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Torsional oscillator experiment on superfluid ^{4}He confined in a porous alumina nanopore array

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Abstract. We studied superfluidity of liquid ^{4}He confined in an array of well-characterized straight nanopores of porous alumina (PA). The PA plate sample of 45 nm pore size is set in an annular flow channel and the superflow is detected by torsional oscillator (TO) technique. Superfluid transition T_c in the nanopores is suppressed by 3.5 mK from the bulk λ point. T_c is consistent with the temperature at which the healing length is equal to the pore radius. We have observed many anti-crossing anomalies in the TO frequency associated with dissipation peaks, which are attributed to the coupling to second sound resonances.

Superfluid ^{4}He confined in nanoporous materials has been attracting renewed interests. Recent studies show that superfluidity of ^{4}He confined in a nanoporous glass (with pore size $d = 2.5$ nm) is strongly suppressed and T_c approaches 0 K at a critical pressure [1, 2]. This behavior is understood as a quantum phase transition (QPT). Moreover, we have shown by a heat capacity study that the QPT occurs by the emergence of the so-called localized Bose-Einstein condensation (LBEC) state, in which the macroscopic phase coherence is lost between many nanoscale condensates by the confinement of ^{4}He to nanopores.

Interestingly, the suppression of superfluidity by nanoscale confinement is never explained by the traditional concept of the superfluid healing length ξ, in which T_c is determined by the condition $\xi \sim d$, because ξ of superfluid ^{4}He is about 0.3 nm, much smaller than the pore size. The anomalous superfluid suppression is interesting not only theoretically [3, 4], but also in realizing a true superfluid Josephson junction (JJ). Josephson effect in superfluid ^{4}He has been observed only in the very vicinity of the λ point T_λ, where ξ becomes comparable to the size of the currently available apertures[5].

We aim at developing a novel Josephson device working at arbitrary temperature based on the superfluid suppression in the nanopores. To realize a well defined JJ, a plate that has an array of nanopores whose axes direct normal to the surface has to be prepared. We employ porous alumina (PA) as such a device. PA is produced by anodic oxidization of aluminum plate. By optimizing the oxidization condition, a triangular array of straight nanopores with a uniform pore size can be formed. Since the pore size of PA is typially $10 \sim 100$ nm, PA itself is not appropriate for the ^{4}He JJ. However, one can decrease the pore size at the end of the pores by depositing other materials such as gold. As a preliminary study, we have fabricated and characterized PA, and examined superfluid properties of ^{4}He in a PA plate.

The PA plate having nanopores boring through the plate is prepared as follows: An Al plate is

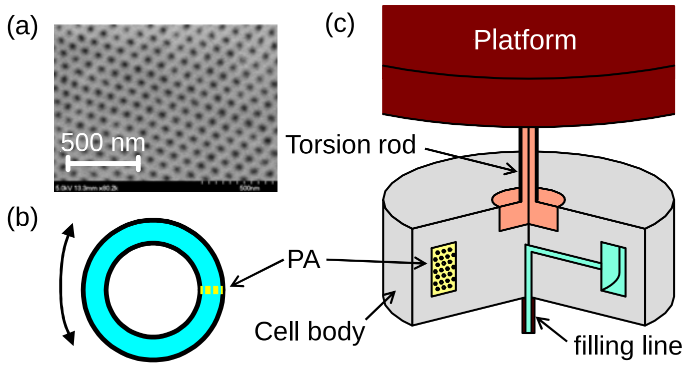

Figure 1. (a) SEM image of porous alumina. The pore diameter is 45 nm, and the distance between the center of two adjacent pores is 100 nm. (b) Schematic cross-sectional view of the torsional oscillator containing the PA plate. (c) Schematic view of the cell setup.

anodized in an oxalic acid. Figure 1 (a) shows a SEM image of the PA. We optimize the condition so as to form a regular honeycomb array of straight nanopores as seen in the photograph. The pore diameter is estimated to be 45 nm and the distance between the pores is 100 nm. Since the nanopores in PA terminate at the opposite side to the oxidized planes, the remaining Al and alumina at the pore ends are removed by a phosphoric acid. By this final procedure all the nanopores pierce through the plate. The final thickness of the PA plate is 165 μm.

We have examined superfluidity of ^{4}He by a torsional oscillator (TO).Figure 1 (b) shows the cross section of the oscillator. The oscillator bob contains an annular conduit of 1.5×1.0 mm^2 cross sectional area. The perimeter of the conduit is about 50 mm. The conduit is filled with bulk liquid He and acts as an "AC" flow channel when the bob oscillates around the torsion axis. The PA plate is inserted in the channel so that the axis of nanopores the flow.

This setup was adopted from flow experiments of superfluid ^{3}He [6, 7]. Unlike the ordinary TO technique in which the superfluid fraction is related to the change in period, the response of the present TO does not correspond to the superfluid component, but to the superflow inside the nanopores. Since the viscous penetration length of liquid ^{4}He is much larger than the pore size, the normal ^{4}He liquid inside the PA plate block perfectly the flow of the bulk liquid in the channel. The moment of inertia of the bulk liquid entirely contribute to the resonant frequency of TO. Below the superfluid transition temperature T_c of liquid *inside* the pores (not at bulk T_λ), the superfluid component can flow in the nanopores. The flow velocity field is determined so that the circulation along the annular channel (through one of the nanopores) is quantized, i.e. zero. The resonant frequency of the TO should show a step-like increase just below T_c due to the occurence of "AC" superflow in the nanopores and the bulk channel.

The torsional oscillator is mounted on a dilution refrigerator. In order to stabilize the temperature around 2 K, it in fact acts as a ^{4}He cryostat by thermally connecting the 1 K pot to the TO. Temperature was calibrated at the superfluid transition temperature of bulk ^{4}He. The resonant frequency f is about 1170 Hz and Q of the resonance is about 8×10^4 at low temperature. The TO is operated using a feedback circuit with a constant driving voltage (force). In the present study, pressure of ^{4}He liquid is set at 0.1 MPa.

Figure 2 shows the shift in frequency from the value at T_λ, $\Delta f(T) \equiv f(T) - f(2.17\text{K})$ and dissipation Q^{-1} derived from the amplitude of the TO. As T decreases, f increases abruptly at 2.17 K, then many anti-crossing resonances appear accompanied with sharp dissipation peak. At the lowest temperature (1.67 K), the difference of Δf reaches about 1 Hz. The magnitude of Δf is consistent with the frequency shift that is estimated from the abovementioned condition, in which all the superfluid in the annulus apparently decouple from the oscillation.

The response of TO reflects more clearly the superfluid properties by looking at a blowup of $\Delta f(T)$ near T_λ shown in Fig. 3. The temperature is plotted as $\Delta T \equiv T - T_\lambda$. The frequency decreases with decreasing temperature above T_λ, and shows a sharp minimum at T_λ, then abruptly increases at temperature 3.5 mK below T_λ, which is denoted as T_c. The small, positive

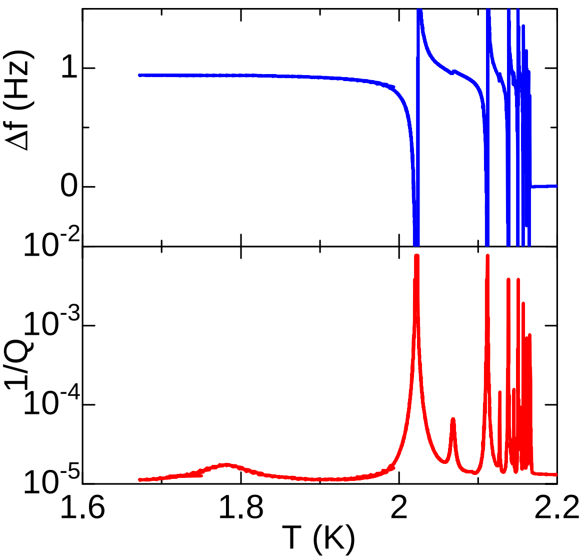

Figure 2. Temperature dependence of the resonance frequency f and the dissipation Q^{-1}. Resonance frequency is subtracted with the frequency at T_c.

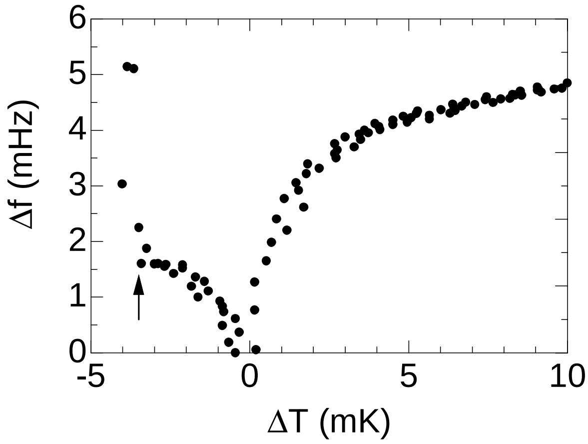

Figure 3. Blowup of Δf as a function of temperature from T_λ, $\Delta T \equiv T - T_\lambda$. Arrow indicates T_c in the nanopores.

Δf between T_λ and T_c is most probably due to the potential flow of superfluid component with a boundaty condition of the annulus with a partition of PA and normal fluid. Since Δf immediately reaches about 0.7 Hz just below T_c, it is clear that the superflow occurs inside the nanopores keeping with the quantized circulation condition along the perimeter of the annulus. Therefore, T_c is firmly identified to be the superfluid transition temperature inside the nanopores.

The healing (coherence) length ξ at T_c is estimated to be 23 nm, which is exactly equal to the pore radius. Therefore, the suppression of superfluidity in the present nanopores is understood well as the effect of suppression the superfluid order parameter near the pore wall, which has been theoretically formalized as the Ψ theory [8–10].Smaller pore size is obviously needed to realize our idea of the JJ by the large superfluid suppression.

Below T_c, a sequence of anomalies is seen in the temperature sweep measurements. The semi-log plot of Fig. 4 clearly shows more than fourteen anti-crossing resonances in Δf accompaning with absorption peak seen in Q^{-1}. These anomalies are attributed to the excitation of second sound resonances by torsional oscillation. It is well known that oscillating superleak can excite second sound [11]. Since the present pore size is much smaller than the viscous penetration length for normal component at all temperatures, the PA plate works as an excellent superleak.

In the annulus channel, the second sound standing waves of wavelength λ should be excited at the resonance condition $\lambda = 2L/n$, where L is the perimeter of the annulus and n is an integer. It is easily inferred that each of many anti-crossing anomalies may correspond to one of the standing waves. Moreover, most of the anomalies are located at rather high temperatures $2\text{K} < T < T_\lambda$. This is understood that the second sound velocity greatly depends on temperature in this regime.

Most interestingly, there are two different types of anomalies, which can be assigned to "large" and "small" resonances from their strengths of the signals. They emerge alternately. We speculate that the large and small modes are standing waves with odd and even n, respectively. In our annulus TO setup, the "ends" of the second sound resonators are regarded as "connected"; i.e. the front and back of the single PA plate act as two transducers. Suppose that the superleak

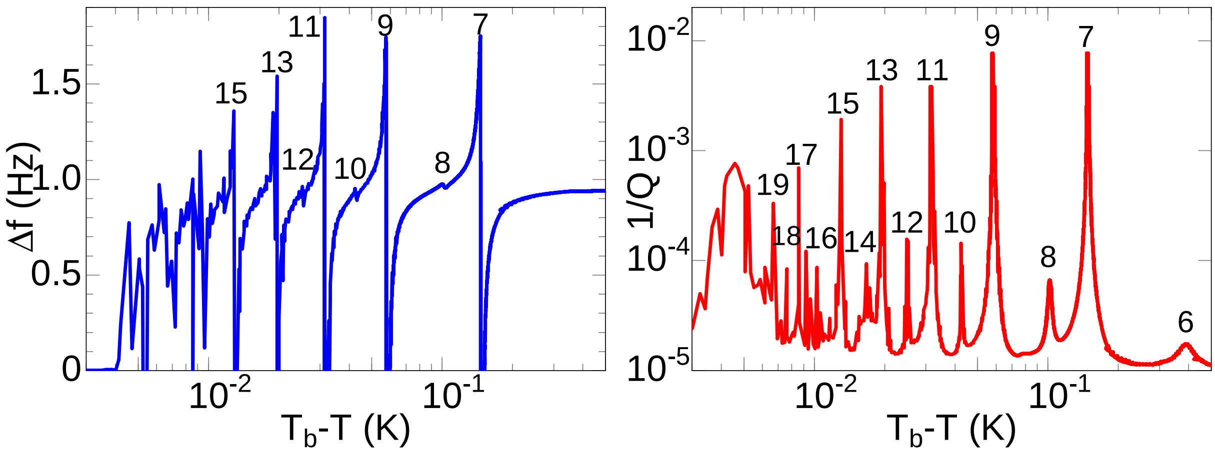

Figure 4. Δf and Q^{-1} as a function of $\log(T_\lambda - T)$. Two kinds of anomalies are seen: "Small" anomaly is always located between two adjacent "large" anomalies.

moves to the right: On the right side of the superleak, the normal fluid density ρ_n increases whereas the superfluid density ρ_s decreases, while on the left side, ρ_n decreases and ρ_s increases. In this situation, the standing waves must have anti-nodes whose phase are opposite on the front and the back of the PA plate, and hence n is odd. Since such waves can easily be excited, larger anomalies are assigned to be this odd $- n$ resonances.

Since the smaller anomalies are located between the larger ones, they can be assigned to be even $- n$ standing waves. In the even $- n$ standing waves, the phases of the two anti-nodes on both PA surfaces are same. The same phase can be realized if the superleak generates heat from viscosity of the normal component. From the data of second sound velocity [12], we assign the anomalies from $n = 6$ to 19.

In conclusion, we have made a porous alumina plate with an array of nanopores of 45 nm in diameter, and have carried out torsional oscillator measurements for confined liquid ^{4}He. The suppression of T_c at 0.1 MPa is 3.5 mK, which comes up to expectation from previous studies. Smaller pore size is needed for realizing the Josephson junction. We attempt to contract the pores by depositing other materials such as gold or carbon on porous alumina.

We have observed a number of anti-crossing resonances due to the coupling of second sound standing waves and torsional oscillation. Torsional oscillator can be an excellent tool for superfluid studies employing second sound.

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