Observation of a glassy phase of ⁴ He in the region of supersolid effects

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High-precision pressure measurements in solid ⁴He, grown by the capillary blocking technique, have been made in temperatures range from 50 to 500 mK. The temperature dependence of pressure indicates that aside from the usual phonon contribution $\sim T^4$, there is an additional contribution $\sim T^2$, the latter becoming dominant at temperatures $T < 300$ mK, where an abnormal behavior attributed to supersolidity has been observed. The data suggest the appearance of a glassy phase (that might be responsible for the anomalous behaviors observed previously). A dramatic pressure decrease has been observed under annealing of the samples. The glassy contribution to the pressure can be eliminated in well-annealed crystals.

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I. INTRODUCTION

The concept of supersolidity in quantum crystals stated in Refs. [1–](#page-4-1)[3](#page-4-2) assumed the concurrent existence of crystalline order and superfluidity. It was shown in Ref. [1](#page-4-1) that the point defects in quantum crystals are delocalized and become the quasiparticles which can move in a gaslike manner within their energy band. The width of the band Δ is determined by the tunneling frequency of the defects. In this case, the occurrence of superfluidity is associated with the so-called zero-point vacancions which can appeared if the half width of the energy band equals the energy of formation of the localized vacancy *W*. As this takes place, the vacancy concentration $x_v \sim \exp[-(W - \Delta/2)/T]$ ceases to decrease with lowering the temperature. Under such conditions, the Bose condensation and superfluidity may be realized.

This interesting idea has immediately attracted the attention of experimentalists, but the first experiments on solid 4 4 4 He (Refs. 4[–8](#page-4-4)) have given no positive results. The upper limit for the vacancy concentration at low temperatures is estimated on the basis of an analysis of various experimental data. In particular, it is evident from NMR data that x_v 10^{-14} at *T*=0.5 K (see Ref. [9](#page-4-5)) means, in fact, that such type of superfluidity cannot be observed.

However, recently, Kim and Chan observed nonclassical moment of inertia in their torsion experiments in solid ⁴He at $T \le 0.2$ K.^{[10](#page-4-6)} The effect was interpreted as manifestation of supersolidity, initiating a new tide of interest to the phenomenon. Independent torsion experiments carried out at different laboratories $11-13$ $11-13$ confirmed the anomalous behavior of solid 4 He at $T < 0.2$ K. These experiments also revealed some new peculiarities, namely, a sample history dependence of the effect and a significant influence of crystal annealing that may result even in a complete suppression of the effect. At the same time, the search for superfluid flow in solid ⁴He with pressure difference provided no positive result for highquality crystals, $14,15$ $14,15$ but the superflow has been registered in the poor-quality crystals.¹⁵

Despite numerous theoretical studies, $16-26$ there is still no unique understanding of the above processes in solid ⁴He. The majority of the authors concluded that supersolidity is impossible in an ideal quantum crystal, $16,17,20-23,26$ $16,17,20-23,26$ $16,17,20-23,26$ $16,17,20-23,26$ $16,17,20-23,26$ and the observed effects are associated with disorder (grain bound-aries, etc.).^{[17,](#page-4-13)[20](#page-4-14)[,22](#page-4-16)[,23,](#page-4-15)[26](#page-4-12)} Suggestions of the existence of superfluid²⁰ and nonsuperfluid^{24[,25](#page-4-18)} glassy states in solid helium and their possible role in the effects observed have been recently made.

The most typical manifestation of the glassy state is a linear contribution to the temperature dependence of heat capacity. Though the heat capacity of solid 4 He was studied rather comprehensively, the situation with its temperature dependence at low temperatures remains controversial. Previous experiments $27-29$ $27-29$ revealed a linear contribution to heat capacity but the subsequent measurements $30-33$ $30-33$ did not find it. Note that the analysis of data of Ref. [33](#page-5-3) made in Ref. [24](#page-4-17) showed a little linear contribution in heat capacity. The difference between the results was supposed to be due to the qualities of the samples studied. It should be borne in mind that the contribution of the linear term to the heat capacity observed at low temperatures in Refs. [27–](#page-5-0)[29](#page-5-1) accounts only to several percent of the calorimeter heat capacity, making its reliable determination difficult.

Heat capacity *C* and pressure *P* of the thermodynamic system are connected by the Mie-Grüneisen equation, which in the pressure of several subsystems takes the form (see, for example, Ref. [34](#page-5-4))

$$
\left(\frac{\partial P}{\partial T}\right)_V = \sum_i \frac{\gamma_i C_i}{V},\tag{1}
$$

where *V* is the molar volume and γ and C_i are the Grüneisen parameter and the heat capacity of the *i*th subsystem. It enables us to find the glassy contribution through pressure measurements. Since linear contribution to heat capacity corresponds to the quadratic-in-temperature contribution to the system pressure, it is convenient to measure pressure to reveal a glassy contribution.

The advantage of such measurements is the high precision. In that case, there is no contribution equivalent to that of an empty calorimeter to heat capacity. The search for the glassy contribution to the thermodynamics of helium crystals by measuring precisely the pressure in different quality specimens is the goal of the present work.

II. EXPERIMENTAL PROCEDURE

The measurements were carried out with the experimental unit previously used to study ³He-⁴He solid solutions and described in detail in Ref. 35 . The 4 He samples in the form of a disk 9 mm in diameter and 1.5 mm high were inside a cooper cell located on the plate of the refrigerator mixing chamber. The cell upper cap made of beryllium bronze acts as a movable electrode of the Straty-Adams capacitive pressure gauge that measured the crystal pressure *in situ* with a ±3 Pa resolution. During calibration of the pressure gauge, we found a weak temperature dependence of the empty cell capacitance. The remedy for this fact is described in the Appendix. Temperature was measured with a $RuO₂$ resistance thermometer calibrated by the crystallization thermometer based on the 3 He melting curve.

The samples were prepared with the use of commercial purity ⁴He by the capillary blocking technique which, as rule, leads to the formation of a polycrystalline sample. To reduce the ³He possible content under producing a high pressure with an adsorption gasificator, the first portion of desorbed helium was removed by pumping, leaving behind no more than 10 ppm ³He in the sample. This is evidenced by the absence of phase separation-induced peculiarities of pressure down to 50 mK.

The typical experimental procedure was as follows. Immediately, upon completing the crystallization, the sample was cooled at a maximum possible rate about 30 mK/min down to \sim 1 K to obtain a maximum concentration of defects. Further cooling was made mostly in a steplike manner with a 30–100 mK step to temperatures below 100 mK. Following the isothermal ageing for several hours at a minimum temperature, the crystal was heated in a steplike manner. Then, the sample was annealed by exposing to a temperature that was \sim 10 mK below the melting point after which the above procedure was repeated. In some cases, the heating was, on purpose, performed up to a temperature at which the annealing does not begin yet, and the sample was recooled to determine the degree of reproducibility of the results. The experiments were carried out on 19 different crystals in a pressure range of 28–43 bar.

III. ANALYSIS OF THE TEMPERATURE DEPENDENCE OF PRESSURE

Figure [1](#page-1-0) shows a typical temperature dependence of relative pressure for a freshly grown crystal during its first cooling (curve 1). To analyze the results obtained, the contributions of different subsystems to the total pressure of solid helium are considered.

As a rule, the temperature dependence thermodynamic properties of helium crystals is well described by the model in which the total pressure is considered as a sum of phonon $P_p(T)$ and vacancy $P_{\text{vac}}(T)$ contributions,³⁶

$$
P(T) = P_0 + P_p(T) + P_{vac}(T),
$$
 (2)

where P_0 is the pressure in the crystal at $T=0$. The phonon contribution can be written down as

FIG. 1. Typical temperature dependence of relative pressure during cooling the freshly grown sample of solid ⁴He (solid line 1 is plotted by the experimental points). Curve 2 is the phonon contribution. Curve 3 is glassy contribution (molar volume of 20.23 cm³/mol).

$$
P_p(T) = \frac{3\pi^4}{5} \frac{\gamma_\Theta}{V} R \frac{T^4}{\Theta_D^3},\tag{3}
$$

where Θ_D is the Debye temperature, $\gamma_{\Theta} = -(\partial \ln \Theta / \partial \ln V)$ is the Grüneisen constant, R is the gas constant, and V is the crystal molar volume.

At low temperatures (below \sim 0.5 K), the vacancy contribution to pressure becomes negligible, and the main contribution is made by the phonon subsystem. If one assumes that the abnormal behavior of solid ⁴He observed in various experiments at $T< 0.2$ K is associated with the formation of a glassy phase, the latter should produce a contribution as P_g $=a_gT^2$, where the coefficient a_g is determined by the density and distribution of tunnel states of glass.

Then, the total pressure of the system at low temperature can be given as follows:

$$
P(T) = P_0 + P_p(T) + P_g(T) = P_0 + a_p T^4 + a_g T^2.
$$
 (4)

To emphasize the existence of the quadratic term in the temperature dependence of pressure, Eq. ([4](#page-1-1)) can be rewritten as

$$
(P - P_0)/T^2 = a_g + a_p T^2.
$$
 (5)

Figure [2](#page-2-0) shows such dependences as a function of T^2 for two samples. The adequate linear dependences suggest the existence of the Debye contribution, and the intercepts on the ordinate axis correspond to the a_g values. However, Eq. ([5](#page-1-2)) is not convenient for quantitative treatment because the values of $(P-P_0)/T^2$ corresponding to different temperatures have nonequivalent accuracy, and introducing weighting factors actually requires Eq. ([4](#page-1-1)). For this reason, Eq. (4) was used in subsequent calculations.

The preliminary processing experimental data have shown that Debye contributions to pressure of the samples are rather close to the values calculated with using the universal relations for Θ_D , which were found in Ref. [36.](#page-5-6) Therefore, under further processing, the value of P_p was determined by using

FIG. 2. $[P(T) - P_0]/T^2$ vs T^2 for the freshly grown ⁴He crystal with molar volume of 20.23 cm^3/mol (curve 1) and annealed crystal with molar volume of $19.83 \text{ cm}^3/\text{mol}$ (curve 2).

Eq. ([3](#page-1-3)), and the value of a_p , according to Ref. [36,](#page-5-6) had been calculated as $a_p = 1.322 \times 10^{-9} V^{5.534}$ bar K⁻⁴ *(V* in cm³/mol).

For $P_0 \ge P_p$ and $P_0 \ge P_g$, it is possible to obtain for the difference of the pressure gauge capacitances ΔC under pressure $[C(P)]$ and $P=0$ $[C(0)]$,

$$
(\Delta C)^{-1} = [C(P) - C(0)]^{-1} = \alpha - \beta T^2 - \gamma T^4,\tag{6}
$$

where the parameters α , β , and γ (see Appendix), in the range under investigation, can be considered to be independent on temperature and pressure, and

$$
a_g = \frac{\beta}{\gamma} a_p. \tag{7}
$$

The advantage of Eq. ([6](#page-2-1)) is the independence of ΔC on the value and temperature dependence of parasitic contributions to the measured capacity, as well as the possibility of carrying out the processing without the preliminary gauge calibration. In Fig. 3 , the dependence given by Eq. ([6](#page-2-1)) for

FIG. 3. (Color online) Temperature dependence of $(\Delta C)^{-1}$ for the crystal with molar volume $V=20.35$ cm³/mol.

TABLE I. Mean value of coefficient a_g , determining the glassy contribution to pressure.

The characteristic of the sample	$\begin{array}{c} a_g \\ (10^3 \end{array}$ $bar K^{-2}$)	Root-mean-square deviation $(bar K^{-2})$	Number of runes
Fresh-prepared samples	2.85	0.5	13
Partially annealed	2.51	0.3	11
Carefully annealed	0.48	0.2	13

one of the samples is presented. The points correspond to experimental data and the curve to the approximation.

The 13 samples have been processed according to the procedure described, and in the rest of the samples, relaxation processes were observed, i.e., the change of pressure at constant temperature (see Sec. IV). The obtained values of a_g have shown rather wide scatter, especially in the case of nonannealed samples. Such a scatter is quitely expected as the value of a_{ϱ} is connected with defect concentration in the sample which we practically could not control. In this case, the evident pressure dependence of a_g was not observed; at different *P*, the values of a_g are within the scattering.

The average values of a_{ϱ} along with the root-mean-square deviation and number of the processed dependences are placed in the table for three types of samples, namely, for fresh-prepared crystals, partially annealed (time of annealing in the vicinity of melting curve is less than 10 h), and well annealed.

We can see from Table [I](#page-2-3) that quasiglass behavior in helium crystals is connected with the formation of steady defects at crystallization by a method of a capillary blocking, which can be eliminated only by long annealing in immediate vicinity to a melting curve. The coincidence of the values of *ag* in the first two lines of the table, within the limits of inaccuracy, supports this supposition. Partial annealing has resulted, mainly, in lesser scatter of the data. At the same time, the careful annealing practically eliminates the glass contribution.

Our value of a_{ϱ} for nonannealed samples can be correlated with corresponding value of the coefficients found from heat-capacity measurements. However, the quantitative comparing is problematic because the value of Grüneisen coefficients for glassy phase is unknown. If one supposes that they have the same order as those for phonon subsystem, our values of a_{ϱ} , within a scatter, correspond to linear contribution in heat capacity found in Ref. [29](#page-5-1) being, however, 1 order of magnitude larger than that found in Ref. [24](#page-4-17) after processing data.³³

The contributions of phonon and glassy components to $P(T)$ are shown in Fig. [1](#page-1-0) (curves 2 and 3). As is evident, the glassy contribution becomes dominant at $T < 0.3$ K, i.e., in the temperature range where the "supersolid" effect was observed.

IV. RELAXATION AND ANNEALING

The experiments on the initially nonequilibrium fresh samples revealed several types of relaxation behavior. Most

FIG. 4. The kinetics of variation in pressure and temperature of the sample in process of annealing. Molar volume of 19.83 cm^3/mol .

of the samples exhibit no considerable relaxation not only on the first cooling but also on the subsequent heating until the temperature approaches the melting point T_m by 100–10 mK. The time behavior of relaxation that is characterized by variations in pressure on the samples heating up to $T-T_m \sim 10$ mK is illustrated in Fig. [4](#page-3-0) where five typical regions are clearly seen. Region 1 corresponds to the initial state (no relaxation) when the sample was at constant temperature (500 mK) and pressure (43.8 bar). Then, the temperature was increased to that of annealing and the pressure first rose with increases in phonon and vacancy contributions with temperature (region 2) and then it decreased when the relaxation contribution was in excess of the influence of temperature rise (region 3). In that case, the sharp reduction in pressure at the first stage gave way to a stage of slow relaxation (region 4). Finally, the pressure reached equilibrium in region 5.

Note a considerable difference in pressure, $\Delta P \sim 2$ bar, before and after annealing at the same temperature. Assuming that the samples studied have an ordinary compressibility of solid helium, this ΔP corresponds to a variation in density by approximately 0.5%. This effect can hardly be caused by the disappearance of vacancies or dislocations, since it is very unlikely to have that high concentration of such defects. We conjecture that the effect is due to metastable liquid or glass trapped in "pockets" in the course of fast crystallization. The existence of such pockets on the measuring cell walls was proposed in Ref. [37.](#page-5-7) Estimating the amount of liquid necessary to quantitatively explain the effect, we get \sim 5%. This value is too large to be attributed to cell walls, and one comes to the scenario of liquid or glassy cavities in the bulk of the sample. It is tempting to associate the state of ⁴He in these regions with metastable disordered state of quenched ⁴He observed in first-principles Monte Carlo simulations by Boninsegni *et al.*[20](#page-4-14) We also note that the estimated concentration of liquid or glassy inclusions turns out to be close to the superfluid fraction found in Ref. [10.](#page-4-6)

The data for one of the annealed samples are presented in Fig. [2](#page-2-0) (curve 2). In this experiment, the measurements were performed under slow heating and all experimental points were processed. The large number of the points compensated their scattering and the experimental error in finding the parameters was practically the same (within 5%) as that under handling the averaged data. The value of *ag* was estimated 1 order of magnitude smaller than in nonannealed crystals. This suggests that annealing of the crystal influences strongly the glass phase contribution.

It should be mentioned that the annealing effect is highly dependent on the duration of annealing in region [4](#page-3-0) (Fig. 4). If the region duration was 1 or 2 h, the variation in a_{φ} did not exceed the normal scatter. The above two results are consistent with assumption that the main reduction in pressure is caused by the disappearance of pockets and that the defects responsible for the glass state disappear at the stage that follows.

In some samples, the pressure relaxation occurred even during the first cooling, sometimes immediately after the onset of cooling, and sometimes at subsequent stages. One of the samples displayed no relaxation on cooling but it started on heating up to 300 mK. It should be noted that at each temperature level, the pressure was reduced only to a certain value and the next decrease in pressure occurred only with the next increase in temperature. The relaxation process will be discussed in detail elsewhere.

V. CONCLUSION

We performed high-precision pressure measurement in solid ⁴He samples grown by capillary blocking technique. In all nonannealed hcp crystals, the temperature dependence of pressure demonstrates a contribution proportional to T^2 , the latter becomes the leading term at temperatures *T* 300 mK, at which "supersolid effects" were observed. Such a behavior may be ascribed to a glassy phase. We find that this glassy contribution to pressure can be eliminated only by a substantial annealing.

A dramatic pressure decrease of \sim 2 bar was observed under annealing at temperatures very close to the melting point. We conjecture that this effect is due to solidification of liquid or glass captured in closed cavities during the growth process. To quantitatively account for the pressure drop, the volume fraction of these regions should be as high as \sim 5%.

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APPENDIX: THE CONNECTION OF PRESSURE WITH CAPACITY CHANGE OF THE CAPACITOR PRESSURE GAUGE

Usually, for the capacitor pressure gauges of Strati-Adams type, the pressure value P is calculated by the equation (see, for example, Ref. [38](#page-5-8)),

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$$
P \sim \left(\frac{1}{C_0(0)} - \frac{1}{C(P)}\right),\tag{A1}
$$

where $C_0(0)$ and $C(P)$ are the capacities of the capacitor at zero pressure and at the pressure *P*, respectively. However, Eq. $(A1)$ $(A1)$ $(A1)$ appears to be incorrect if the measured capacity includes the additional background component C_b which does not dependent on pressure. Let us assume

$$
C_0(0) = C_{00} + C_b, \tag{A2}
$$

where $C_{00} = (a/d_0)$ is the capacity of the main capacitor, d_0 is the distance between the capacitor plates at $P=0$ (for circular plates with radius *R*, if one neglects edge effects, $a=R^2/4$. Then, for $C(P)$, one can write

$$
C(P) = \frac{C_{00}}{1 - \frac{\Delta d(P)}{d_0}} + C_b
$$
 (A3a)

or

$$
\Delta C = C(P) - C_0(0) = \frac{C_{00} \Delta d(P)}{d_0 \left(1 - \frac{\Delta d(P)}{d_0}\right)},
$$
 (A3b)

where $\Delta d(P)$ is the change of distance between plates after applying the pressure P , which we assume to be a linear function of pressure $\Delta d(P) = kP$. Then, from Eq. ([A3b](#page-4-20)), we obtain

$$
\frac{1}{\Delta C} = \frac{1}{C_{00}} \left(\frac{d_0}{k} \frac{1}{P} - 1 \right). \tag{A4}
$$

Relationship $(A4)$ $(A4)$ $(A4)$ permits us to determine constants entering into it after plotting the dependence ΔC^{-1} vs P^{-1} . As for the values C_{00} , d_0 , and k in helium temperature range, we can consider them to be independent of temperature. Then, it is sufficient to calibrate the pressure gauge at one temperature value only. It should be noticed that Eq. $(A4)$ $(A4)$ $(A4)$ remains also valid in the case when the value C_b depends on temperature.

If one supposes the temperature dependence of the total pressure of the system as that given by Eq. ([4](#page-1-1)) and considers that at low temperature, the strong inequalities $P_0 \ge a_p T^4$ and $P_0 \ge a_g T^2$ are satisfied, one obtains

$$
\frac{1}{\Delta C} = \frac{1}{C_{00}} \left[\frac{d_0}{k P_0} \left(1 - \frac{a_g}{P_0} T^2 - \frac{a_p}{P_0} T^4 \right) - 1 \right] = \alpha - \beta T^2 - \gamma T^4,
$$
\n(A5)

where

$$
\alpha = \left(\frac{d_0}{kP_0} - 1\right) \frac{1}{C_{00}}, \quad \beta = \frac{d_0 a_g}{kP_0^2 C_{00}}, \quad \gamma = \frac{d_0 a_p}{kP_0^2 C_{00}}.
$$

Then, the parameter a_g is determined by Eq. ([7](#page-2-4)). Hence, after processing experimental data on temperature dependence of ΔC and using the known data on a_p , we determine the glass contribution to pressure. It should be noted that such method does not require preliminary calibration of pressure gauges at all.

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