (4)$$

where ε_s^* is the binding energy at a rough solid–liquid interface, q is the momentum and M is the effective mass. Following Treiner [3], we shall assume that $M = 2.3 m_3$, where m_3 is the bare mass of ^3He atoms. The expression for $\Delta\alpha$ is then

$$\Delta\alpha = - \frac{M}{\pi\hbar^2} \int_{-\infty}^{\mu} d\mu' \int_0^{\varepsilon_{\text{max}}} \frac{d\varepsilon}{1 + \exp[(\varepsilon - \varepsilon_s^* - \mu')/k_B T]}. \quad (5)$$

If the interface is not saturated, the upper bound of the second integral can be taken equal to $+\infty$. The saturation is taken into account in the following empirical manner: we introduce a finite upper bound ε_{max} which is fixed by the requirement that n_s should be equal to the density at saturation, n_{sat} . At this point n_{sat} is an unknown parameter, which should not exceed $8.1 \times 10^{14} \text{ cm}^{-2}$ according to Treiner. If the interface is not saturated, and if the 2D gas is degenerate, the expression of $\Delta\alpha$ reduces to the simple form

$$\Delta\alpha = - \frac{\pi\hbar^2}{2M} n_s^2. \quad (6)$$

We have neglected interactions between adsorbed atoms, although one could take them into account (see

Ref. [1]). Given the poor accuracy of our data, we did not wish to add more unknown parameters. Our ambition is only to show that a simple model of ideal Fermi gas is sufficient to understand the main features of our results.

The total amount of ^3He in our cell ($N_0 = 10^{18}$ atoms) is much larger than the quantity required to saturate the interface. Most impurities are dissolved in a reservoir which fixes the chemical potential. At first sight, the three-dimensional (3D) bulk liquid ^4He is an obvious candidate. In this reservoir, ^3He atoms would form an ideal non degenerate 3D gas. The chemical potential would then be $\mu_{3D} = k_B T \ln(n_{3b} \lambda^3/2)$, where n_{3b} is the number density and λ the de Broglie wavelength: $\lambda = (2\pi\hbar^2/m_3^* k_B T)^{1/2}$, where $m_3^* = 3m_3$ is the effective mass of ^3He in the bulk liquid. One finds $\lambda \approx 17 \text{ \AA}$ at 0.2 K, and an approximate expression for μ_{3D} is $\mu_{3D} = -12k_B T$. When using $\mu = \mu_{3D}$ in Eq. (5), we cannot find any satisfactory fit to the data whatever the values of the parameters ε_s^* and n_{sat} . This is due to the large temperature dependence of μ , which in turn implies a large temperature dependence of $\Delta\alpha$. Indeed, once saturation is reached, Eq. (2) gives $\partial\alpha/\partial T = n_{\text{sat}}(\partial\mu/\partial T) \approx -12k_B n_{\text{sat}} \approx -1.4 \text{ erg/cm}^2 \text{ K}$.

In order to reconcile the theory with our data, we need a reservoir whose chemical potential varies more slowly with temperature. As seen before, ^3He is also bound to the vortices. Assuming that the total length L is of the order of $2 \times 10^{10} \text{ m}$, the mean distance between atoms along a vortex is about 200 \AA much larger than λ . If we assume that ^3He atoms can be described as an ideal 1D Fermi gas, this gas is non-degenerate and the chemical potential is now

$$\mu_{1D} = -\varepsilon_v + k_B T \ln(n_{3v} \lambda/2). \quad (7)$$

In the following, we take the same value for λ in bulk liquid and in the vortex core. This is not exactly true if the effective masses are not equal, but the dependence of μ on λ is weak. At low temperature most ^3He atoms are trapped on vortices but they start dissolving in the bulk at some finite temperature. The actual value of μ in the whole range of temperature can be computed by writing that μ_{3D} equals μ_{1D} and that the total number of ^3He is equal to N_0 . We thus find

$$\mu = -\varepsilon_v + k_B T \ln \left[\frac{N_0 \lambda}{L \frac{1}{2}} \right] - k_B T \ln \left[1 + \frac{V}{L \lambda^2} \exp \left(-\varepsilon_v/k_B T \right) \right]. \quad (8)$$

Three parameters should be adjusted: the binding energy ε_s^* , the density at saturation n_{sat} and the total length of vortices L , for which we have only an order of magnitude (we assume that $\varepsilon_v = 3 \text{ K}$).

From the above analysis, we know that the surface should be saturated below 0.2 K. If we suppose that saturation occurs at 0.2 K, we can evaluate n_{sat} using Eq. (6) and the measured value $\Delta\alpha = -15 \times 10^{-3} \text{ erg/cm}^2$ at $T = 0.2 \text{ K}$. We find

$$n_{\text{sat}} \approx 3 \times 10^{14} \text{ cm}^{-2}.$$

This value corresponds to 0.4 monolayer, which seems reasonable. If we had supposed that saturation occurs at 0.3 K, we would have taken $\Delta\alpha = -10 \times 10^{-3} \text{ erg/cm}^2$ and found $n_{\text{sat}} \approx 2.5 \times 10^{14} \text{ cm}^{-2}$. From n_{sat} , we estimate the band width for ^3He at the interface to be 0.7 K. This value proves that the 2D gas is degenerate, and justifies the use of Eq. (6) to evaluate n_{sat} .

Now we are left with only two adjustable parameters to fit the variation of $\Delta\alpha$, namely L and ε_s^* . As shown in Fig. 1, agreement can be found with the values

$$\varepsilon_s^* = 4.3 \pm 0.3 \text{ K},$$

$$L = (1 \pm 0.5) \times 10^{10} \text{ m}.$$

The fitting procedure is somewhat empirical. Other parameters are not well known: M , ε_v , n_{sat} and the mass M_v of ^3He atoms trapped on vortices, which was taken equal to m_3^* . However, M_v and L show up in μ only through the quantity $\rho = LM_v^{1/2}$. Adjusting M_v in addition to ε_s^* and L would not have improved the fit. The uncertainty on M_v only increases the uncertainty on L . As far as ε_v and n_{sat} are concerned, the fitted values of ε_s^* and L are not very sensitive to the precise value of these parameters, although the fit would have been worse for a larger coverage at saturation or a smaller binding energy on vortices. Finally, changing M has only a small influence on L .

Only ε_s^* and L were taken as adjustable parameters: the curve $\Delta\alpha(T)$ is sensitive to the value of ε_s^* and it is important to compare the fitted value of L with our previous estimate (one vortex per pore in the sinter). It turns out that the value of L is close to this crude estimate, and this confirms our hypothesis that most ^3He atoms are trapped in the sinter. The large uncertainty on L means that the fit is not very sensitive to its precise value; this is also satisfactory since this means that a possible evolution of the vorticity between experimental runs would not affect our results very much. Finally we find a value of ε_s^* which is comparable, although somewhat larger than the prediction of Treiner. ε_s^* mostly depends on the temperature at which $\Delta\alpha$ vanishes (around 0.35 K). At this temperature, adsorbed atoms dissolve in bulk liquid, and so do atoms trapped on vortices as ε_v is smaller than ε_s^* . This means that μ is controlled by the bulk, and that the variation of $\Delta\alpha$ between 0.3 and 0.4 K is not very sensitive to what happens with vortices. We thus think that our determination of ε_s^* does not depend

much on the precise behavior of the ^3He atoms trapped on vortices.

3.2. The change in step energy $\Delta\beta$

We expect the pinning of ^3He atoms on steps to be small, since steps are broad at the solid–liquid interface of ^4He . The change in height by one lattice spacing occurs on a width $w \approx 25 \text{ \AA}$ [15, 4].

As our measurements of $\Delta\beta$ are performed at low temperature, the 2D ^3He gas is degenerate and we can write

$$\Delta\beta = - \int_{-\infty}^{\mu} d\mu' \int_{-\infty}^{\mu} \delta v(\varepsilon) d\varepsilon = - \int_{-\infty}^{\mu} (\mu - \varepsilon) \delta v(\varepsilon) d\varepsilon, \quad (9)$$

where $\delta v(\varepsilon)$ is the increment in 2D density of states due to one step. The total change of β between $n_s = 0$ and $n_s = n_{\text{sat}}$ is

$$\Delta\beta = - \int_{-\infty}^{+\infty} \delta v(\varepsilon) \varepsilon d\varepsilon. \quad (10)$$

However, the use of this general expression is difficult since $\delta v(\varepsilon)$ is not known. An order of magnitude can still be obtained from dimensional arguments: we expect that a density n_{sat} of ^3He atoms will feel a pinning potential V on a step width w , so that

$$\Delta\beta \approx V n_{\text{sat}} w.$$

We thus find $V \approx 9 \text{ mK}$. The substrate potential on stepped surfaces is weakly modulated by the steps, so that the binding energy is nearly the same on rough, stepped or smooth surfaces. Still, the presence of ^3He impurities is able to lower the step energy by about 20%.

4. Conclusion

We have found further evidence that ^3He atoms adsorb on the surface of ^4He crystals. Surface tension measurements are consistent with the existence of 2D bound states, provided that up to 0.3 K, most impurities are trapped on vortices in the sinter rather than dissolved in bulk liquid. We find a binding energy $\varepsilon_s \approx 4.3 \text{ K}$, and we estimate the coverage at saturation to be of the order of 0.4 monolayer. The lowering of the step energy due to

^3He shows that the energy of the 2D Fermi gas is slightly lower on stepped surfaces than on facets.

Still our work is rather exploratory, and more precise experiments are needed in order to confirm our hypotheses. In particular, the trapping on vortices could be checked by cycling the cell over the λ point.

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