# Fabrication of Nanofluidic Cavities for Superfluid <sup>3</sup>He Studies

## CNF Project Number: 1520-07

Principal Investigator: Jeevak M. Parpia

Users: Abhilash Thanniyil Sebastian, Nikolay Zhelev, Roberto DeAlba

Affiliation: Department of Physics, Cornell University Primary Source of Research Funding: National Science Foundation Contact: jmp9@cornell.edu Website: http://parpia.lassp.cornell.edu Primary CNF Tools Used: Furnaces, Oxford PECVD, contact photolithography, Oxford RIE, Plasma-Therm RIE, Unaxis DRIE, critical point dryer

### Abstract:

We demonstrated nanoscale cavities that withstand 30 bar cooled to ultralow temperatures [1]. The surfaces of silicon-glass cavities are pristine having AFM characterized roughness under 1 nm [1,2]. We also examined the resonant properties of single layer graphene structures above and below room temperature. Despite extensive cleaning, there is evidence of residual polymer coatings on the surface [3].

#### **Summary of Research:**

Superfluid <sup>3</sup>He is a unique system for study. <sup>3</sup>He is a Fermion (like electrons), but its pairing into the superfluid state is more complex than its electronic counterparts producing a multiplicity of superfluid phases. In the bulk, the anisotropic A phase and the isotropically gapped B phase emerge. Confinement favors the A phase over the B phase [2,4].

The superfluid state is attained between 0 and 35 bar and between 0.9 and 2.5 mK (respectively). Below the superfluid transition, pairs condense into the coherent superfluid state. The pairing length-scale (pair diameter) varies from ~ 80 nm at 0 bar to 14 nm at high pressure. Confinement alters the phase diagram and as the <sup>3</sup>He



Figure 1: A. The bonded heat flow channel (5 mm square) with dimensions alongside. B. Typical quartz fork. C. Schematic of experiment. D. Photograph of experimental chamber [5]. See full color version on pages xxviii-xxix.

is progressively restricted to smaller sizes, the B phase should yield to the A phase and new phases should emerge.

In one series of experiments [5], we explore the thermal conductivity of confined <sup>3</sup>He. Two chambers are separated by a micromachined channel of dimensions  $3 \text{ mm} \times 100 \text{ }\mu\text{m} \times 1.1 \text{ }\mu\text{m}$  (Figure 1a). To fabricate this channel, 1 mm thick silicon wafers were oxidized using the oxide furnace in CNF to grow thick oxide  $(> 2 \mu m)$ , then further oxide was deposited using the Oxford PECVD. Long channels with a width of 3 mm were patterned using contact photolithography and the oxide within them removed using both dry plasma etch (Oxford and Plasma-Therm RIE) and wet etch (6:1 BOE). Wafers were further oxidized to create a step in the Si-SiO<sub>2</sub> interface (modified LOCOS process) and oxide removed using HF. To define the channels further laterally to 100  $\mu$ m × 3 mm, a second contact lithography step was used and the exposed Si was etched using the Unaxis DRIE to 200  $\mu$ m depth. After removal of masking oxide, resist, and Bosch polymer, wafers were further oxidized with an oxide film target of 300 nm to protect surface during dicing and then diced using CNF's dicing saw. Matching glass pieces were also diced. Final step is to make the cells was to remove oxide off Si pieces, clean in SC-1 solution and bond using custom made anodic bonding jig.

Chambers are monitored by Quartz "tuning fork" resonators (Figure 1b). One chamber (Fork 1) contains a heat exchanger to cool the <sup>3</sup>He. Fork 2 monitors the "isolated" chamber (Figure 1c,d). Pulses applied to Fork 2 heat the isolated chamber and heat flows through the micromachined channel. The relaxation following a heat pulse is measured. We have identified a thermal transport mechanism (thermomechanical heat flow) that is present only in the superfluid and then only when the mean free path is much longer than the height constraint. [5]

Measurements on supercooling of the A phase are in progress. The two phases (A and B) that are stable in the bulk are separated by a first order phase transition line. When the A phase is cooled below its stable region, it persists in a metastable state because nucleation of the B phase requires a significant energy cost. We observe



Figure 2: A. Cartoon cross-section of a graphene device. Suspended graphene (green) is pulled toward the back-gate via an applied voltage. B. False-color SEM image, showing the suspended graphene (green), metallic Source, Drain, and Gate (yellow), and surrounding SiO<sub>2</sub> substrate (grey). C. Liquid N<sub>2</sub> flow cryostat with optical access. Sample is mounted on a 24-pin Dual In-line Package (purple). D. Heated test chamber with optical window [3]. See full color version on pages xxviii-xxix.

supercooling and measure the width (in temperature) of the distribution of A to B nucleation events. We intend to map the pressure dependence of the supercooling and determine if supercooling is affected by the thermal channel which favors the A phase.

We have also examined the resonant frequency and quality factor of single layer graphene resonators both above (300-500 K) and below (80-300 K) room temperature (Figure 2). The critical point dryer was an essential tool in this fabrication. We find that the resonant frequency and quality factor increase as the temperature deviates from room temperature. The results are interpreted as evidence of the persistence of traces of polymer coatings used as part of the transfer process to deposit the single layer material from growth chamber to device. [3]

Former Physics Ph.D. students Nikolay Zhelev (now Corning Research), Roberto DeAlba (now NIST), and post-doctoral scientist Abhilash Sebastian (now Aalto University) fabricated these structures before they left Cornell. Results from these structures are now emerging or have been recently published.

#### **References:**

- Fabrication of microfluidic cavities using Si-to-glass anodic bonding, N. Zhelev, T. S. Abhilash, R. G. Bennett, E. N. Smith, B. Ilic, L. V. Levitin, X. Rojas, A. Casey, J. Saunders, and J. M. Parpia arXiv:1805.00958.
- [2] The A-B transition in superfluid helium-3 under confinement in a thin slab geometry, N. Zhelev, T.S. Abhilash, E.N. Smith, R.G. Bennett, X. Rojas, L. Levitin, J. Saunders, and J.M. Parpia, Nature Comm. 15963 (2017).
- [3] Temperature-dependence of stress and elasticity in wettransferred graphene membranes, Roberto De Alba, T. S. Abhilash, Aaron Hui, Isaac R. Storch, Harold G. Craighead, and Jeevak M. Parpia, Journal of Applied Physics 123, 095109 (2018).
- [4] Phase Diagram of the Topological Superfluid <sup>3</sup>He Confined in a Nanoscale Slab Geometry, L. V. Levitin, R. G. Bennett, A. Casey, B. Cowan, J. Saunders, D. Drung, Th. Schurig and J. M. Parpia, Science, 340 841-844, (2013).
- [5] Thermal Conductivity of <sup>3</sup>He confined in a 1.1 μm sized channel, Jeevak Parpia, Anna Eyal, Nikolay Zhelev, Dmytro Lotnyk, Michael Terilli, Abhilash Sebastian, Eric Smith, http://meetings. aps.org/Meeting/MAR18/Session/A45.3.