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Influence of plastic deformation and of impurities on internal friction in solid He^4

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Internal friction at frequencies 15 and 78 kHz were measured in samples of solid He^4 with molar volume 20.55 cm^3. The samples were grown at constant pressure, and also by the blocked-capillary method. The construction of the container made it possible to carry out measurements of the damping in plastic deformation of solid helium. The internal friction in samples of solid helium with He^3 admixture (0.01–0.1 at. %) was also investigated. The reduction of the temperature and amplitude dependences of the damping by the theory of Granato and Lucke has made it possible to determine a number of dislocation parameters.

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Measurements of the internal friction in crystalline helium make it possible to investigate defects in the crystal structure. Because of the high purity of crystal (impurity concentration less than 10^{-6} at. %), such defects are vacancies and dislocations. The study of these defects is of great fundamental interest, since the large amplitude of the zero-point oscillations of the helium atoms can lead to specific effects not observed in ordinary substances. Meĭerovich[1] has discussed the contribution of vacancies to the internal friction; it was shown that delocalization of the vacancies leads, for example, to absorption of the energy of the mechanical oscillations even under spatially homogeneous deformation. This effect was not observed in ordinary materials.

The measurements of the internal friction in crystalline He^4 were carried out up to now on pure single-crystal samples grown at constant pressure, at megahertz[2,3] and kilohertz[4,5] frequencies. On the basis of these results it was suggested that the main mechanism of the damping is due to dislocations. A reduction of the measurement results[2,5,6] by a Granato-Lucke theory[7] has made it possible to determine a number of dislocation parameters. However, the values of the parameters determined at various frequencies are in poor agreement. Thus, at the present time there is no model that describes well all the experimental results.

The internal friction due to dislocations depends on the internal state of the sample. By changing this state, for example by changing the crystal-growth conditions or by plastic deformation, it is possible to attempt to separate the contribution of the dislocations to the internal friction. The presence of a small amount of impurities also influences the value of the dislocation internal friction.

The present study was devoted to the influence of these actions on the internal friction in He^4 crystal with molar volume $V_{mol} = 20.55$ cm^3.

EXPERIMENTAL PROCEDURE

To excite and register the oscillations of solid helium we used quartz resonators of two types. The container in which the resonator of the first type was mounted, with the fundamental flexural oscillation mode (in vacu-

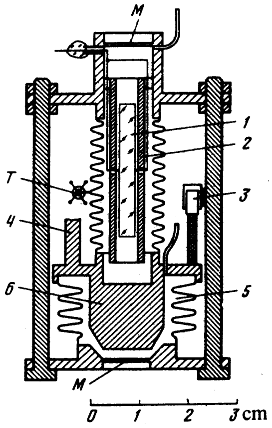

FIG. 1. Construction of container: 1—quartz resonator, 2—stainless tube, 3—capacitive displacement pickup, 4—copper cold finger, 5—pressure chamber, 6—movable bottom, T—resistance thermometer, M—membrane of capacitive pickup.

um at $T=4.2$ K, $f_0 \approx 9.9$ kHz), is described in Ref. 5.

Figure 1 shows the construction of the container, in which was mounted a quartz resonator of the second type. The quartz crystal 1, cut in the form of a cylinder of 3.4 mm diameter and 26.6 mm length, was located on the axis of tube 2. The placement of the electrodes made it possible to excite in the quartz cylinder torsional vibrations. The parameters of the fundamental mode in vacuum at $T=4.2$ K are the following: $f_0 = 74.3$ kHz and $Q=2\times10^5$. The quartz was secured at points corresponding to the nodes of the vibrations. The current leads were electrically insulated from the body of the ampoule. The gap between the outer surface of the quartz crystal and the inner wall of the tube was 0.5 mm.

The internal friction in the quartz + solid helium was determined from the damping of the free oscillation of the system. The measurement system and the procedure for determining the logarithmic damping decrement Δ are described in Ref. 5; the relative error in the measurement of the decrement did not exceed 10%. We note in addition that in the measurement of the amplitude dependences of the damping the initial amplitude of the recorded system exceeded the final amplitude by not more than 1.4 times. Thus, the amplitude was determined accurate to $\leq 20\%$.

The sample was grown at constant pressure of 32 atm. This pressure distended the bellows and pressed the bottom 6 to the support. The crystal growth in both types of container occurred in two ways. To obtain perfect samples, the crystals were grown at constant pressure with low speed ~$(5-10)\times10^{-4}$ cm/sec. The crystal was initiated on the cold finger and after a time of the order of several hours it filled the entire internal volume of the ampoule. To obtain samples with defects, the growth was by the blocked-capillary method. The ampoule was filled with liquid helium at a pressure of 61 atm; the final pressure measured after the end of the growth of the crystal with a capacitive pickup was ~35 atm. The growth time in this method was ~10 min.

The temperature was monitored with a carbon resistance thermometer (see Fig. 1) and was maintained accurate to ~0.01 K during the time of the damping measurements.

The construction of the ampoule (Fig. 1) made it possible to deform plastically the helium located in the gap between the quartz crystal 1 and the tube 2. In these experiments we measured first the amplitude and temperature dependences of the damping in the undeformed crystal. The temperature of the container was then set equal at 1.60—1.68 K, after which liquid helium was fed into volume 5 under pressure. At a pressure exceeding ~7 atm, displacement of the moving bottom 6 of chamber 5 started. The displacement was measured with a capacitive pickup 3 and reached 1.5 mm in our experiments. This displacement of the bottom 6 (the relative change of the distance from the bottom to the end of the crystal was ~20%) on account of the compression of the solid helium produced a force acting on the end fact of the crystal 1. This force led in turn to shear stresses σ in the gap between the crystal and the tube, much greater than the yield point σ_y ($\sigma \sim 5\times10^5$ dyn/cm^2, $\sigma_y \leq 4\times10^4$ dyn/cm^2), and caused plastic flow of the helium in the gap. After the maximum displacement of the bottom was reached, we waited ~15 min, after which the pressure in the chamber 5 decreased to ~1 atm. The solid helium in the gap between the quartz and the tube was then again plastically deformed. The time 15 minutes was chosen on the basis of measurements of the relaxations of the mechanical stresses.[8] To prevent the container from being heated by more than 0.02 K by the plastic deformations, the speed of the bottom was chosen to be of the order of $(5-10)\times10^{-5}$ cm/sec.

After the end of the deformation, we measured the time and amplitude dependences of the damping. The temperature of the container was then lowered to 1.21—1.30 K and a second deformation was produced by the same method.

In the experiments, besides helium purified by the thermomechanical effect, we used for the crystal growth also mixtures of He^3 and He^4 with He^3 concentration 0.01 and 0.1 at.%.

EXPERIMENTAL RESULTS

1. Undeformed crystals

The results of the measurements of the damping decrement Δ of the quartz + solid helium system at the frequency ~15 kHz in pure samples are reported in Ref. 5. Up to helium deformation amplitudes $\varepsilon_m \sim 10^{-5}$, the decrement was independent of the amplitude, and the averaged $\Delta(T)$ dependence for crystals with $V_{mol}=20.5$ cm^3 is shown for the purpose of illustration in Fig. 5a below.

In the container with the torsion quartz (Fig. 1), the solid-helium crystals were grown at a pressure 32 atm ($V_{mol}=20.55$ cm^3). The resonant frequency of the quartz + solid helium system was $f=78.4$ kHz and was constant with accuracy ~1% in all the experiments. The shear modulus of the solid helium, determined from the ratio f/f_0, agreed with the average shear modulus obtained in ultrasound measurements, accurate to ~10%.

The amplitude dependences of the decrement were measured at temperatures 0.62—1.75 K at maximum relative solid-helium deformations $\varepsilon_m = 3.10^{-8} - 10^{-5}$. In this interval, within the limits of the measurement error (~10%), the damping is independent of the amplitude.

Figure 2a shows the results of measurements of the temperature dependence of the damping on two samples. It is seen from this figure that: a) the lowering of the temperature leads to a monotonic decrease of the damping; b) the measured values of the decrement lie in the interval $0.015 < \Delta < 0.05$.

2. Internal friction in pure plastically deformed samples

Observations of the damping produced during the time of plastic deformation of solid helium have shown that

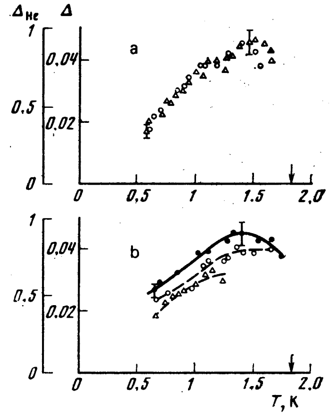

FIG. 2. Dependence of the damping on the temperature: a) for two samples, b) ●—undeformed sample, ○—sample plastically deformed at $T = 1.62$ K, △—sample plastically deformed at $T = 1.28$ K. Vertical arrow—melting temperature of sample.

the process of plastic flow of helium in the gap between the quartz and the tube does not influence substantially the internal friction (the change of the decrement is less than 20%). These observations were made with a displacement ~0.8 mm of the bottom. With further displacement, one of the electrodes was short circuited to the case and the measuring system became inoperative. This fact indicates that the longitudinal displacements of the quartz reached ~0.3 mm—the value of the gap between the current leads and the tube 2. When the pressure in the chamber 5 was decreased to ~1 atm, the bottom moved back to the initial position; the current leads were again electrically insulated from the case of the ampoule.

At the instant when the bottom stopped, we measured the time dependence of the decrement. Examples of such dependences are shown in Fig. 3. The value of the decrement at $t = 0$ is given for the undeformed sample. As seen from Fig. 3, the time dependences reveal no regularity whatever and after a time of ~30 min the value of the decrement does not change substantially. The measurements of the dependences of the damping on the amplitude of the oscillations in the plastically deformed samples have shown that prior to the deformation of the helium $\varepsilon_m \sim 10^{-5}$ the decrement is constant. These measurements were performed at temperatures 0.6–1.7 K.

The plastic deformation of the solid helium led both to an increase and to a decrease of the damping. The maximum relative change of the decrement did not

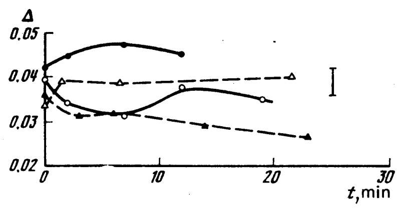

FIG. 3. Time dependences of the damping after deformation for two samples (dark and light symbols, respectively): ○, ●—plastic deformation at $T = 1.62$ K; △, ▲—plastic deformation at $T = 1.24$ K.

exceed 30%. Figure 2b shows examples of the temperature dependences of the damping of the sample of helium before and after the deformation at temperature 1.66 and 1.28 K. In this sample, the plastic deformation led to a small decrease of the damping. In all the experiments, the form of the temperature dependence of the decrement, particularly the decrease of the damping with decreasing temperature, is preserved.

3. Internal friction in samples grown by the blocked-capillary method

Measurements of the internal friction in samples grown by the blocked-capillary methods were made in the container described in Ref. 5 (flexural oscillations of the quartz; the frequency of the quartz + solid helium system was 15 kHz). Attempts to grow a solid-helium sample in the ampoule described in the present paper led to mechanical breaking of the supports of the quartz resonator because of the large mechanical stresses produced in the course of the growth (change of hydrostatic pressure from 61 to 35 atm in the course of the growth). This fact indicates that in the course of the growth the solid-helium sample flowed plastically and after the end of the growth the sample should apparently contain a large number of various defects such as grain boundaries or dislocations.

The frequency of the natural oscillations of the quartz + solid helium system was 15 kHz (the frequency of the oscillations of the system for the helium crystal grown at constant pressure 32 atm in the same ampoule was 15.2 kHz).

The measurement of the time dependence of the decrement started with the instant that the container temperature ~1.3 K was set. The total measurement time reached 160 min. The value of the decrement during this time changed by not more than 20%. The measurements of the decrement have shown that at solid-helium oscillation amplitudes $\varepsilon_m = 3.10^{-8} - 10^{-5}$ the damping does not depend on the amplitude.

The temperature dependences of the internal friction of the two samples are shown in Fig. 4. As seen from this figure, the dependences $\Delta(T)$ are in good agreement with those measured for crystals grown at constant pressure and annealed for several hours at a

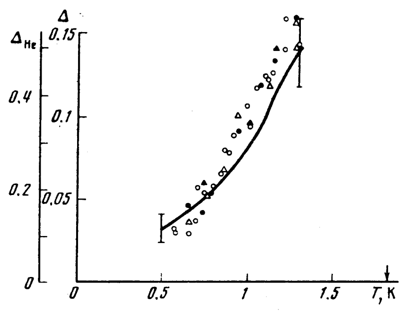

FIG. 4. Temperature dependences of the damping in two samples grown by the capillary-blocking method, measured at decreased (○, ●) and increased (△, ▲) temperatures. Solid line—averaged $\Delta(T)$ dependence in the samples grown at constant pressure; vertical segments show the scatter of the decrement from sample to sample.

temperature 0.05—0.1 K lower than the melting temperature. The temperature dependences measured with a decrease and increase of the temperature are in good agreement.

4. Influence of He^3 impurities on the internal friction

Crystals of a mixture of helium isotopes with concentration 0.1 at.% He^3 were grown at constant pressure in an ampoule with the quartz in flexure. The mixture with concentration of 0.1% He^3 was used to grow crystals also at constant pressure in both types of ampoules.

In the crystals with the 0.1% He^3, we observed an amplitude dependence of the decrement at helium deformation amplitudes exceeding 5×10^{-7}. These dependences were not investigated detail. A typical temperature dependence of the damping is shown in Fig. 5a; the decrement was determined in the amplitude-dependent region. It is seen from the figure that the value of the decrement decreased by 10—30%; the monotonic decrease of the damping remained the same with decreasing temperature.

In crystals with 0.1% He^3 concentration, amplitude dependences of the damping were observed at temperatures 0.55—1.7 K, both in the ampoule with the quartz in flexure as well as in torsion. These dependences were investigated in greater detail in the ampoule with the quartz in flexure. Figure 6 shows a typical plot of the decrement against the oscillation amplitude in such an ampoule. As seen from the figure, at deformation amplitudes less than 10^{-6} the internal friction does not depend on the amplitude. At larger amplitudes the damping increase rapidly. This character of the amplitude dependences was observed in all samples both at 15 kHz and at 78 kHz.

Figures 5a and 5b show the temperature dependences of the damping in the samples with 0.1% He^3; the decrement was determined in the amplitude-independent region. It is seen that the values of the decrement decrease by a factor ~5; with decreasing temperature, the damping decreased monotonically. This character of the temperature dependence was observed in all samples.

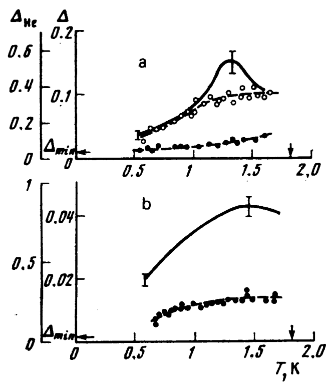

FIG. 5. Plots of $\Delta(T)$ for samples with different He^3 impurity concentration. Solid curves—averaged $\Delta(T)$ dependences in pure samples; the vertical segments show the scatter from sample to sample. a) Measurements at 15 kHz; ○ – 0.01 at.% He^3, ● – 0.1 at.% He^3; b) measurements at 78 kHz, He^3 concentration 0.1 at.%.

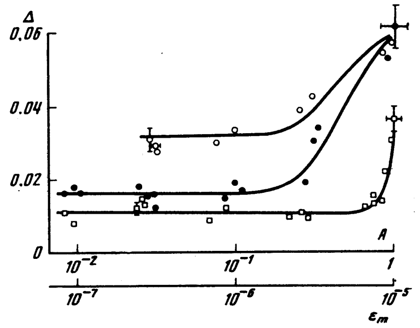

FIG. 6. Amplitude dependences of the damping decrement in sample with 0.1 at.% He^3: ○ – T = 1.62 K, ● – T = 1.21 K, □ – T = 0.56 K. Solid curves—approximation of the points by the relation (6).

DISCUSSION OF RESULTS

The energy loss in half the period of the oscillation of the quartz + solid helium system consists of the loss in the He^4 sample (W_1), in the quartz crystal (W_2), in the container walls, and the loss due to the outward radiation of sound (W_3). The measured damping decrement is

$$\Delta=\frac{W_1+W_2+W_3}{E_1+E_2}=\frac{W_1+W_2}{E_1+E_2}+\Delta_3, \qquad (1)$$

where E_1 and E_2 are the average mechanical energies of the oscillation of the solid helium and of the quartz. The decrement Δ_3, corresponding to the energy loss W_3, can be estimated from the minimum dampings Δ_{min} obtained in the experiment. For the ampoule with the quartz in flexure, $\Delta_3 < 10^{-2}$, and for the ampoule with the quartz in torsion $\Delta_3 < 10^{-3}$. The damping decrement in the helium sample is $\Delta_{He} = W_1/E_1$, and when account is taken that $W_2/E_2 \ll \Delta$, we get from (1)

$$\Delta_{He}=\Delta(1+E_2/E_1). \qquad (2)$$

The ratio of the oscillation energies E_1 and E_2 can be estimated from the resonant frequencies of the oscillations of free quartz f_0 and of the quartz + solid helium system f. The solution of the equation of the oscillations of quartz together with the helium sample yields

$$\Delta_{He}\cong\frac{2\Delta}{1-f_0^2/f^2}. \qquad (3)$$

In the ampoule with the bar quartz $\Delta_{He}\approx 3.6\Delta$ (the value $\Delta_{He}\approx 1.3\Delta$ in the earlier paper[5] is too low), while in the ampoule with the quartz in torsion $\Delta_{He}\approx 20\Delta$. The second scales in Figs. 2, 4, and 5 show the damping in helium, recalculated with the aid of these relations. As seen from these figures, the damping in the helium is large and reaches, at the maxima, values of the order of unity. Since the damping Δ_{vac} due to vacancies is much less than these values (according to the estimates of Meĭerovich[1] $\Delta_{vac} = 10^{-4}-10^{-3}$), the observed damping is apparently due to dislocations.

At the present there is no theory that describes the behavior of dislocations in an He^4 crystal and makes it possible to calculate the dislocation internal friction. Besides the quantum effects, contributions should be made to the internal friction by processes that occur in ordinary bodies: 1) losses due to thermal conductivity; 2) losses due to relaxation processes (for example thermally activated motion of the kinks on the dislocations); 3) losses due to oscillations of a dislocation section between pinning points (the theory of Granato and Lucke[7]).

In the first case the damping decrement, according to our estimates,[5] is less than 10^{-4}. In the second case the temperature dependent dependence of the decrement is determined mainly by the temperature dependence of the relaxation time $\tau_r \sim \exp(q/k_B T)$, and at temperatures below the maximum the damping should decrease exponentially with decreasing temperature. The decrease of the decrement observed in experiment is slower.

In the third case, assuming that the damping constant depends on the temperature in accordance with a power law $B = gT^k$, we can obtain from the theory of Granato and Lucke[7] the temperature dependence of the dislocation decrement Δ_d (see Ref. 5):

$$\Delta_d = \Delta_{0d} \frac{(T/T_m)^k}{1+(T/T_m)^{2k}}, \tag{4}$$

$$T_m \approx \left[\frac{0.53Gb^2}{(1-\nu)\omega g L^2}\right]^{1/k}, \quad \Delta_{0d} \approx 0.22(1-\nu)\Omega\Lambda L^2,$$

where G is the shear modulus, b is the Burgers vector, L is the length of the dislocation loop, Ω is the orientation factor, ω is the angular frequency of the oscillations, ν is the Poisson ratio, and Λ is the dislocation concentration. Reduction by formulas (4) of the temperature dependences of the decrement of pure undeformed samples, measured at ~78 kHz, in the ampoule with the quartz in torsion, yields values of $\Omega\Lambda L^2$, gL^2, and k in the intervals $gL^2 = (1-1.3)\cdot 10^{-13}$ cgs units, $\Omega\Lambda L^2 = 9.3-9.7$, $k = 1.4-1.7$. The values of gL^2 and k are in satisfactory agreement with the same parameters as determined in Ref. 5 ($gL^2 = (5-8)\cdot 10^{-13}$, $k = 2.15-2.85$) and in the paper of Wanner, Iwasa, and Wales[6] ($gL^2 = (1-3)\cdot 10^{-13}$, $k = 1.45-2.36$). This confirms indirectly the correctness of the chosen model. However, the quantities $\Omega\Lambda L^2$ differ greatly; $\Omega\Lambda L^2 = 3.8-6.2$ in Ref. 5 and $\Omega\Lambda L^2 = 0.02-0.1$ in Ref. 6. The causes of this difference are not clear.

Measurements of the damping of ultrasound at frequencies 5—50 MHz in crystals with $V_{mol} = 20.5$ cm^3, carried out by Hiki and Tsuruoka[2] yield dislocation parameters that differ substantially from those given above. Thus, at $T = 1.7$ K they obtained $BL^2 = 1.4 \cdot 10^{-15}$ cgs units and $\Lambda L^2 = 0.36$. They also measured the temperature dependence of the damping in the temperature interval 1.60—1.77 K and found that the frequency at which the maximum damping is observed, $f_{max} \sim 1/BL^2$, does not depend on the temperature. If we assume that the length of the loop L does not change in this temperature interval, then the constant B likewise does not change with temperature. This form of the $B(T)$ dependence differs substantially from that obtained in the present paper, and also from the results of Refs. 5 and 6. For a final answer to this question it is necessary to measure the frequency dependence of the damping in the kilohertz band at different temperatures.

Nor is the cause of the large $\Delta_{He} \sim 1$ damping clear, as well (if the model 3 is correct) the large value of the product $\Omega\Lambda L^2 (\Omega\Lambda L^2 \leq 10)$. The maximum value of the orientational factor Ω is 0.5, meaning that ΛL^2 reaches 20, which is larger by one order of magnitude than that observed in ordinary bodies.

The growth of the sample of solid helium by the block-capillary method is accompanied by plastic flow of the solid helium, and consequently the obtained sample may contain a large dislocation density. The internal states of the samples grown by this method and of the samples grown at constant pressure and then plastically deformed are similar. In either type of sample, the values of the internal friction are close to the values of the dampings obtained for undeformed crystals grown at constant pressure, i.e., the plastic deformation does not influence substantially the internal friction. This behavior of the damping differs substantially from the behavior of the internal friction under plastic deformation of ordinary substances, where even a small plastic deformation leads to a strong increase of the damping (for example, Refs. 9 and 10). The cause of this difference is not clear.

The crystallization temperature of solutions of helium isotopes with He^3 concentration 0.01—0.1% differs from the crystallization temperature of pure He^4 at the same pressure, according to the data of Esel'son *et al.*,[11] by not more than 0.002 K. At the initial of the crystal growth, the concentration of the He^3 in the solid phase is approximately half as large, and at the end of the growth it is approximately one and a half times larger than the concentration of the solution. For example, for a solution with 0.1% He^3, the He^3 concentration in the solid phase will change from ~0.05 to ~0.15%.

The presence of He^3 impurities leads to an amplitude dependence of the damping decrement; in the amplitude-independent region, the dampings are approximately a fifth as small as the dampings in pure samples. A similar phenomenon is observed in ordinary materials (e.g., Ref. 12) and is attributed to the decrease of the length of the dislocation loop because of blocking of part of the dislocation by the impurity. The amplitude-dependent internal friction was theoretically considered by Granato and Lucke,[7] who derived an expression for the decrement Δ_A:

$$\Delta_A = \frac{c_1}{\varepsilon_0}\exp\left(-\frac{c_2}{\varepsilon_0}\right), \quad c_1 = \frac{4(1-\nu)}{\pi^3}\Omega\Lambda L_N^2 \frac{L_N}{L_C} c_2,$$

$$c_2 = \frac{K\eta b}{L_C}, \tag{5}$$

where L_N is the dislocation length between the nodes of the dislocation grid, L_C is the average length of the dislocation loop, $K = G/4RE$, E is Young's modulus, η is the Cottrell gap, and R is the shear-stress reduction coefficient.

In our geometry ε_0 depends on the coordinate along the ampoule: $\varepsilon_0 = \varepsilon_0(z)$ (in a plane perpendicular to the ampoule axis, ε_0 is constant, according to estimates, accurate to ~20%). Recognizing that the average mechanical energy of the oscillation of the solid helium is proportional to the square of the amplitude, we get

$$\bar{\Delta}_A = \int_{-l/2}^{l/2} \Delta_A(\varepsilon_0)\varepsilon_0^2(z)\,dz \Big/ \int_{-l/2}^{l/2} \varepsilon_0^2(z)\,dz, \quad (6)$$

where l is the length of the quartz resonator. The $\varepsilon_0(z)$ dependence is obtained from the solutions of the equations of the oscillations of the quartz + solid helium system and if the forces and torques on the ends of the quartz are neglected, it will be the same as in the case of free oscillations of quartz (for a claculation of $\varepsilon_0(z)$ see Ref. 13).

The experimental points in Fig. 6 were approximated by the relation (6) (solid curves). As follows from the figures, the points agree satisfactorily with the relation (6). At 1.62 K the values obtained were $c_1 = 6.5 \cdot 10^{-7}$, $c_2 = 8 \cdot 10^{-6}$; at $T = 0.56$ K we have $c_1 = (2-8) \cdot 10^{-4}$, $c_2 = 6.5 \cdot 10^{-5}$. From (5) we can calculate L_C, L_N, and Λ; the value of $\Omega\Lambda L_N^2$ was taken from the present paper as well as from Refs. 5 and 6. The values of L_C lie in the interval $L_C \approx 3 \cdot 10^{-5}$ cm at $T = 1.62$ K and $L_C \approx 4 \cdot 10^{-6}$ cm at $T = 0.56$ K.

The decrease of L_C with temperature can be attributed[7] to the increase of the concentration of the impurity atoms on the dislocation: $L_C \propto \exp(-T_0/T)$. For T_0 we obtain the value $T_0 \sim 1.8$ K, which agrees in order of magnitude with the value $T_0 \sim 5$ K calculated from the formula given in Ref. 14. At temperatures $T \sim T_0$ we have $L_N \sim L_C \sim 10^{-4}$ cm, and the concentration of the dislocations is $\Lambda = 10^{10} - 10^{8}$ cm^{-2}, whereas in undeformed crystals of ordinary substances we have 10^2 cm^{-2} $< \Lambda < 10^6$ cm^{-2}. The possible cause of this different is that formulas (5) do not hold for dislocations in solid helium. Another possible explanation is that the concentration of the dislocations in the undeformed helium crystals, even those grown at low rates at constant pressure, is actually large ($\Lambda \sim 10^{10}$ cm^{-2}). Further plastic deformation therefore does not increase substantially the density of the dislocations and the internal friction does not change substantially, as is confirmed by experiment. However, this assumption does not agree well with the measurements of the thermal conductivity of undeformed helium crystals,[15] from which it follows that the phonon mean free path in such samples is comparable at low temperatures with the crystal dimensions.

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