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Do Dislocation Loops Destroy Facet Flatness?

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Following our discovery of a new surface state near the facet edges of ^4He crystals, it was argued that thermally excited dislocation loops could destroy the facet flatness. Recent measurements of facet profiles of very small ^4He crystals indicate that the activation energy of these loops would have to be more than 20 K, which is quite high. The possible consequences of this result are discussed.

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1. INTRODUCTION

Generally, the shape of a ^{4}He-crystal consists of smooth, planar facets, which are joined by atomically rough curved sections. We have performed high-resolution interferometric investigations of the c-facet, corresponding to the (0001) basal plane of the crystal lattice, and the adjoining vicinal surfaces of hcp ^{4}He-crystals at low temperatures. In our previous experiments,[1] performed at temperatures in the range 0.1 K $\leq T \leq$ 0.6 K, we made the surprising discovery that the facet is slightly curved in the vicinity of its edges. One possible explanation was proposed based on a theory by Andreev: in any non-zero temperature there should be a non-vanishing amount of thermally activated dislocation-loop-like point defects, which cause a plastic deformation of the crystal.[2] According to Andreev, the loops may break the long-range order of the surface, and thus the flatness of the facets can be destroyed.

In the central part of the facet, sufficiently far from the facet edge, the effect of the loops should become more pronounced at higher temperatures.

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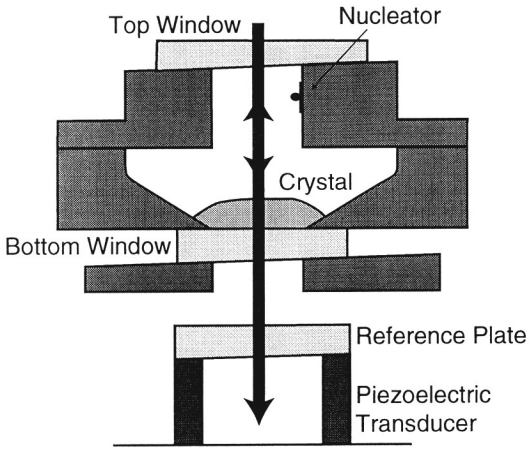

Fig. 1. Optical cell.

Also, if the long-range order is destroyed, the surface stiffness γ of the facet becomes finite, and thus the smaller the crystal, the more curved its facet should be. In order to test this model, we have observed c-facets of small ^{4}He crystals at relatively high temperatures (0.9 K), and determined a lower limit for the activation energy of the loops.

2. EXPERIMENTAL TECHNIQUES

These investigations have been performed using the two-beam optical interferometer developed in our laboratory for experiments on solid/liquid and liquid/vapor interfaces at low temperatures.[1] Laser light from a He-Ne-laser (λ =632.8 nm) is guided into the cryostat via a single-mode optical fiber. The beam is expanded to a diameter of 5 mm and guided through mirrors and a beam splitter to the cell, where a part of the light is reflected from the crystal surface and a slightly larger fraction from the anti-reflection-coated reference plate, located under the bottom window. The ensuing interference pattern is focused onto a CCD sensor located inside the 4-K-vacuum can. The sensor is controlled by a slow-scan STAR I camera (Photometrics, Tucson, Arizona), from which the images are transferred to a computer for processing and analysis.

The optical cell, as illustrated in Fig. 1, is a copper cylinder with a diameter of 40 mm, and a polished conical lower part. The fused silica windows sealing the cell from the top and bottom are wedge-shaped, in order to minimize the effect of extra reflections on the interference image. The ref-

erence plate is supported by a cylindrical piezo-electric transducer, allowing the plate to be moved vertically in order to create a phase shift in the interference pattern. By digitally averaging images having phase shifts of $+\pi/2$ and $-\pi/2$, the fringes disappear resulting in an image of the background light only. This image is finally subtracted from the interferograms to fully reveal the interference fringes.

The sample crystal was nucleated using the capacitor technique developed by Babkin *et al.*[3] to achieve the correct orientation with the crystal resting on its c-facet on the reference plate. The crystal was grown from ultra-pure ^{4}He, where the fraction of ^{3}He impurities was on the order of 3×10^{-10}

The amount of ^{4}He entering or leaving the cell was controlled by a flow control device at the room temperature part of the cell filling line. This enabled us to melt the sample crystal to relatively small sizes, with the facet radii ranging from 2 to 5 mm, in order to maximize the possible effect of the dislocation loops. Below this size, the crystal easily became unstable and collapsed.

3. THEORY

The shape of a liquid/solid interface is governed by its surface stiffness γ. In an one-dimensional case, γ is a function of the angle ϕ between the surface and the plane of the facet. Facets correspond to high-symmetry orientations of the crystal lattice, and thus near the facet edge, where ϕ is small, the surface can be viewed as consisting of steps separated by terraces. $\gamma(\phi)$ depends on the form of the interaction between the steps. In the case of a repulsive $1/r^2$-step-step-interaction, $\gamma(\phi)$ is linear and the profile of the surface $\zeta \propto x^{3/2}$, where x is the distance from the facet edge.[4] Our previous measurements[1] indicated, however, that near the edge $\zeta \propto \exp(x/x_0)$, resulting in $\gamma \propto 1/\phi$. At angles larger than a critical angle $\phi_c \approx 100$ μrad, our results were consistent with the $\zeta \propto x^{3/2}$-behavior.

In the model based on Andreev's theory, the presence of the thermally activated dislocation loops, which break the long-range order of the crystal lattice, results in a spontaneous reconstruction of the interface. The surface parts of the loops form small elementary steps on the facet, and may so destroy its flatness.

It has been statistically calculated[1] that the presence of the dislocation loops results in a surface stiffness of the form $\gamma \propto 1/\phi$, which is in accordance with the experiments. In order to test the validity of this hypothesis, knowledge of the activation energy of these loops is required. In cylindrical

geometry near the center of the facet Andreev's model results in a surface profile

$$\zeta(r) = \frac{2k_BT}{\xi} e^{-\epsilon/T} \cosh\left(\frac{\xi\sigma}{k_BT} r\right), \tag{1}$$

where σ is the mean number of unit cells in the piece of elementary step (≈ 100, an estimate consistent with the results reported in Ref. 1) generated by the loop and $\xi = (C\beta s_0)/(2ar_0)$, where β/a is the energy of an elementary step on the surface, s_0 the unit cell area, r_0 the facet radius and $C \approx 1$ a parameter describing how far from equilibrium the facet size is. From the maximum deviation of the surface $\Delta\zeta_{max} < \delta$ one can, using Eq. 1, estimate a lower limit for the activation energy:

$$\epsilon \geq \frac{C\beta}{2\kappa a} + T \ln\left(\frac{2k_BTar_0}{Cs_0\beta\delta}\right). \tag{2}$$

The numerical value of $\kappa = \gamma\phi/T = k_B/s_0\sigma$, $\kappa = 1.1 \times 10^{-3}$ erg/cm^{2}K is known from previous experiments,[1] and $s_0 \approx 1 \times 10^{-15}$ cm^2. For the the step energy, we used the value $\beta/a = 1.4 \times 10^{-2}$ erg/cm^2 measured by Rolley *et al.*[5]

4. RESULTS

Several interference images of a single crystal were taken at temperatures in the range 0.8 K $\leq T \leq$ 0.9 K. A typical interference image after filtering and background subtraction is displayed in Fig. 2; a large fraction of the facet is clearly visible as a disc-shaped set of straight fringes.

The lower limit for the activation energy ϵ was estimated using Eq. 2 and the data extracted from the images. The facet size was calculated by fitting a circle on the visible part of the facet edge, and the surface profile by taking radial slices and analyzing the intensity patterns in comparison to those obtained by imaging a larger crystal, where the facet fills the entire field of view. It was difficult to deduce the size of the entire crystal, as its edge was completely outside the field of view, so we estimated that the facet is close to equilibrium and thus $C \approx 1$. This procedure is also justified by the fact that we were looking for a lower limit of the activation energy ϵ, and larger values of C would result in an increase of ϵ.

Fig. 3 displays the profile of a small crystal ($r_0 = 3.2$ mm), imaged at $T = 0.9$ K. The maximum possible deviation of this surface from a flat plane is found to be $\delta = 0.176$ μm, and inserting this, the facet radius and the required parameters into Eq. 2, we obtain $\epsilon \geq 17.8$ K.

The results of measurements on the same sample at slightly lower temperatures were similar, and thus we conclude that the activation energy of

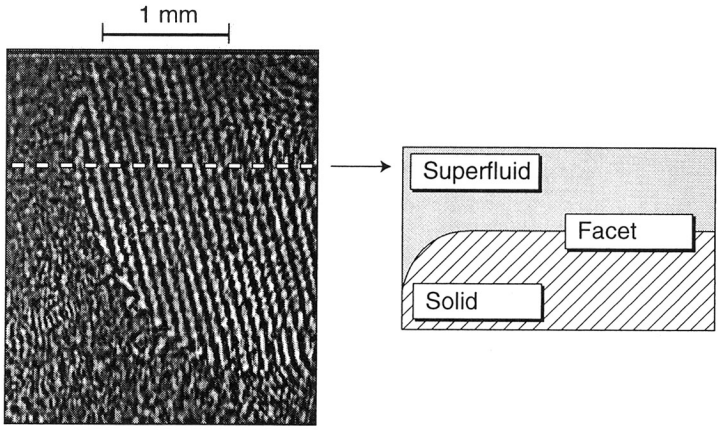

Fig. 2. a) Interference image of a crystal surface. The set of straight fringes represents the flat facet, slightly tilted with respect to the reference plate. On the adjoining curved part the fringes become too dense to be visible. The edge of the crystal falls outside the field of view. b) Sketch of the crystal profile along the dashed line in Fig. 2 a).

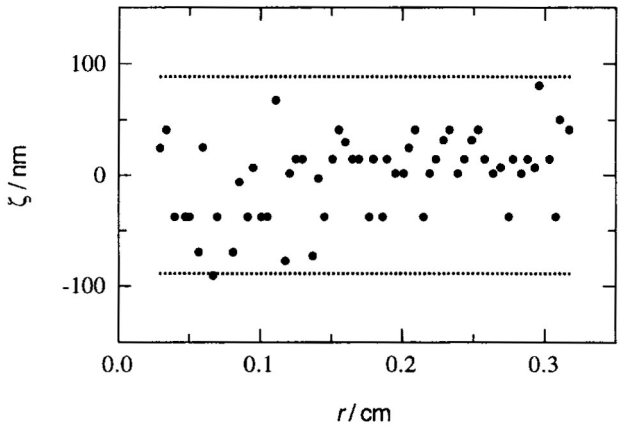

Fig. 3. Facet profile of a ^{4}He crystal at $T = 0.9$ K. The facet appears flat within the limit of our resolution. The dashed lines indicate the limits used to estimate the maximum possible deviation of the facet from a flat plane.

the dislocation loops, if present, is $\epsilon \geq 18$ K. No temperature variation of the upper limit of the facet deviation was observed.

5. DISCUSSION

The density of dislocation loops in the central part of the facet is roughly proportional to $\exp(-\epsilon/k_B T)$. At temperatures 0.1 K $\leq T \leq$ 0.6 K, where the new surface state was observed, this density is exponentially small due to the high activation energy and so these loops cannot be observed; nor do they influence the properties of the central part of the facet (in the mentioned experiments, only facet regions close to the edge were imaged). On the other hand, the obtained lower limit for the activation energy itself is quite reasonable for dislocation loops of such size ($\approx$ 30 nm). Thus our measurements raise the question of which possible new experiments could help to clarify the situation. One possibility is to try to observe the facet curvature in its central part at low temperatures, $\approx$ 0.1 K, and at rather high overpressures, up to 100-200 μbar (this case corresponds to very high values of the constant C, see Eq. 1). According to Ref. 6, such conditions can be achieved in perfect crystals, without growth dislocations. Moreover, it cannot be excluded at this moment that the mechanism of so-called slow growth of perfect crystals, which is still unknown (see Ref. 6), is due to Andreev's loops.

Of course, at this stage we cannot exclude other possible mechanisms of the facet curvature. Some of them were discussed briefly in Ref. 1. One more mechanism, based on the pinning of steps by defects, was suggested by Thuneberg.[7] However, this mechanism is hardly consistent with the observed very high mobility of steps at low temperatures.[6]

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