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Elastic properties of solid helium

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Abstract

Following recent torsional oscillator measurements which appear to show the 'non-classical rotational inertia' which characterizes a supersolid, a number of experiments have searched for evidence of unusual behavior in other properties. We have developed a new technique for measuring the shear modulus of solid helium at low frequencies and small strains. In hexagonal close packed ^{4}He, the shear modulus increases dramatically below 200 mK, the temperature range where decoupling is seen in torsional oscillators. The modulus anomaly is frequency independent, depends strongly on strain amplitude, and is very sensitive to ^{3}He impurities. In these and other ways, the shear modulus closely mirrors the torsional oscillator behavior and it is clear that the two phenomena are closely related. We attribute the shear modulus effects to the elastic response of mobile dislocations and their pinning by ^{3}He impurities at low temperatures. A question then arises: are the modulus increases responsible for the frequency changes seen in torsional oscillator experiments? The expected frequency shifts appear to be much too small to explain the apparent decoupling, nor can elastic effects explain the 'blocked annulus' results or the behavior in small pores. In order to clarify the relationship between the shear modulus and torsional oscillator behaviors, we have recently made modulus measurements on ^{3}He, where no supersolid response is expected. Since dislocation motion depends on crystal structure it was important that these measurements be extended to the hexagonal close packed phase of ^{3}He, not just the body centered cubic phase.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Recent torsional oscillator (TO) measurements with solid ^{4}He [1] showed frequency increases at low temperatures which have been interpreted in terms of mass decoupling, i.e. the 'non-classical rotational inertia' (NCRI) which characterizes supersolidity. This effect has been confirmed in TO experiments in a number of laboratories [2–7]. The decoupling is gradual and begins around 200 mK in polycrystals grown with the blocked capillary method using standard ^{4}He gas (containing about 300 ppb of ^{3}He isotopic impurities). The onset is accompanied by a dissipation peak and shifts to lower temperature (around 75 mK) in isotopically pure (1 ppb ^{3}He) crystals [8] and in single crystals [9] grown at constant pressure. The NCRI is amplitude dependent, decreasing rapidly for TO velocities above about 10 μm s^{-1}. The onset temperature increases with frequency but the total NCRI appears to be independent of frequency [5]. The NCRI changes (usually decreasing) when crystals are annealed near melting, suggesting that defects are involved [2]. Although these features are common to the different experiments, the magnitude of the NCRI varies by three orders of magnitude in different cells (from about 0.015% to 16%). The largest NCRI was seen in cells with large surface to volume ratios (i.e. when the helium was in a narrow gap) [2] and in porous media [1, 6] but this variation is not understood.

There have been a number of experiments which looked for other unusual behavior which might be associated with supersolidity. Several, some of which predated the TO experiments, looked for superflow in solid ^{4}He. An early experiment involving solid helium in coexistence with liquid [10] did not show any flow down to 4 mK, but recently a similar experiment [11] did see evidence of superflow for some crystals when grain boundaries were visible (likely occurring along liquid channels where grain boundaries met a glass wall). An early experiment [12] at pressures well above the melting curve (where such channels should not exist) did not show any low temperature pressure relaxation which might signal superflow in solid helium. We recently [13] performed similar measurements with higher resolution and showed that pressure

differences of order 100 mbar did not produce superflow in solid helium. Our experiments put an upper limit on flow velocity of 10^{-12} m s^{-1}, seven orders of magnitude lower than the critical velocities inferred from TO measurements. If helium forms a supersolid, it does not flow in response to pressure differences the way a superfluid would. Some very recent experiments [14] show interesting flow behavior through solid helium (in contact with superfluid on both sides) but these measurements have not yet reached the temperature range where TO decoupling has been seen.

There have also been attempts to look for unusual acoustic behavior. Measurements [15] of the speed and attenuation of longitudinal ultrasound showed interesting but complex behavior which was interpreted in terms of Bose condensation of defects. Other experiments looked for new second sound modes [16] without success. Neutron scattering has been used to look for structural changes [17, 18] or signatures of Bose condensation [19] but without finding signatures of a supersolid state. Thermodynamic measurements could provide evidence of a supersolid transition. Recent heat capacity measurements [20] do show a small, broad peak near the onset temperature for NCRI. However, high resolution measurements of the melting curve pressure [21] do not show any corresponding anomalous behavior. Recent review articles summarize the experimental [22] and theoretical [23] status of the field: there are many puzzles, but an emerging consensus that defects, like dislocations or grain boundaries, are involved and perhaps essential in supersolid behavior. New experiments which complement the torsional oscillator NCRI measurements could clarify the role of such defects.

Solids differ most obviously from liquids in their elastic properties—they have a non-zero shear modulus μ which allows them to sustain pressure gradients without flow and which supports transverse sound modes. It is therefore interesting to look at the behavior of the shear modulus of solid helium under the conditions where NCRI was observed in TO measurements. We have developed a technique [24] to directly measure μ at low temperatures (down to 17 mK), low frequencies (20–2000 Hz) and small strains (ε as low as 10^{-9}). We found [25] a large and unexpected increase of μ in hcp ^{4}He in the temperature range where NCRI was seen. Its dependences on temperature, frequency, amplitude, ^{3}He concentration and sample annealing are so similar to those seen in torsional oscillators that the shear stiffening must be closely connected to the NCRI seen in TO measurements. The behavior of μ can be explained in terms of the elastic response of a dislocation network which is pinned by ^{3}He impurities at the lowest temperatures but which becomes mobile as the ^{3}He atoms thermally unbind from dislocations above 100 mK. Given the close connection between μ and NCRI behavior, it is important to see how the shear modulus depends on statistics (^{4}He versus ^{3}He) and on crystal structure (hcp versus bcc). In this paper, we show the behavior of μ in hcp ^{4}He and compare it to that in ^{3}He.

2. Experimental details

Helium crystals were grown using the constant density ('blocked capillary') method, which is expected to produce polycrystalline samples with many defects. We used both standard ^{4}He gas (containing ~0.3 ppm ^{3}He) and isotopically pure ^{4}He (1 ppb ^{3}He). The ^{3}He crystals were grown from gas with 1 ppm ^{4}He. The cell included a Straty–Adams capacitive gauge which allowed us to measure the pressure *in situ* with a resolution of 0.2 mbar. Elastic displacements were generated and stresses were detected [24, 25] by two rigidly mounted piezoelectric shear transducers (PZT 5A material, width W = 9.6 mm, length L = 12.8 mm, thickness t = 2.1 mm, fundamental resonance at 500 kHz). The gap D between their front faces was 180 μm for most of the ^{4}He crystals (and 500 μm for the ^{3}He measurements). A voltage V applied to the driving transducer (at frequency f) produces a proportional shear displacement δx at its front face and thus a shear strain $\varepsilon = \delta x/D$ in the solid helium in the gap. The resulting shear stress σ produces a charge on the second transducer and thus a current I, which we measured with an ultra-low-noise current preamplifier (14 fA pHz^{-1} equivalent input noise current) and a digital two-phase lock-in amplifier. The minimum detectable stress is set by noise in our preamplifier (~2.5 fA for 30 s averaging), giving $\sigma \sim 10^{-5}$ Pa at 2000 Hz (which corresponds to a displacement $\delta x \sim 2 \times 10^{-16}$ m and strain $\varepsilon \sim 10^{-12}$). The shear modulus of the solid helium is $\mu = \sigma/\varepsilon$, which is proportional to the measured ratio I/f.

3. Results and discussion

Figure 1 shows the temperature dependence of the shear modulus (normalized to the value at the lowest temperatures) for a hcp ^{4}He crystal at a pressure of 33.3 bar (melting temperature T_m = 1.86 K). The modulus, measured at a frequency of 200 Hz and at low strain ($\varepsilon = 2 \times 10^{-8}$), changes by less than 0.5% between 1 K and 200 mK. However, at lower temperatures there is a large (~8%) increase in μ. The pressure in the cell remains constant within 0.2 mbar, indicating that there is no comparable change in bulk modulus. The onset temperature (around 150 mK) and the shape of this unexpected stiffening closely resemble the typical NCRI measured in TO measurements. For comparison, figure 1 also shows the NCRI (i.e. the change in the frequency—roughly 910 Hz—of the torsional oscillator) in a 65 bar ^{4}He crystal [1].

Figure 2 shows the frequency dependence of the shear modulus changes seen in figure 1. The bottom three curves compare the behavior at 20 and 2000 Hz to that at 200 Hz. The measurements at the three frequencies were made at the same strain (2×10^{-8}) and have been offset vertically for clarity. The magnitude of the increase in μ does not change but the transition is slightly sharper at 20 Hz and significantly broader at 2000 Hz (where the onset temperature is above 200 mK). This frequency dependence is essentially the same as was seen in a TO measurement at 496 and 1173 Hz [5]. We were not able to extend our direct shear modulus measurements to higher frequencies because of an acoustic resonance in the solid helium filling the cell around 8000 Hz. However, we could measure the frequency f_r of this resonance, which is roughly proportional to the shear sound speed: the upper curve in figure 2 shows this resonance frequency (right axis). Its temperature dependence is similar to that of the directly

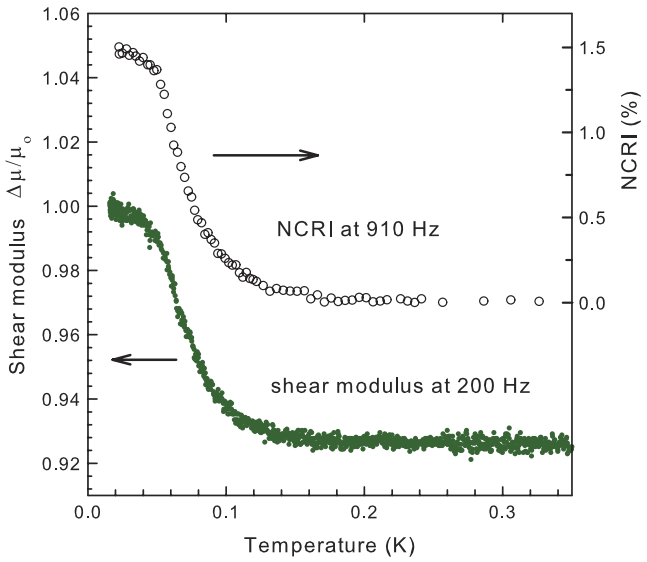

Figure 1. Temperature dependence of shear modulus and NCRI in hcp ^{4}He.

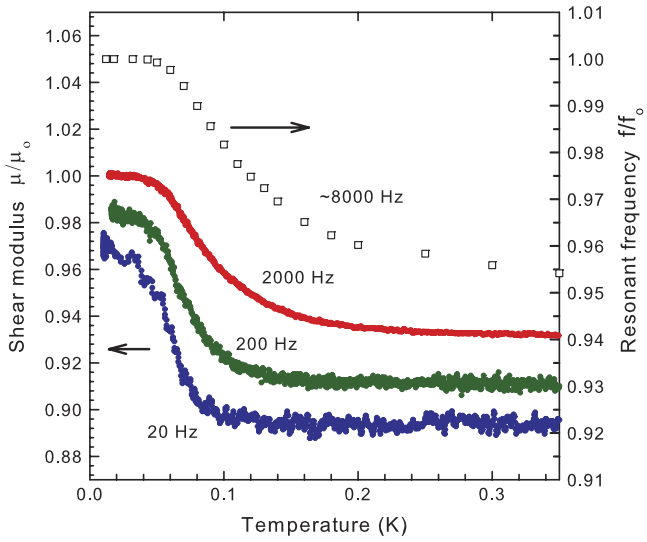

Figure 2. Frequency dependence of shear modulus and acoustic resonance frequency in hcp ^{4}He.

measured shear modulus but the low temperature increase is smaller (only about 4%), as expected since sound speeds are proportional to the square root of elastic constants. The transition is even broader than at 2000 Hz.

Given these similarities between the behavior of the shear modulus and the TO frequency, it is interesting to know whether μ is as sensitive to amplitude as the NCRI. Figure 3 shows the shear modulus changes at 2000 Hz at different amplitudes (the curves have been shifted slightly to agree at 0.3 K). The strains were calculated from the applied voltage using the piezoelectric coefficient of the drive transducer and range from $\varepsilon = 2.2 \times 10^{-9}$ to 1.7×10^{-6} (with corresponding stresses in the helium from $\sigma = 0.03$ to 25 Pa). For strains below about 4×10^{-8} (which corresponds to a stress of about 0.6 Pa), the modulus is independent of amplitude.

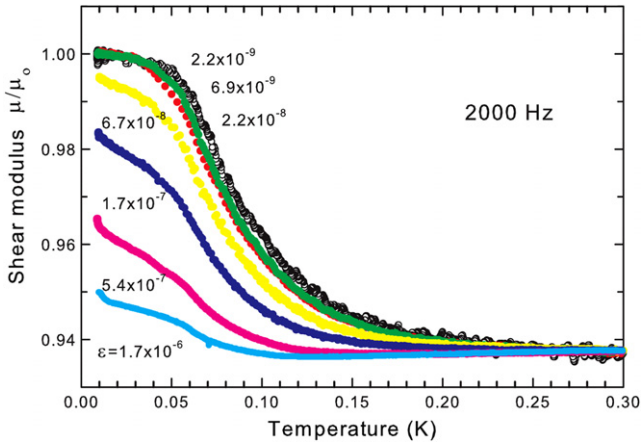

Figure 3. Strain amplitude dependence of the shear modulus anomaly in hcp ^{4}He.

At higher strains, the anomalous low temperature stiffening decreases rapidly, almost disappearing at the highest drive level. The onset temperature for the stiffening also shifts to lower temperature as the amplitude increases (from above 200 mK at the lowest drives to about 100 mK at the highest drive). This amplitude dependence is very similar to that seen in TO experiments [1], where it is interpreted in terms of critical velocity for flow in a supersolid. To distinguish between a critical velocity and a critical stress/strain (or a critical displacement) in our experiments, we made the same measurements at a lower frequency, 200 Hz. The results were essentially identical, with the μ anomaly beginning to decrease at essentially the same strain, $\sigma = 5 \times 10^{-8}$. If the amplitude dependence scaled with velocity rather than strain, it would have appeared at a strain 10 times larger at the lower frequency. This seems to be different from the behavior of the NCRI, where measurements [5] at 496 and 1173 Hz appear to show an amplitude dependence which scales with the velocity.

Another striking feature of the TO measurements is the sensitivity of the NCRI onset temperature to ^{3}He impurity concentrations below 1 ppm. In addition to measurements with ^{4}He of standard isotopic purity (roughly 300 ppb ^{3}He), we have grown crystals from isotopically pure ^{4}He (1 ppb ^{3}He). Figure 4 shows the effect of ^{3}He concentration. The onset of the shear modulus stiffening shifts from above 200 mK in the 300 ppb ^{3}He samples to below 100 mK in 1ppb ^{3}He crystals. The magnitude of the stiffening does not seem to change—it varies by about a factor of two from one sample to the next but is always of order 10% for both standard and isotopically pure samples. The modulus changes in figure 4 have been normalized by their total changes at low temperature (18 mK), in order to compare their temperature dependences. This remarkable variation with very small ^{3}He concentration is essentially the same as is seen [8] in the NCRI in TO experiments.

In order to understand the role of defects, we studied the effects of annealing. For the 33.3 bar sample of figures 1–3, annealing for 15 h at 1.70 K (160 mK below T_m) reduced the modulus anomaly $\Delta\mu$ by about 20%. However, it was the high temperature behavior which changed: the values

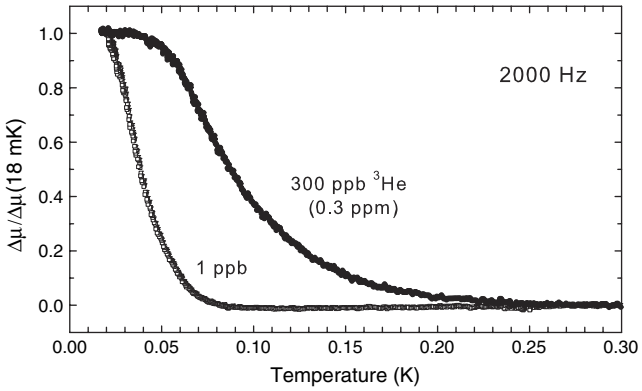

Figure 4. Effect of ^{3}He impurities on the shear modulus of hcp ^{4}He.

of μ (and acoustic resonance frequency f_r) at the lowest temperature were virtually unaffected. For example, annealing changed the resonance frequency at 18 mK only by 0.1% but at 400 mK it increased by about 4%. It appears that the low temperature values reflect the intrinsic shear modulus while the high temperature modulus is reduced by defects.

The shear modulus changes we see are orders of magnitude larger than expected in defect-free crystals and it is difficult to imagine small concentrations of point defects having such large effects. However, dislocations (one-dimensional structural defects created during crystal growth or deformation) can dramatically affect elastic properties, even at low concentrations. They form three-dimensional networks characterized by their density Λ (total dislocation length per unit volume) and their average distance L between pinning sites. They are strongly pinned at nodes where they intersect (with a network pinning length L_N) and can also be pinned, less strongly, by impurities. The impurity pinning length L_P is determined by the binding energy E_B between the impurity and a dislocation, the temperature and the impurity concentration x. When a shear stress is applied, dislocations respond by bowing out between pinning sites and produce an extra strain [26]. At low frequencies this reduces the shear modulus by an amount $R\Lambda L^2$ (where R is an orientation factor which is roughly 0.2 for the random orientations expected in polycrystalline samples). If the only pinning sites are the nodes of the network, the combination ΛL_N^2 depends on the geometry of the network but can be essentially independent of the dislocation density in well-annealed crystals (e.g. $\Lambda L_N^2 = 3$ for a simple cubic network). Even at low densities, dislocations can reduce the shear modulus by large amounts—by 40% in one experiment on solid helium [27]. When impurities are added, L_P can become smaller than L_N, greatly reducing the dislocation strain and increasing the shear modulus toward its intrinsic value. At temperatures below E_B, impurities condense onto dislocations, effectively pinning them at sufficiently high concentration (when the average distance between impurities on dislocation lines, L_P is smaller than the network length L_N). This crossover between network and impurity pinning occurs at lower temperatures for lower impurity concentrations.

Our results are consistent with this picture. Ultrasonic measurements [28, 29] on helium single crystals give dislocation densities $\Lambda \sim 10^6$ cm^{-2} (polycrystals are expected to have higher densities) and values of $R\Lambda L^2$ range from about 0.01 in ultrasonic experiments to 1.0 in a low frequency measurement [27]. The dominant slip system for hcp ^{4}He is edge dislocations gliding in the basal plane and ^{3}He impurities bind to these with E_B in the range 0.3–0.6 K. Using values $E_B = 0.6$ K, we estimate [25] that 300 ppb of ^{3}He impurities will pin dislocations at temperatures below 110 mK, which decreases to 54 mK for 1 ppb of ^{3}He. These are close to the temperatures where μ increases. Large stresses can tear dislocations away from ^{3}He pinning sites and reduce the shear modulus. The critical stress for this breakaway can be estimated [26] as about 4 Pa for $L_P = 5$ μm. This corresponds to strain $\varepsilon \sim 3 \times 10^{-7}$, a level where we see strong amplitude dependence.

The increase in μ, its magnitude and frequency dependence, the temperature at which stiffening occurs and its dependence on ^{3}He concentration, the amplitude dependence—these are all consistent with a picture of a network of dislocations pinned by ^{3}He impurities (using dislocation parameters determined in earlier experiments on hcp ^{4}He). The effects of annealing can be understood in terms of changes in dislocation density Λ, although $\Delta\mu$ depends only on the combination ΛL^2 and so eliminating large numbers of dislocations may have a much smaller effect on μ. Our experiments show that the low temperature modulus is unaffected by annealing, as expected since dislocations are then pinned by impurities so μ recovers to its intrinsic value in a defect-free crystal. The shear modulus decreases when the temperature is raised to the point where ^{3}He impurities thermally unbind from the dislocations, allowing them to move in response to shear stress.

Are the shear modulus changes we see directly related to the frequency shifts in torsional oscillator experiments? Although they measure very different properties (elastic modulus versus moment of inertia), the two sets of measurements share all essential features (dependence on temperature, frequency, amplitude, ^{3}He concentration, annealing). Given these remarkable similarities, the two sets of effects must be closely related. The obvious question is 'how?' One possibility is that the modulus increase we see stiffens the torsional oscillator, increasing its frequency and mimicking mass decoupling. Although this must occur, numerical calculations with realistic models of different torsional oscillators [30] show stiffening effects that are at least an order of magnitude too small to explain the observed decoupling. Also, the period change due to stiffening of solid helium in typical oscillators would scale as the square of the operating frequency, but the total decoupling is independent of frequency [5]. Finally, such an elastic mechanism cannot explain the NCRI in porous media [1, 8] or the elimination of NCRI in 'blocked annulus' experiments [1, 31]. Our shear modulus anomaly does not provide an obvious mechanical, non-supersolid explanation of the frequency changes seen in torsional oscillators.

This raises the question of how the shear modulus and TO are related. They could both be fundamental properties of a supersolid state, for example if supersolidity occurs along

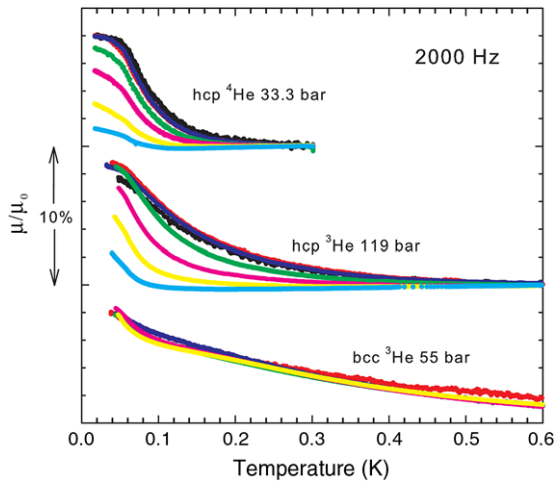

Figure 5. Temperature and strain amplitude dependence of the shear modulus in hcp ^{4}He versus hcp and bcc ^{3}He. Curves are offset vertically for clarity.

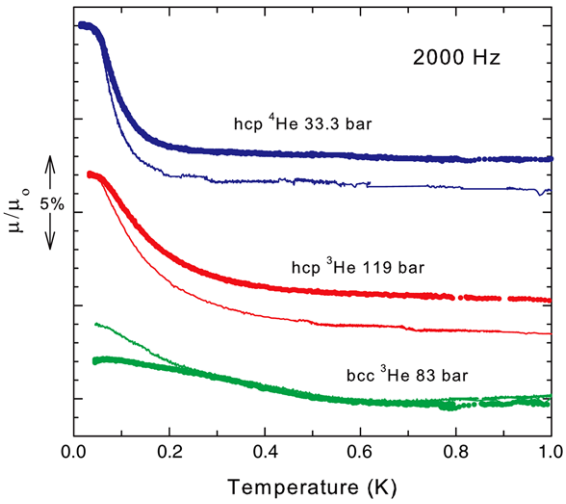

Figure 6. Effect of annealing on the shear modulus: hcp ^{4}He versus hcp and bcc ^{3}He. The thin lines are data taken on first cooling after crystals were grown. The heavier curves were measured after annealing near melting. Curves for different samples have been shifted vertically for clarity.

dislocation networks. Alternatively, the μ anomaly could, as we propose, be due to dislocations becoming mobile, which in turn could affect the supersolid response. A comparison to solid ^{3}He could clarify the roles of defects and quantum statistics in the two sets of properties, since ^{3}He (a fermion) is not expected to exhibit supersolidity but will have dislocations which affect elastic behavior [29]. It is important that a direct comparison be made using ^{3}He of high isotopic purity and with ^{3}He crystals in the hcp phase. Previous TO ^{3}He control experiments [1, 32] were made in the low pressure bcc phase (which extends to zero temperature in ^{3}He). However, dislocation densities and properties depend strongly on crystallographic structure. For example, dislocations in bcc crystals have different slip systems and usually a large Peierls barrier which prevents them from moving at low stresses.

We have used the same techniques to measure the shear modulus of ^{3}He crystals. Figure 5 compares the temperature and amplitude dependence of μ (measured at 2000 Hz) for hcp ^{4}He (the 33.3 bar data of figure 3) to that for bcc (55 bar) and hcp (119 bar) ^{3}He. The two hcp samples show the same behavior—shear modulus anomalies with similar magnitudes (8% and 9% for ^{4}He and ^{3}He, respectively) and temperature dependences. The amplitude dependences are essentially the same (the different curves represent similar strain amplitudes for the two samples). The modulus is independent of strain at low amplitudes but the μ anomaly decreases and shifts to lower temperatures at large strains. The transition in ^{3}He is broader and begins at higher temperature (around 400 mK), which may be due to its higher impurity concentration (1.35 ppm ^{4}He, nearly five times larger than the ^{3}He concentration in the ^{4}He sample). The behavior of bcc ^{3}He crystals is completely different, with no anomalous stiffening at low temperatures. The modulus does not show any systematic amplitude dependence over the same range of strains. This behavior is consistent with our interpretation of the ^{4}He modulus anomaly, since dislocation properties should be similar in hcp ^{3}He but quite different in bcc ^{3}He.

We also studied how the shear modulus is affected by annealing, which is expected to reduce the number of defects and change the density of the dislocation network. As shown in figure 6, the size of the low temperature anomaly in hcp ^{4}He and hcp ^{3}He crystals is changed by annealing. In both cases, the low temperature value of μ is virtually unaffected but the high temperature modulus does change, as expected for annealing of a dislocation network. The bcc ^{3}He crystals do not behave in the same way. Annealing did not change the high temperature behavior, but reduced the temperature dependence at low T. For bcc crystals, the effect of annealing varied from sample to sample but was never like that seen in hcp crystals. Instead of a low temperature anomaly which decreases upon annealing, the bcc crystals had a more significant background and less stable temperature dependence, but it varied from crystal to crystal and did not change systematically during annealing.

The similarities in the shear modulus behavior of hcp ^{3}He and ^{4}He and the differences for bcc ^{3}He are consistent with a dislocation origin for the low temperature stiffening, since dislocation structure and mobility depend strongly on crystal structure but not directly on quantum statistics. This leads to a very interesting question: will torsional oscillator measurements with hcp ^{3}He show a low temperature anomaly like that in the shear modulus? If so, then the apparent mass decoupling and NCRI in ^{4}He may simply reflect the effect of dislocations on elastic properties. If not, then the inertial effects probed in torsional oscillator measurements indeed depend on whether it is a Bose or Fermi quantum solid—the most interesting possibility.

Acknowledgments

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