FINAL REPORT

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Title: **Tunneling and Transport in Nanowires**

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ABSTRACT

The goal of this program was to study new physical phenomena that might be relevant to the performance of conductive devices and circuits of the smallest realizable feature sizes possible using physical rather than biological techniques. Although the initial scientific work supported involved the use of scanning tunneling microscopy and spectroscopy to ascertain the statistics of the energy level distribution of randomly sized and randomly shaped quantum dots, or nano-crystals, the main focus was on the investigation of selected properties, including superconductivity, of conducting and superconducting nanowires prepared using electron-beam-lithography. We discovered a magnetic-field-restoration of superconductivity in out-of-equilibrium nanowires driven resistive by current. This phenomenon was explained by the existence of a state in which dissipation coexisted with nonvanishing superconducting order. We also produced ultra-small superconducting loops to study a predicted anomalous fluxoid quantization, but instead, found a magnetic-field-dependent, high-resistance state, rather than superconductivity. Finally, we developed a simple and controllable nanowire in an induced charged layer near the surface of a masked single-crystal insulator, $SrTiO_3$. The layer was induced using an electric double layer transistor employing an ionic liquid (IL). The transport properties of the induced nanowire resembled those of collective electronic transport through an array of quantum dots.

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PROJECT DESCRIPTION
General Introduction and Summary

The goal of this program was to obtain a fundamental understanding of phenomena that might be relevant to the performance of conductive devices and circuits at the limit of the smallest realizable feature sizes where new physics in the form of quantum effects would be realized. The experimental approach was to study structures prepared using *physical* rather than *chemical* or *biological* techniques. Given the limits of lithography, the structures would all be in the mesoscopic regime.

The initial scientific work supported by the program involved the use of scanning tunneling microscopy and spectroscopy to ascertain the energy level distribution of randomly sized and randomly shaped quantum dots, or nano-crystals. This was followed by the major focus, which was on the investigation of selected properties, including superconductivity, of conducting and superconducting nanowires that were quasi-one-dimensional (quasi-1D) rather than one-dimensional (1D). The quasi-1D regime is one in which the transverse dimensions of a wire are smaller than the characteristic length associated with a phenomenon, such as the superconducting coherence length in the case of superconductivity. The true electronic 1D regime of a nanowire would require transverse dimensions less than the Thomas-Fermi screening length. To achieve this regime in *metals* would require wires with transverse dimensions of the order of 1 nm.

We originally intended to form such wires on vicinal cut $SrTiO_3$ surfaces, taking advantage of the periodic array of steps that results from the faceting of such surfaces. The growth of a deposited material on such a faceted surface could yield 1D wires, provided it occurred in the grooves of the substrate. At a fraction of a monolayer of coverage, and after annealing, highly ordered atomic chains can form. Unfortunately we found that we could only grow nano-crystalline chains, rather than continuous wires on the faceted $SrTiO_3$ available to us. As a consequence, we restricted our considerations to the quasi-1D regime and using electron beam lithography to fabricate our samples. We refined this process, making wires with widths and thicknesses in the 10 to 100 nm range, well into the quasi-1D regime for phenomena such as superconductivity.

An important direction was the effort to verify the existence of the reported superconducting "anti-proximity" effect (Tian *et al.*, 2005 and 2006), preparing quasi-1D wires in a manner different from that employed in the original work. This work led to the discovery of a magnetic-field-restoration of superconductivity in out-of-equilibrium nanowires driven resistive by current (Chen, Snyder and Goldman, 2009, and Chen, Lin, Snyder and Goldman, 2011). Until recently there was only a qualitative theoretical explanation for these observations, involving dissipation (Vodolazov and Peeters, 2012), but no quantitative theory. We produced data together with a theoretical explanation developed in collaboration with our colleague Professor Kamenev, which appears to lead to a resolution of these puzzling findings (Chen *et al.* 2014). The explanation involves the existence of a state in which dissipation coexists with a nonvanishing superconducting order parameter.

Using the same fabrication technology employed to produce nanowires, we have produced ultra-small superconducting loops, which were used to investigate the prediction that for sufficiently small loops, *i.e.*, for loop dimensions less than the coherence length, the flux quantum is h/e rather than the usual $h/2e$ (Wei and Goldbart, 2008 and references cited therein). The result of these investigations was the observation of an unexpected, anomalous high-resistance state, rather than superconductivity. This precluded further study of h/e oscillations.

Finally, we developed a simple and controllable system, the induced charged layer near the surface of a single-crystal insulator such as $SrTiO_3$ that is produced in an electric double layer transistor employing an ionic liquid (IL) as the dielectric. We have been able to pattern conducting nanowires in these electric double layer configurations (Bretz-Sullivan and Goldman, 2015).

The following sections contain brief discussions of our findings in each of the areas mentioned above.

Nanoparticles

The first project undertaken was an investigation of the energy levels of metallic clusters. In the many years that have passed since the first observation of discrete energy levels in metallic clusters there still remained the question of how the levels are statistically distributed in these systems. It was suggested that random matrix theory (RMT) is applicable to the statistical properties of the spectra of metallic clusters in much the same way that it is applicable to the slow neutron resonant spectra observed in the 1950s and 1960s (For a review see: Y. Alhassid, 2000). However experimental verification of the applicability of RMT in these systems is challenging because of the difficulty in gathering a sufficient number of levels to analyze their statistical distribution.

Ralph, Black and Tinkham (1995) made the first observations of discrete energy levels or “particle in a box energy levels” in metallic clusters. The energy levels were observed as irregular steps contained within the Coulomb staircase in the current-voltage characteristics of clusters that were fabricated using a fixed tunneling geometry with metallic electrodes. These uneven steps in the current-voltage measurements might be a consequence of RMT. This suggestion arises from earlier predictions that address these systems from various theoretical standpoints, including Efetov's supersymmetry derivation (Efetov, 1983). While these expectations are theoretically well established they are experimentally difficult to realize because of non-equilibrium effects and capacitive charging energy terms that have a tendency to mask the energy levels in mesoscopic systems. Subsequent tunneling experiments have been performed on metallic clusters and semiconducting dots which have yielded results similar to those of Ralph, Black and Tinkham, although the nature of the level statistics still remained elusive.

In contrast to metallic clusters, experimental work has exhaustively addressed the issue of distributions of level spacings and eigenfunctions of quantum dots fabricated from two dimensional electron gas systems of various pre-defined shapes. In these systems, electron-

electron interactions dominate transport and the level spacing distributions appear to be Gaussian, while the distributions of the amplitudes of the eigenfunctions follow a Porter-Thomas distribution, which is a signature of RMT.

Random matrix theory and quantum chaos were merged in the conjecture put forward by Bohigas, Giannoni and Schmit (1984). This conjecture states that the nearest neighbor energy level spacings of classically chaotic systems should be distributed according the Gaussian Orthogonal Ensemble (GOE), or Wigner-Dyson distribution (Wigner, 1957) and this conjecture is strongly supported by aggregated numerical studies.

The Wigner-Dyson distribution, which describes the statistical distribution of nearest neighbor energy level spacings normalized to the mean energy level spacing, has several important features: first, the probability of having nearest neighbors with zero spacing disappears. Second, the probability of a level spacing is linear in energy before approaching a maximum, with the maximum occurring close to the mean energy and the tail of the distribution being fairly small. This is in contrast with completely random levels (a classically non-chaotic system) where the distribution is Poissonian. For the latter distribution the probability is largest at zero level spacing. The absence of small spacings in the Wigner-Dyson distribution is known as the: "repulsion of energy levels". This is the key ingredient of a Wigner-Dyson distribution and distinguishes classically chaotic from classically non-chaotic systems.

It is also possible to study the statistics of the amplitudes of the eigenfunctions within the context of RMT. The corresponding distribution is called the Porter-Thomas distribution. The Porter-Thomas distribution is simply a statement that the amplitude $|\psi|$ of a wavefunction at any given point is a random variable. The distribution of the square of a random variable, which is Gaussian distributed, is the Porter-Thomas distribution.

Comprehensive accounts of our experimental work, which has been published in Physical Review Letters (Adams, Lang and Goldman, 2005) and Physical Review B (Adams, Lang, and Goldman, 2007) indicate that statistical distributions of the energy levels of highly irregularly shaped Pb clusters follow RMT. The unique device geometry is discussed in detail. The connection of the measured energy spectra to the geometry and the observed features of the spectra are described completely. The energy levels are described in terms of statistical distributions of both the eigenvalues and the amplitude of the moduli of the eigenfunctions. We also demonstrate the use of scanning tunneling spectroscopy to obtain a quantity that is proportional to the square of the amplitude of the eigenfunctions in these systems.

Nanowire Fabrication

We developed a simple approach to the formation of unbroken metal nanowires with widths as narrow as 5 nm and lengths some tens of thousands of nanometers long. A signature of weak anti-localization was found in the temperature dependence of the resistance of Au nanowires fabricated using this approach. Measurements of the resistance vs. temperature in high and zero applied magnetic field were used to extract the temperature dependence of the phase coherence time, which was found to be in fair agreement with theory, especially for multiple-wire samples. This technique of wire formation may be generalized to permit the growth of even narrower metal wires.

Our approach to the fabrication of ultra-narrow metal wires uses conventional electron beam lithography (EBL) patterning followed by an angled film deposition at reduced temperature. This permits control of the positioning and the design of the nanostructures. Besides the flexibility provided by EBL patterning, additional advantages include the absence of special requirements relating to substrates and deposition materials and the possibility of producing, using the same deposition angle, wires with different widths simply by changing the initial widths of the resist channels. This will permit the formation of different patterns on the same wafer.

The first step of process involves writing channel patterns of 50nm in width, using the photoresist C2 polymethylmethacrylate (PMMA). The writing parameters are 20kV for the acceleration voltage and 20 mm for the aperture. The second step involves an angled thermal deposition of metal at a reduced temperature. The substrate is mounted on a stage attached to a liquid Nitrogen reservoir. The sample holder enables us to adjust the angle between the sample channels and a metal vapor beam. As shown in the cartoon in Fig. 1, the channel patterns can act as a shadow masks narrowing the wire width when the vapor deposition is at an angle.

Then, the width of the wire can in principle be expected to be d when the angle is set to $\theta = \tan^{-1}((l-d)/h)$, where l is the 50nm width of lithographic channels and h is the 120 nm height of the PMMA photoresist channel wall.

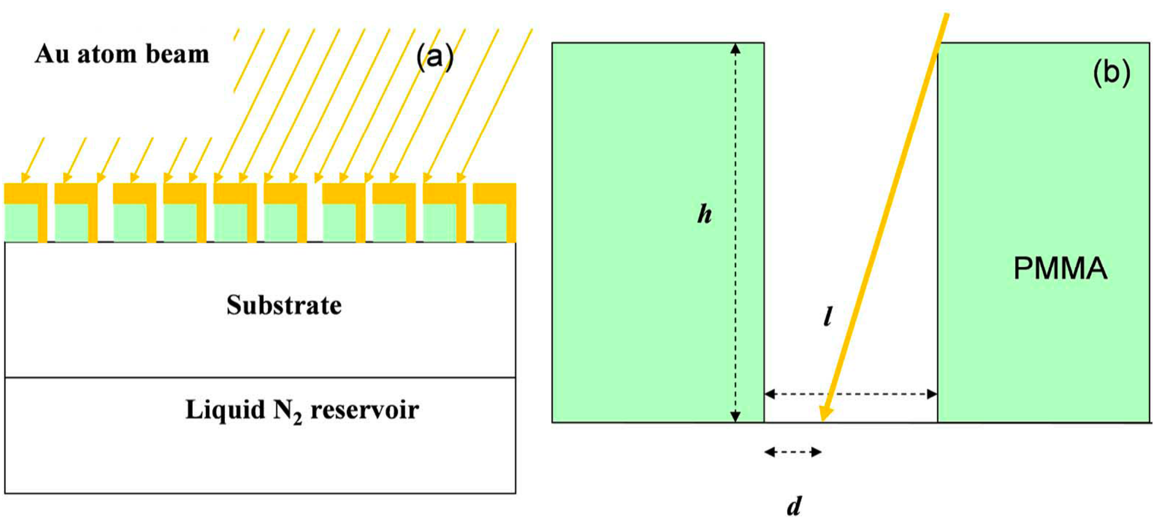

Fig. 1. a) Schematic showing the angled deposition onto a substrate with channels pre-patterned using e-beam lithography. b) The width of the wires can in principle be expected to be d when the deposition angle set to $\theta = \tan^{-1}((l-d)/h)$.

Also worthy of mention is the importance of liquid nitrogen cooling in the formation of narrow wires. For the granular wires that we have fabricated, the grain size will eventually set the limit to the wire width. Holding the substrate at a reduced temperature suppresses the growth of the grains and thus lowers this limit to 5nm in our case. In addition, at reduced temperatures the lateral surface diffusion of atoms is also reduced, which will further narrows the wires. Cooling with liquid helium can in principle improve this method and enable even smaller wire widths to be produced. Figure 2 shows SEM pictures of some wires.

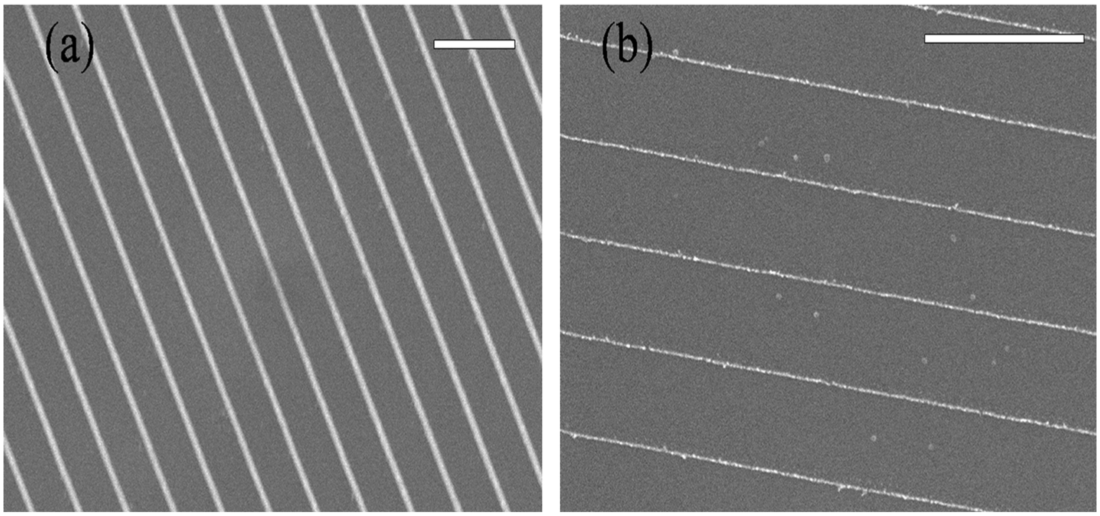

Fig. 2. Scanning Electron Microscope (SEM) images of a) 50nm-wide wires deposited in the usual way θ=0 (the white scale bar is 1μm) b) 15nm-widewires deposited in the usual using an angled deposition with θ =28 (the white scale bar is 1μm)

Electrical transport measurements have been carried out on wires made using these techniques. A direct signature of weak anti-localization was observed in the temperature dependence of the resistance. This enabled us to utilize R(T) measurements, in high and zero applied magnetic field, to extract τ_{φ}(T). The comparison with theory showed this method worked well, especially for multiple-wire samples. Even though this approach, when applied to single wires, did not yield parameters in good agreement with theory.

As the conventional approach involving magnetoresistance measurements, it is valuable given its simplicity. The generalization that may result in even smaller wires would be film growth with the substrates held at liquid helium temperatures. The results of this experiment were published in the Journal of Applied Physics (Chen and Goldman, 2008).

Nanoparticle Chains

Nanoparticles (NP) or quantum dots are of interest to both for scientists and engineers because of their unique electronic, photonic, and magnetic properties. A key to future electronics involving nanoparticles is the controllable and precise assembly, and their linkage to devices and structures of macroscopic size. Compared with two- and three-dimensional and assemblies, the construction of one-dimensional chains of nanoparticles is more challenging. However it may be of great use in fundamental phenomena. A good example is the work of Kouwenhoven and collaborators (1990), who studied the evolution of energy levels as function of inter-particle coupling and thus the formation of energy bands. Despite the numerous potential applications, research on 1D chains of NPs is still on its infancy. The bottlenecks involve difficulties in fabrication such as the precise positioning of chains, and the control of inter-particle distances.

As a byproduct of our work on nanowires, we have developed a new method for fabricating one-dimensional chains of nanoparticles (Chen and Goldman, 2007). It is a physical process that is simple to carry out. We have worked at controlling the size of the dots and the inter-dot gaps by adjusting the conditions of the post-annealing process. Taking the advantage of the precise positioning of E-beam lithographic patterning, this method provides opportunities to fabricate devices with specific requirements of 1D NPs chains.

Our approach involves two stages: 1) the fabrication of nanowires using the method mentioned in the previous section, with the positioning of the wires defining the position of the dot chains. Given the flexibility of E-beam lithography, one can have a lot of control of the design of the chains. They can be linear, curved or even circular. The need for different nanoparticles can be realized by depositing different materials onto the substrates. 2) the post-annealing on the wires. Upon being heated up, atoms start moving around to find positions that minimize the formation energy. Figure 3 shows an image of Au nanowires and chains of nanoparticles after post-annealing at 250^0C for three hours in air. We can control dot in inter-dot gap size by changing the annealing temperature, initial wire width and the substrate.

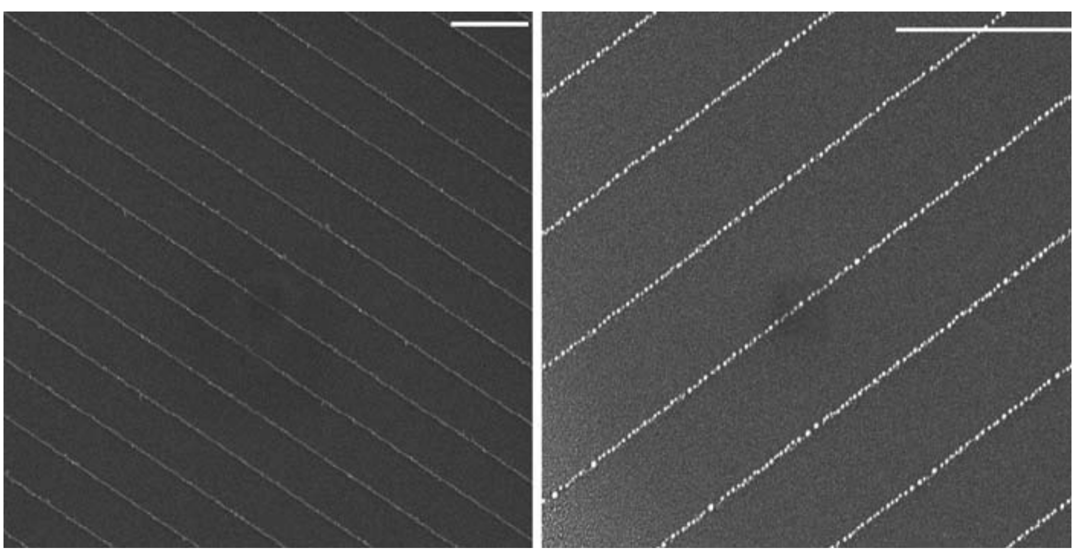

Fig. 3. SEM images, Left: Au nanowires with widths of around 20 nm and Right: linear chains of nanoparticles after post deposition annealing at 250 °C for 3 hours. The white bars in both figures are 1 mm.

In order to study how various factors affect the final configurations of the dot chain, wires with different widths and formed on different substrates were post-annealed at various temperatures and for various periods of time. Afterwards, statistical studies were performed to determine the dot and inter-dot gap size distributions by randomly sampling 100 dots and 100 gaps using SEM imaging.

SUPERCONDUCTING NANOWIRES

This work was originally motivated by two developments in the physics of quasi-one-dimensional superconducting systems. The first was the report by Tian *et al*. (2005 and 2006) of what they termed an "antiproximity" effect in a system consisting of Zn nanowires, 40 nm in diameter, sandwiched between either bulk Sn and In electrodes. When the electrodes were superconducting, the superconductivity of the Zn nanowires was suppressed. When the electrodes were driven normal by a magnetic field, the superconductivity of the nanowires recovered. The second development concerned evolving studies of the quantum suppression of superconductivity in nanowires (Bezryadin, 2008 and Bollinger *et al*., 2008).

Magnetic field induced restoration of superconductivity in out-of-equilibrium Zn nanowires

The wires used in the work of Tian *et al*. (2005 and 2006) were produced by a template-assembly electro-deposition technique in which the diameter and length of the nanowires were controlled by the pore diameter and thickness of porous polycarbonate and aluminum oxide membranes. The wires were mechanically squeezed between bulk electrodes. The measurements were two-terminal and the measured resistance included contributions from the bulk superconducting electrodes, the nanowires themselves, and the two interfaces between the bulk superconductors and the nanowires.

Our goal was to determine whether the "antiproximity effect" would manifest itself in wire configurations prepared using lithographic rather than electrochemical techniques and whether the effect could be observed in a four-terminal planar configuration. To this end a four-terminal configuration was fabricated initially entirely of Zn, the metal yielding the largest effect in the antiproximity experiments of the Penn State group. The idea was to study this all-Zn configuration and then progress to overlaying the contact pads with Sn or Pb, to achieve the configuration discussed by the Penn State group. The overlay step did not work, as the nanowire structures always disintegrated. However in characterizing the all-Zn configuration, we found a different, but possibly related phenomenon, superconductivity turning on, or being restored upon the application of small magnetic fields to wires driven out of equilibrium and into a resistive state by an externally supplied current.

Standard four-terminal configurations of a single Zn wire connected with 1μm wide Zn electrodes of various lengths were studied. As shown in Fig. 4(a), the higher resistance part of the transition moved to lower temperatures with increasing magnetic field. The lower resistance part exhibited a different behavior, moving to higher temperatures with increasing field. Also the temperature at which the wire resistance vanished increased. As a result, the transition sharpened with increasing field. However this eventually stopped and the onset temperature as well as the temperature

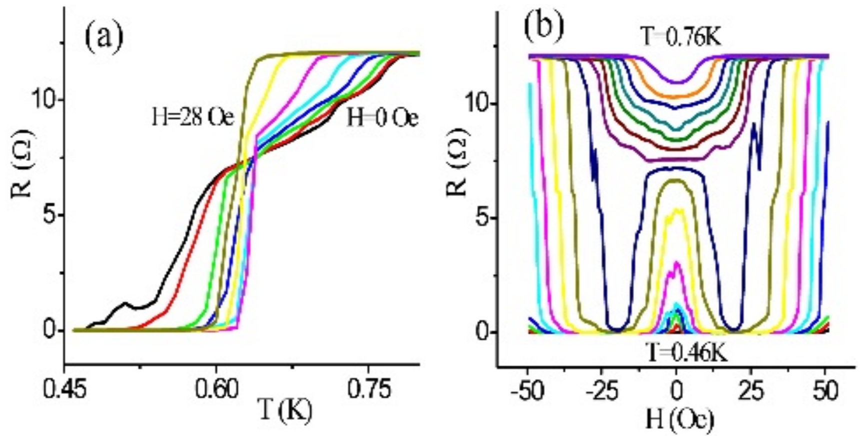

FIG. 4 (a) Temperature dependence of the wire resistance, at I = 4.4 μA, with varying applied magnetic fields from 0 Oe to 28 Oe, every 4 Oe. **(b)** Magnetic field dependence of the wire resistance, at I = 4.4μA, with temperatures ranging from 0.46K to 0.76K, every 0.02K.

at which the resistance vanished, both moved together towards lower values upon increasing the field. In effect, there is a magnetic field induced restoration of the superconducting state over the range of temperatures corresponding to the lower part of the zero field transition as illustrated in Fig. 4(b).

The initial results of this project were published in Physical Review Letters (Chen, Snyder and Goldman, 2009), and a long paper detailing the extensive results of changing the magnetic field orientation as well as the length of the nanowires has been published in Physical Review B (Chen, Lin, Snyder and Goldman, 2011).

The most recent results, involve the extension of the measurements to temperatures as low as 50 mK, and studies of the I-V characteristics with short time constants. There were two features that emerged from these low temperature studies. Over a range of currents, flanked by the bottom and top threshold values $I_b < I < I_t$, the voltage across the nanowire exhibits a nearly flat plateau at $V_0 = 52.5\pm1.2$ mV (for T < 450 mK). It indicates a peculiar dissipative state, which is distinctly different from the normal state. It is important to note that both I_t and I_b are factors of 30 to 50 times smaller than the estimated depairing critical current of the wire. Performing measurements on different devices, we found a remarkable universality associated with the voltage plateau.

Another remarkable feature of the voltage plateau state is its onset through a region of the stochastic bistability. It is revealed by time-domain measurements, with the voltage measured with a repetition rate of 3 Hz under a sustained constant current. The system exhibits random switching between the superconducting and voltage plateau states with a characteristic time scale of a few seconds, indicating an intrinsic bi-stability. It is the dependencies on temperature and magnetic field, of the switching probabilities from the superconducting state to the voltage step state and subsequently to the normal state that hold the key to the magnetic field enhanced superconductivity of nanowires. This work was published in 2014 in Nature Physics (Chen *et al*., 2014).

The most recent work has involved the application of transverse magnetic fields to 100nm wide Al nanowires, 1 μm in length. The response to in-plane transverse magnetic fields in this geometry is qualitatively different from that previously reported for perpendicular-to-the-plane field experiments and for in-plane longitudinal field studies. The different feature in the data is an abrupt return to the superconducting state with increasing field at values of field corresponding to a single flux quantum for a short wire and a fractional flux quantum for a long wire. Since these findings are dramatically different from those involving perpendicular-to-the-plane magnetic fields, a different mechanism, as yet unidentified, may be at work. This work has been published in Physical Review B (Bretz-Sullivan and Goldman, 2016).

The search for *hc/e* period oscillations in the critical temperature of small superconducting rings threaded by magnetic flux

We carried out experiments on nanowire rings to search for oscillations in the critical temperature of small superconducting rings threaded by magnetic flux that were periodic with a period *h/e* (Wei and Goldbart, 2008 and Loder *et al.*, 2009 and references cited therein). The usual Little-Parks oscillations display a period of *h/2e*, which is a reflection of the binding of electrons into Cooper pairs. (Little and Parks, 1962). Single-electron Aharonov-Bohm oscillations in resistance or persistent current in a clean metallic ring also have a period *hc/e* (Webb *et al.*, 1985). The question of *h/e* periodicity arises in superconductors when the radius of a ring becomes comparable to or smaller than the coherence length. Numerous calculations using various techniques have predicted *h/e* period oscillations in the supercurrent.

The attempts at this experiment have involved rings with very small feature sizes that were prepared using the Vistec electron beam writer. The results with the smallest rings have been of an unexpected nature, which has been the subject of a paper published in Physical Review B (Snyder *et al.*, 2013). The main feature of the data is the observation of an apparently phase coherent high resistance state (HRS) found in the presence of a magnetic field with the resistance exceeding fifteen times the normal state value. This is the first measurement of such a HRS in nanorings. However, through the experiments and simulations we have reported, this phenomenon can be understood as a manifestation of tilted normal metal-superconducting (NS) boundaries, which arise naturally in these nanostructures (Arutyunov et al., 2008).

Details of the dependence of this effect on the measurement geometry and the results of a numerical simulation are reported in the paper (Snyder *et al.*, 2013), which summarizes the doctoral dissertation of Stephen Snyder, who is now a staff member at Intel. Because the high resistance state is almost certainly unavoidable in the small structures that would be needed to search for h/e oscillations, we decided to call off the search for them.

ELECTROSTATIC TUNING OF THE PROPERTIES OF NANOWIRES USING AN IONIC LIQUID

We established that we are able to use electric double layer transistor (EDLT) configurations to modify the properties of insulating $SrTiO_3$ crystals. The phase diagram obtained was discussed in Lee *et al*. (2011). The ionic liquid (IL) used in this work was N, N-diethyl-N-(2-methoxyethyl)-N-methylammonium bis (trifluoromethyl sulphonyl)-imide (DEME-TFSI). This particular IL has produced very high charge accumulations on the surface of semiconductors such as ZnO with induced sheet carrier densities of up to 10^{15} cm^{-2} being reported (Yuan *et al.,* 2007).

We have succeeded in producing TiO_2 terminated $SrTiO_3$ surfaces exhibiting a terrace structure as a result of the vicinal miscut, and patterning source and drain electrodes such that the nanowires when patterned do not cross a terrace boundary. The areas under the Ti/Au contacts were ion milled with Ar^+ ions before depositing the contacts. A side gate configuration was established. This was accomplished using electron beam lithography and lift-off.

Recently, measurements of the current-voltage (*I–V*) characteristics of ionic liquid gated nanometer scale channels of strontium titanate were completed (Bretz-Sullivan and Goldman, 2015). At low gate voltages, the *I–V* characteristics exhibit a large voltage threshold for conduction and a nonlinear power law behavior at all temperatures measured. The source-drain current of these nanowires scaled as a power law of the difference between the source-drain voltage and the threshold voltage. The scaling behavior of the *I–V* characteristic was similar to that of collective electronic transport through an array of quantum dots. At large gate voltages, the narrow channel acts as a quasi-1D wire whose conductance follows Landauer's formula for multichannel transport.

PUBLICATIONS SUPPORTED BY THE GRANT

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