PHYSICAL REVIEW B **83**, 132201 (2011)

Search for a disordered phase in solid ^{3}He deformed *in situ*

A. A. Lisunov, V. A. Maidanov, V. Yu. Rubanskyi, S. P. Rubets, E. Ya. Rudavskii,[*] A. S. Rybalko, and V. A. Tikhiy
B.I.Verkin Institute for Low Temperature Physics and Engineering, NAS of Ukraine,
47, Lenin Av., Kharkov 61103, Ukraine

(Received 28 December 2010; revised manuscript received 28 January 2011; published 5 April 2011)

A disordered (glassy) state has been searched for in solid ^{3}He deformed in the course of the experiment through precise measurements of pressure. The analysis of the temperature dependence of the crystal pressure measured at a constant volume shows that the main contribution to the pressure is made by the phonon subsystem, with the influence of the disordered phase being very weak. Annealing of the deformed crystal does not affect this state. The results obtained differ greatly from the corresponding data for solid ^{4}He measured in the region of supersolid effects, where a pressure excessive in comparison with the phonon one was registered. The excess pressure had a quadratic dependence on temperature, which is typical of a disordered system. Absence of the distinct excess pressure in solid ^{3}He is yet unclear; some speculative interpretations are suggested.

DOI: 10.1103/PhysRevB.83.132201 PACS number(s): 67.80.bd, 67.80.dj

I. INTRODUCTION

Recently, an unusual behavior of solid ^{4}He has been observed in the region where a special state of quantum crystal, supersolidity, was predicted. The first manifestation of this state was observed in the torsion experiments[1] as a nonclassical rotational moment of inertia. Then an anomalous shear modulus,[2] a specific heat peak,[3] and unusual mass transfer[4] were registered in the same temperature region. The fraction of nonclassical rotational inertia was very sensitive to the crystal quality and decreased sharply after annealing,[5] which suggests a nonsuperfluid origin of the supersolid effects.[6] Those effects might be connected to the formation of a disordered (glassy) state in solid helium.

It has been found that the disordered state can be registered suitably using the technique of precise measurements of the crystal pressure.[7–9] The performed experiments showed that, in solid ^{4}He, the pressure in the region of expected supersolidity, apart from the phonon contribution proportional to T^4, takes a contribution proportional to T^2, which is typical of a disordered (glass) phase. Below $T \sim 200$ mK, this contribution was much higher than the phonon one, but the glass phase disappeared after thorough annealing of the crystal.

Note that these features were observed in solid ^{4}He. As concerns the second isotope, ^{3}He, which is a Fermi solid instead of a Bose solid, the first torsion experiments[1] showed an absence of the nonclassical rotational inertia. In the experiment,[1] solid ^{3}He was confined in a porous Vycor glass, therefore the torsion measurements were repeated later[10] without the porous matrix. The absence of the effect was confirmed, which illustrates the importance of quantum statistics. In the meantime, a surprising result was obtained in acoustic measurements on solid ^{3}He:[10] the shear modulus of ^{3}He changed, just like in ^{4}He, in the hexagonal closed pack (hcp) phase but remained normal in the body-centered cubic (bcc) phase. It is likely that the difference in the "supersolid" behavior of the solid helium isotopes is caused not only by the type of statistics, but by the type of crystal structure, defects, or other factors as well.

It was therefore reasonable to search for the disordered state in both phases of ^{3}He and compare the results obtained with the corresponding experimental data for ^{4}He. The goal of this study was to investigate this problem through precise measurements of pressure of helium crystals at a constant volume. The experimental conditions were similar to those for solid ^{4}He.[9] The temperature dependences of the pressure measured after deformation and thorough annealing of the sample were then compared.

II. EXPERIMENTAL TECHNIQUE

Like in the case of solid ^{4}He, the disordered phase was searched for in solid ^{3}He samples deformed in the course of the experiment using the same two-chamber cell.[9] The samples were grown in one of the chambers in the form of a disk that was 0.5 mm high and 32 mm in diameter. A beryllium bronze membrane (2 mm thick) separated this chamber from the other (control) chamber. The samples cooled below 100 mK were deformed using the control chamber. For this purpose, liquid ^{4}He was condensed into the control chamber, which permitted cyclic variations of the pressure in it from 0 to $\sim$25 atm. The deformation of the sample due to deflection of the membrane was nonhomogeneous and shear components of stress appeared. The change in the pressure of the solid ^{3}He sample was $\sim$6 atm. Generally, each sample was put through five deformation cycles. The kinetics of pressure variations in the sample and corresponding temperature response are shown in Fig. 1.

The first spike of the temperature is due to the condensation heat released while filling helium into the control chamber. The subsequent slight rises of temperature on each change (decrease or increase) in the pressure are caused by the viscous heating in the filling line.

After the deformation process was completed, the pressure in the control chamber was released to zero and ^{4}He was pumped out with an adsorption pump for 4–6 hours. Note that the pressure of the deformed sample practically came back to its starting value, though the process of relaxation was rather slow (up to 24–48 hours).

The starting ^{3}He contained 0.25% of the ^{4}He impurity and was rectified in a void column (its basic structure is described in Ref. 11). Normally, such columns yield 0.999 997 pure ^{3}He. For lack of an accurate analysis, the upper limit of possible ^{4}He impurities was estimated indirectly. The crystal grown

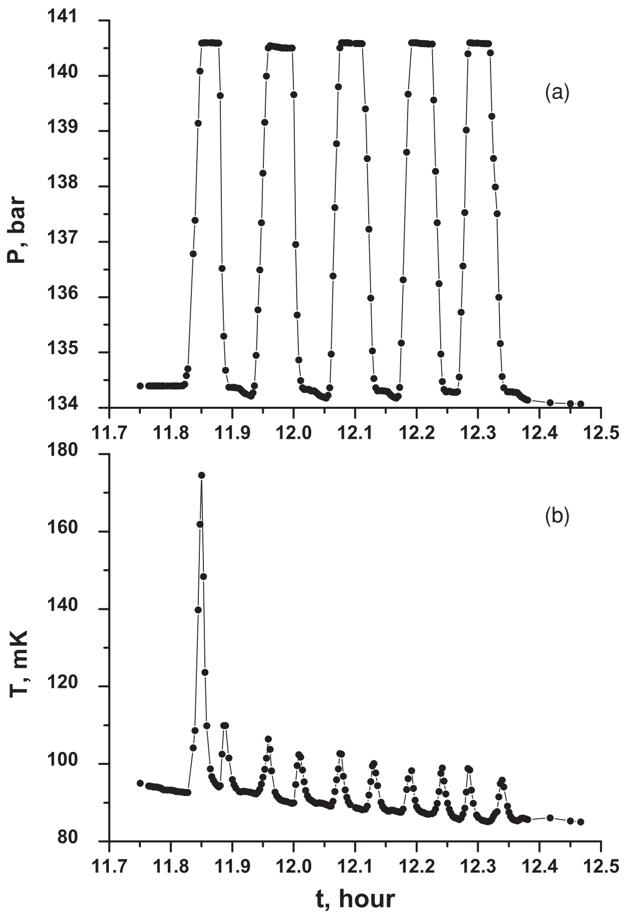

FIG. 1. (a) Kinetics of pressure and (b) temperature variations in the course of sample deformation.

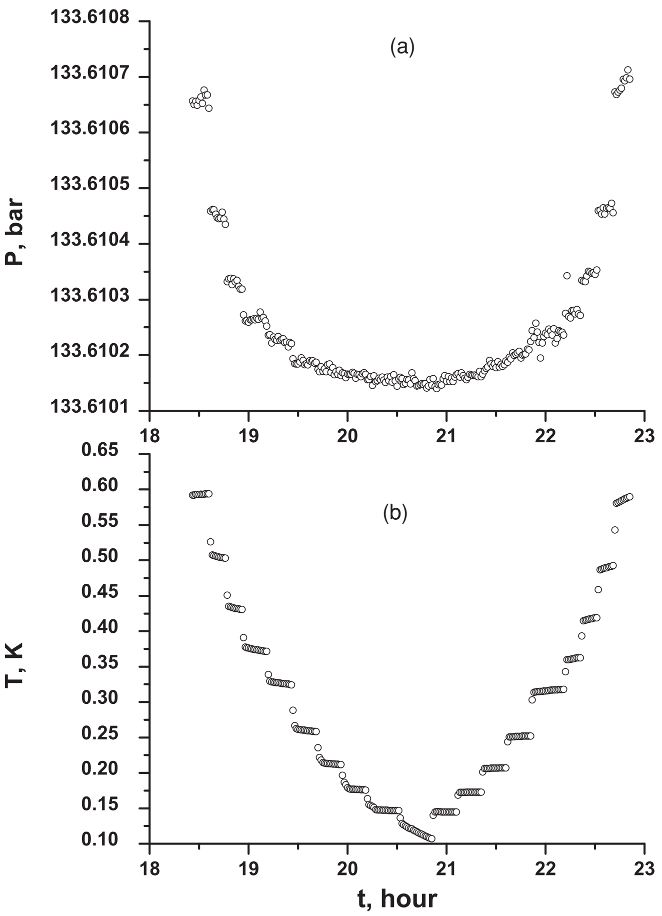

FIG. 2. Typical pressure variations during stepwise cooling and heating.

from the purified ^{3}He, on cooling to 65 mK, had no evidence of phase separation, which suggests the ^{4}He concentration in the sample was below 10 ppm.

The samples were grown by the capillary-blockage technique and investigated in an interval of 100–600 mK. The pressure of the sample was measured with a Straty-Adams capacitive gauge and a precise GR-1615A capacitance bridge, with the accuracy being ±3 Pa. Five samples were investigated in the region of molar volumes 19–22 cm^3/mole, which corresponds to both bcc and hcp phases of ^{3}He. The measurements were performed on deformed samples before and after their thorough annealing for 20–24 hours near the melting temperature.

III. ANALYSIS OF PRESSURE VERSUS TEMPERATURE DEPENDENCE

The temperature dependence of pressure $P(T)$ was measured for a stepwise decreasing and increasing of the temperature. The measurements were repeated several times during cooling and heating. The primary experimental results are illustrated in Fig. 2.

Since vacancy excitations have a negligible effect on pressure in this temperature region, the dependences $P(T)$ obtained on different samples were approximated by the relation

$$P(T) = P_0 + P_{\mathrm{ph}}(T) + P_{\mathrm{d}}(T) \tag{1}$$

where P_0 is the crystal pressure at $T = 0$, and $P_{\mathrm{ph}}(T) = a_{\mathrm{ph}}T^4$ is the phonon contribution to the pressure. The last term describes the contribution of a disordered phase that might be generated by deformation of the ^{3}He crystal. Normally, the temperature dependence of pressure in this phase is $P_{\mathrm{d}}(T) = a_{\mathrm{d}}T^2$.

Since the nucleus of ^{3}He has a spin of 1/2, this contributes to the thermodynamic characteristics of the system at rather low temperatures. The contribution to pressure is proportional to T^{-1} and J^2, where J is an exchange energy,[12]

$$P_{\mathrm{spin}} = (3R\gamma/V)(J/k)^2T^{-1}, \tag{2}$$

and where R is the gas constant, γ is the Gruneisen parameter $\partial lnJ/\partial lnV$, V is the molar volume, and k is the Boltzmann constant. The exchange energy J essentially increases with increasing of the molar volume of solid helium. It means that the nuclear-spin contribution to thermodynamic properties is important only for crystals with large molar volumes or small pressures. Estimation of the P_{spin} according to (2) gives, for the sample under investigation with the largest molar volume, a value of 1 Pa that is less than the experimental accuracy. For our temperature and molar-volume region, the contribution of nuclear spins to pressure is negligible.

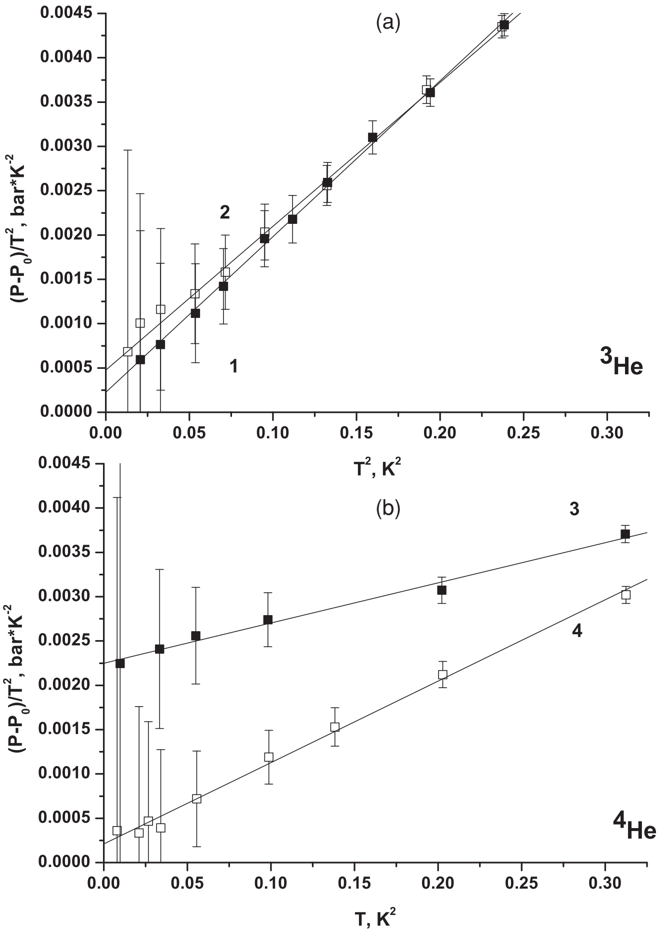

FIG. 3. Typical dependences $[P(T) - P_0]/T^2$ vs T^2 of deformed solid helium samples: (a) solid ^{3}He before (line 1) and after (line 2) annealing (molar volume 22.2 cm^3/mole) (this work); (b) solid ^{4}He before (line 3) and after (line 4) annealing (molar volume 20.0 cm^3/mole) (taken from Ref. 8).

It is then convenient to rewrite Eq. (1) as

$$[P(T) - P_0]/T^2 = a_\mathrm{d} + a_\mathrm{ph}T^2. \qquad (3)$$

As a result, the dependences $P(T)$ replotted in the coordinates $[P(T) - P_0]/T^2$ vs T^2 permit a straightforward estimation of the fitting parameters a_d and a_ph.

Such a dependence of one of the ^{3}He samples is shown as an example in Fig. 3. As was expected, these dependences are straight lines. Their slope determines the parameter a_ph, and the intercept on the ordinate axis yields the parameter a_d. In Fig. 3 the error bars and the scatter in the data increase with decreasing temperature, because the value of $P - P_0$ becomes very small and has the same order of magnitude as the accuracy of measurement.

The lines in Fig. 3 were taken on the deformed samples before and after annealing. It is seen that both lines 1 and 2 coincide within the experimental data scatter and give a_d rather close to zero, which suggests that a disordered phase is practically absent or very small in the crystal.

For comparison, Fig. 3 contains also the corresponding dependences for a solid ^{4}He sample,[8] which show distinctly the presence of a disordered (glassy) phase in the deformed crystal. It is described by the parameter $a_\mathrm{d} = 0.022$ bar/K^2 (line 3). After thorough annealing (line 4), a_d is close to zero, i.e., the contribution of the disordered phase is very small. Thus, unlike the case of ^{4}He, deformation of a solid ^{3}He crystal does not cause the formation of an appreciable disordered phase.

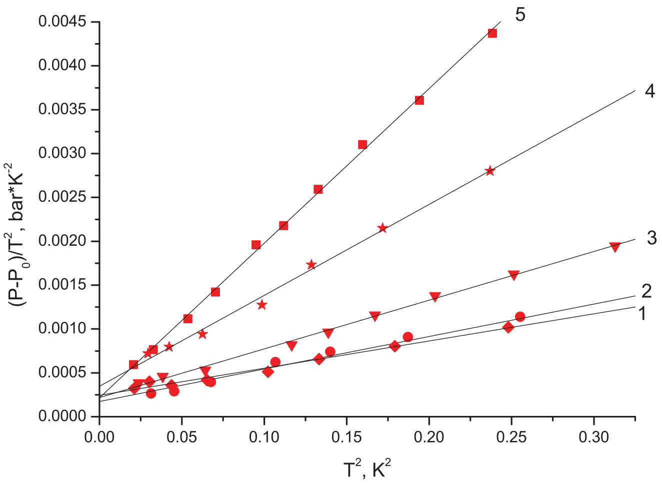

FIG. 4. (Color online) Average values of $[P(T) - P_0]/T^2$ vs T^2 for deformed solid ^{3}He samples of various molar volumes (cm^3/mole): line 1 equals 19.2 (hcp), line 2 equals 19.0 (hcp), line 3 equals 19.8 (bcc), line 4 equals 20.7 (bcc), and line 5 equals 22.2 (bcc).

According to the experimental findings, this is true for all of the five ^{3}He samples (Fig. 4). It should be emphasized that lines 1 and 2 refer to the hcp phase of ^{3}He, while lines 3–5 describe the bcc phase, i.e., the result is independent of the crystalline structure of the solid ^{3}He.

It is natural to compare the obtained temperature dependences of pressure with available data on another thermodynamic characteristic of solid ^{3}He: specific heat. Authors of the first experimental studies of ^{3}He specific heat[13–16] made claims about "low temperature anomaly" at $T < 0.5$ K. This anomaly is characterized by a specific heat in excess of the T^3-dependent specific heat predicted by the Debye phonon theory for an ideal crystal. The excess heat capacity was proportional to T that is typical for a disordered (glassy) phase. However, the subsequent reliable experiment[17] showed no evidence of excess contribution to the specific heat that had been observed in previous measurements, indicating that this anomaly is not due to an intrinsic property of the quantum solid. The author of the paper[16] noted that the origin of the anomaly in the previous works remains a matter of interest.

It should be pointed out that the specific heat measurement has disadvantages in comparison with precise pressure measurements. Specific heat investigation requires one to subtract the rather high value of heat capacity of the empty calorimeter and to take into account the filling effect in the capillary. Therefore the pressure measurement is more suitable; it can be carried out easily under both cooling and heating of the samples.

IV. CONCLUSIONS

The reason deformation of a solid ^{3}He crystal, unlike a ^{4}He one, does not cause a substantial quantity of the disordered phase is still physically obscure and only speculative interpretations can be suggested.

Normally, deformation gives rise to a system of dislocations in a crystal, which may therefore be considered as a key factor in the above effects. The behavior of the pressure in deformed ^{4}He (Ref. 9) was analyzed by calculating the contribution of dislocations to the thermodynamic properties of the crystal,[18] which enabled the authors to describe the experimental results and to estimate the density of dislocations in the crystal. In the case of ^{3}He, the dislocations in the crystal become more mobile because of the larger amplitudes of zero-point oscillations, which may be obstacles to forming and maintaining the required density of dislocations.

The experimental results for ^{4}He (Ref. 9) were also in qualitative agreement with the glass model of two-level tunnel states,[19–22] which predict a quadratic temperature dependence of pressure. The analysis of the experiment within this model allowed estimation of the density of two-level states. Since the microscopic atom model of such two-level states has not been proposed yet, the applicability of this approach for describing the solid ^{3}He behavior remains open.

In connection with different types of quantum statistics of ^{3}He and ^{4}He, the scenario of quasi-one-dimensional superfluidity along the dislocation lines[23–26] in a Bose system should not be ruled out either. The contribution of such a system to pressure is also proportional to T^2.[27] It is natural that this scenario is impossible in solid ^{3}He.

The conclusive interpretation of the results obtained in this study calls for additional theoretical and experimental investigations.

ACKNOWLEDGMENTS

This study was partially supported by STCU (Project No. 5211). The authors are grateful to S. I. Shevchenko, V. D. Natsik, S. N. Smirnov, A. V. Balatsky, M. J. Graf, and A. B. Kuklov for helpful discussions.

---

[*] rudavskii@ilt.kharkov.ua

[1] E. Kim and M. H. W. Chan, Nature (London) **427**, 225 (2004).

[2] J. Day and J. Beamish, Nature (London) **450**, 853 (2007).

[3] X. Lin, A. Clark, and M. H. W. Chan, Nature (London) **449**, 1025 (2007).

[4] M. W. Ray and R. B. Hallock, Phys. Rev. Lett. **100**, 235301 (2008).

[5] Ann Sophie C. Rittner and J. D. Reppy, Phys. Rev. Lett. **98**, 175302 (2007).

[6] J. D. Reppy, Phys. Rev. Lett. **104**, 255301 (2010).

[7] V. N. Grigor'ev, V. A. Maidanov, V. Y. Rubanskii, S. P. Rubets, E. Y. Rudavskii, A. S. Rybalko, Y. V. Syrnikov, and V. A. Tikhii, Phys. Rev. B **76**, 224524 (2007).

[8] E. Ya. Rudavskii, V. N. Grigor'ev, A. A. Lisunov, V. A. Maidanov, V. Yu. Rubanskiy, S. P. Rubets, A. S. Rybalko, and V. A. Tikhii, J. Low Temp. Phys. **158**, 578 (2010).

[9] I. A. Degtyarev, A. A. Lisunov, V. A. Maidanov, V. Yu. Rubanskiy, S. P. Rubets, E. Ya. Rudavskii, A. S. Rybalko, and V. A. Tikhii, JETP **138**, 699 (2010); I. A. Degtyarev, A. A. Lisunov, V. A. Maidanov, V. Yu. Rubanskiy, S. P. Rubets, E. Ya. Rudavskii, A. S. Rybalko, and V. A. Tikhii, JETP **111**, 619 (2010).

[10] J. T. West, O. Syshenko, J. Beamish, and M. H. W. Chan, Nature Phys. **5**, 598 (2009).

[11] R. H. Sherman, *Proceedings of the 10th International Conference on Low Temperature Physics*, edited by M. P. Malkov, L. P. Pitaevski, and Yu. D. Anufriyev (Moscow, 1966), p. 188.

[12] S. Trickey, W. Kirk, and E. Adams, Rev. Mod. Phys. **44**, 668 (1972).

[13] E. S. Heltemes and C. A. Swenson, Phys. Rev. **128**, 1512 (1962).

[14] H. H. Sample and C. A. Swenson, Phys. Rev. **158**, 188 (1967).

[15] R. S. Pandorf and D. O. Edwards, Phys. Rev. **169**, 2228 (1968).

[16] S. H. Castles and E. D. Adams, Phys. Rev. Lett. **30**, 1125 (1973); J. Low Temp. Phys. **19**, 397 (1975).

[17] D. S. Greywall, Phys. Rev. B **15**, 2604 (1977).

[18] V. D. Natsik and S. N. Smirnov, Fiz. Nizk. Temp. **18**, 185 (1992); [Sov. J. Low Temp. Phys. **18**, 128 (1992)].

[19] A. F. Andreev, JETP Lett. **85**, 585 (2007); **136**, 118 (2008).

[20] A. V. Balatsky, M. J. Graf, Z. Nussinov, and S. A. Trugman, Phys. Rev. B **75**, 094201 (2007).

[21] J. J. Su, M. Graf, and A. V. Balatsky, J. Low Temp. Phys. **159**, 431 (2010).

[22] S. E. Korshunov, JETP Lett. **90**, 156 (2009).

[23] S. I. Shevchenko, Sov. J. Low Temp. Phys. **14**, 553 (1988).

[24] D. V. Fil and S. I. Shevchenko, Phys. Rev. B **80**, 100501 (2009).

[25] M. Boninsegni, A. B. Kuklov, L. Pollet, N. V. Prokof'ev, B. V. Svistunov, and M. Troyer, Phys. Rev. Lett. **99**, 035301 (2007).

[26] J. Toner, Phys. Rev. Lett. **100**, 035302 (2008).

[27] S. I. Shevchenko (private communication).