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14. ABSTRACT
This report documents the design and construction of a low temperature scanning tunneling microscope optimized for the study of superconductors. The microscope was first used to study the vortex liquid state in the cuprate high-Tc superconductor Bi2Sr2CuO6+x. This work distinguished between a homogenous superconducting gap and an additional inhomogenous 'pseudogap' which may be linked to an alternative electronic ordered state. The microscope then performed the first atomic resolution, high-field investigation of the new iron arsenic high-Tc superconductors. In particular, the vortex state was mapped in Ba(CoxFe1-x)2As2. Vortices formed a disordered lattice, uncorrelated with surface impurities, which demonstrated strong bulk pinning. Vortex core states were used to measure an electronic correlation length of 2.8 nanometers, from which an upper critical field of 43 Tesla was calculated.

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Vortex Pinning in the High-T_c Superconductor $YBa_2Cu_3O_{6+x}$

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Abstract:

This report documents the design and construction of a low temperature scanning tunneling microscope optimized for the study of superconductors. The microscope was first used to study the vortex liquid state in the cuprate high-T_c superconductor $Bi_2Sr_2CuO_{6+x}$. This work distinguished between a homogenous superconducting gap and an additional inhomogenous 'pseudogap' which may be linked to an alternative electronic ordered state. The microscope then performed the first atomic resolution, high-field investigation of the new iron arsenic high-T_c superconductors. In particular, the vortex state was mapped in $Ba(Co_xFe_{1-x})_2As_2$. Vortices formed a disordered lattice, uncorrelated with surface impurities, which demonstrated strong bulk pinning. Vortex core states were used to measure an electronic correlation length of $\xi = 2.8$ nm, from which an upper critical field of $B = 43$ T was calculated.

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Summary:

Thc stated objeetive of this projcet was to eonstruet a high resolution, low temperature seanning tunneling mieroseope to determine the roles of different crystal defcets in pinning vortiees in supereondueting $YBa_2Cu_3O_{6+x}$. The proposed STM would operate down to sub-Kclvin temperatures on a ^{3}He fridge within a dual-axis supereonducting magnet. The systcm would include in-situ sample cleaving for single erystal studies, and in-situ sample exehange for quiek studies of multiple samples. These eapabilities would enable the study of vortex pinning and dissipation in high-T_c superconductors with potential for DOD applications (e.g. for use in efficient filters for high-frequency communication, or in motors or generators where minimizing weight is an important consideration) [Hassenzahl: 2004].

This report documents the sueeessful design and eonstruetion of thc microscope head, eryostat, vibration isolation, and clectronics for the STM system. Although the original goal was vortex imaging in $YBa_2Cu_3O_{6+x}$, initial tests of the new system wcrc performed on the better-eharactcrizcd cuprate high-T_c superconduetor $Ba_2Sr_2CuO_{6+x}$. The initial test results were intercsting in their own right: a vortex liquid was imaged up to 9T, and was used to distinguish bctwecn a homogenous superconducting gap and an inhomogeneous pseudogap baekground.

Just as the new STM system was fully eharaeterized and rcady to study $YBa_2Cu_3O_{6+x}$, thcre was an exeiting new development in Japan: the diseovery of a seeond family of high-T_c superconductors, the iron pnictides. There was tremendous worldwidc optimism that these iron pnictidcs would bc more suitable for applications than the cuprates had been, due to their metallic ground state and significantly better isotropy. Upon eonsultation with the AFOSR program manager, the new STM system was thercfore uscd to characterize vortcx pinning in an iron pnictide superconductor, $Ba(Co_xFe_{1-x})_2As_2$, rathcr than thc initially planncd $YBa_2Cu_3O_{6+x}$. By comparing the locations of vortiees and surfacc pinning sites in $Ba(Co_xFe_{1-x})_2As_2$, it was found that vortiees are strongly pinned in the bulk of this matcrial. Vortex eore states wcrc uscd to mcasurc an electronic correlation length of $\xi = 2.8$ nm, from whieh an upper eritieal field of B = 43 T was calculated.

Introduction:

Twenty-three years have elapsed since the discovery of high temperature superconductors, but these materials have yet to find their way into mainstream technological applications. A significant problem is the motion of magnetic vortices: their motion leads to undesired dissipation and places severe limits on the critical current J_c in superconducting devices. This problem has motivated research into the nature of vortex pinning in superconducting materials. Effort focused initially on BSCCO/Ag cables, but in the last decade more effort has been focused on vortex pinning in YBCO coated conductors, which offer potentially significant cost and engineering benefits. However, the problem of vortex pinning in YBCO coated conductors is far from solved.

Most recently, in 2008, a second family of HTSCs was discovered: the iron-pnictides. A flood of preliminary research indicated that these new materials may not suffer from the same technical challenges as cuprates: they have a metallic ground state, better electronic isotropy, and preliminary evidence for a symmetric order parameter and strong native vortex pinning [Grant: 2008]. Therefore, more detailed vortex pinning studies in these new iron pnictide materials are urgently needed.

Vortex pinning work so far in all materials has primarily focused on bulk transport measurements and magnetic imaging techniques. Transport measures J_c but reveals very little about the specific vortex pinning sites. Magnetic imaging has progressed from magneto-optical imaging, which images flux penetration with a resolution of ~10 μm, to scanning Hall probe microscopy and magnetic force microscopy, which image single vortices with resolution as high as 20 nm. However, these techniques image vortices via their magnetic field profile, which can never be made smaller than the superconductor penetration depth λ. In cuprate and in iron pnictide high-T_c superconductors, this sets an absolute lower limit of ~100 nm resolution for direct magnetic imaging techniques. Vortices at fields higher than ~500 Gauss will never be resolved.

Scanning tunneling microscopy, on the other hand, can theoretically image vortices up to the upper critical field, H_{c2}, typically tens of Tesla in high-T_c materials. A vortex core contains a patch of non-superconducting electrons whose density of states signature can be imaged by STM. The core size is determined by the superconducting coherence length, which is typically ~2 nm in high-T_c materials, almost two orders of magnitude smaller than the penetration depth λ. Furthermore, STM can directly image many point or line defects near the surface of a material, such as twin boundaries, grain boundaries, edge dislocations, screw dislocations, surface depressions, oxygen inhomogeneities, or chemical inclusions. The combined knowledge about structural defects and actual vortex locations can yield much more detailed insight into the nature of vortex pinning.

This report will focus on the construction of a new STM system, optimized for study of superconductors, on test images of a vortex liquid in $Ba_2Sr_2CuO_{6+x}$, and on the first high-field imaging of vortices in an iron pnictide superconductor $Ba(Co_xFe_{1-x})_2As_2$.

Methods, Assumptions, and Procedures:

Most of the work reported here was carried out by two graduate students: Yi Yin and Tess Williams, and by 2 postdocs Dr. Heonick Ha and, more significantly, Dr. Martin Zech. Some additional work was carried out by undergraduates Hasan Korre, Stefan Wernli, and Sam Cross.

Scanning tunneling microscope: The scanning tunneling microscope was designed in Solidworks by Dr. Heonick Ha, who modeled it closely after an STM in the J.C. Davis lab at Cornell, originally built by Shuheng Pan and Eric Hudson. Parts were machined from macor, copper, and beryllium copper in Harvard's School of Engineering and Applied Sciences professional machine shop.

Cryostat: The flow cryostat was designed by graduate student Yi Yin. Although it was initially designed to operate with ^{3}He pumped by single-shot charcoal down to 250 mK, such low temperatures were not required to study vortex pinning in the iron pnictide superconductors. The cryostat was therefore operated with only ^{4}He, bringing the STM base temperature down to ~5K, during the period of this grant.

Magnet: The 2-axis superconducting magnet from AMI was installed inside a dewar from Kadel. The magnet consisted of a 9T solenoid and a 3T split coil. The entire dewar was mounted on a goniometer, allowing the split coil to rotate with respect to the microscope: this resulted in an effective 3-dimensional vector field of up to 3T.

Scanning control electronics: A thorough survey of existing vendors of scanning probe microscopy control hardware & software was carried out. An initial web search turned up approximately 60 vendors. Phone calls and emails to vendors and current users of their equipment narrowed the list to four systems compatible with the needs of this project. Four controllers were borrowed for on-site testing with our STM. Tests included the following: feedback loop bandwidth, noise on the bias output and high voltage scanner outputs, input noise, and robustness and user-friendliness of software.

Summary of custom electronics designed and constructed:

- high voltage tip withdrawal safety circuit (analog circuit protects against tip crashes in case of a software crash of the data acquisition computer)
- field emission power supply
- attoFarad capacitance detection circuit
- controller for coarse motion stepper motors
- various filters and breakout boxes

Results and Discussion:

Because part of the goal of the project was to construct a new instrument, this section of the report provides photographs of assembled components. The two major scientific results discussed here are: imaging of a vortex liquid in $Bi_2Sr_2CuO_{6+x}$ to distinguish between the superconducting gap and pseudogap, and imaging of a disordered vortex lattice in a new iron pnictide superconductor $Ba(Co_xFe_{1-x})_2As_2$.

Scanning tunneling microscope system construction

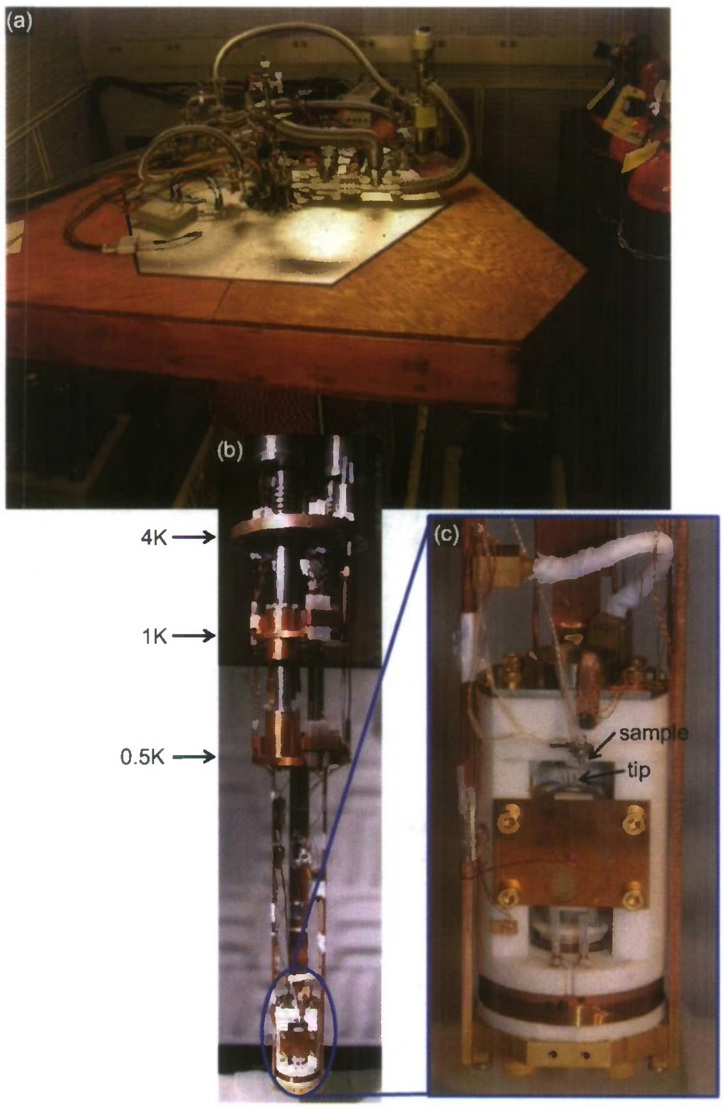

Figure 1: Photograph of the STM system. **(a)** Lead-filled wood table floats on 3 air springs inside a soundproof 20-ton room, which floats on 4 additional air springs. Dewar hanging from table is wrapped in bungees for additional damping. **(b)** Cryostat, designed for ^{3}He operation down to < 500 mK. **(c)** Scanning tunneling microscope head. System construction is more thoroughly documented in the PhD thesis of Yi Yin [Yin: 2009c].

$Ba_2Sr_2CuO_{6+x}$

Although the goal of the grant was to apply the STM to vortex pinning studies in superconductors with promise for technological applications, we felt it important to test our new instrument first on samples whose cleaving properties had been well established by previous STM experiments. Our first experiment was therefore to look for vortices in $Bi_2Sr_2CuO_{6+x}$. One of the most important questions in the cuprates is whether the pseudogap is a competing or unrelated phase, or whether it signifies the higher temperature formation of pairs which lack phase coherence until the sample is cooled through the global superconducting T_c. Recent STM work normalized each superconducting density of states spectrum by dividing out the corresponding spectrum from the same atomic location, just above T_c [Boyer: 2007]. Their work discovered a previously unnoticed homogenous low-energy gap, presumed to be the superconducting gap, amongst the inhomogeneous larger energy gaps, which now seem to be pseudogaps without relation to superconductivity. One weakness of this study is the effect of thermal broadening on the high temperature spectra. We have confronted this problem at a single, low temperature, destroying the superconducting state instead by application of a magnetic field. When we normalize each superconducting (zero field) spectrum by the corresponding high field spectrum in the vortex liquid state at the same atomic location, we verify the two-gap picture. We find a homogenous low energy gap in the superconducting state, which is not present above H_{c2}. But we find the inhomogeneous, higher energy pseudogap remains unchanged through the superconducting transition in fields up to 9T. These results are more thoroughly documented in the PhD thesis of Yi Yin [Yin: 2009c].

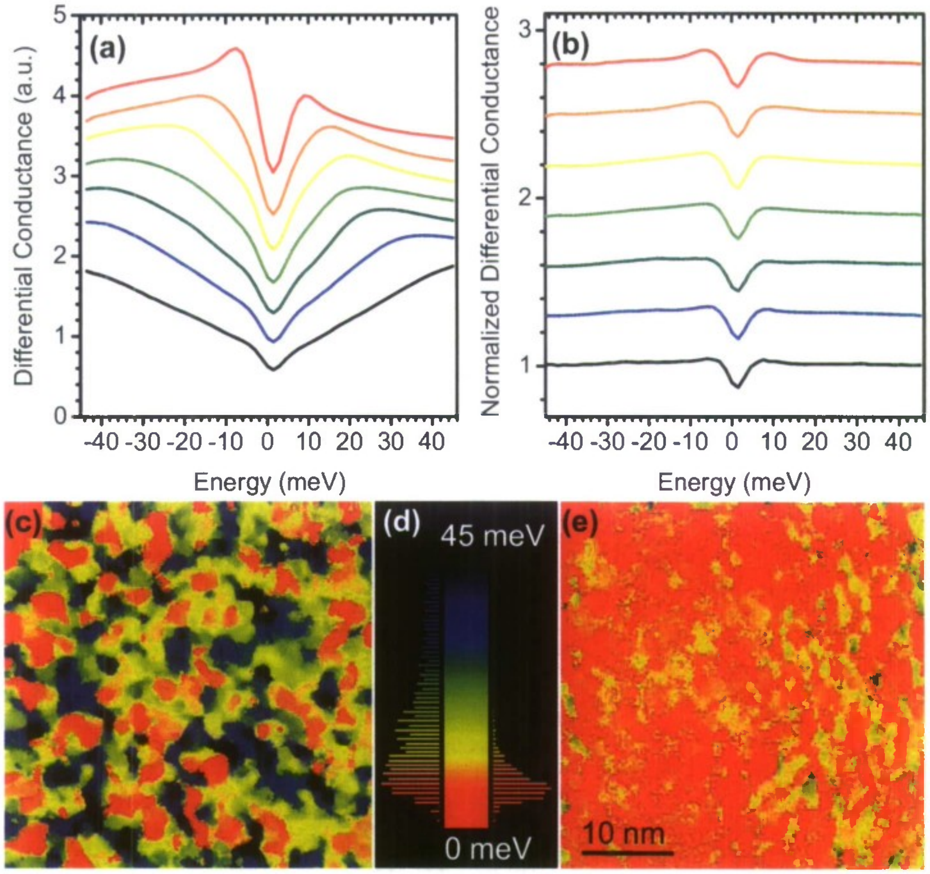

Figure 2: (a) Spectra from T_c=16K overdoped $Ba_2Sr_2CuO_{6+x}$ show an inhomogeneous large energy gap. **(b)** Normalization by corresponding spectra at the same atomic locations in applied 9T field show a homogenous low-energy gap with Δ=7meV. **(c)** Map of the large energy inhomogeneous Δ in a 40nm area in 0T field. **(d)** Color scale and histograms for Δ-maps shown in (c) and (e). **(e)** Map of the Δ that emerges on point-by-point normalization of 0T spectra by dividing out the corresponding 9T spectra.

$Ba(Co_xFe_{1-x})_2As_2$

Following the worldwide excitement upon discovery of a second family of high-Tc superconductors, the iron pnictides, in early 2008, wc choose to study optimally doped single crystal $Ba(Co_xFe_{1-x})_2As_2$. Since the Co dopants are incorporated into the strongly bound FeAs layer, thc topmost layer is presumed to be more like the bulk and more stable while tunneling. Our samplcs, grown with FeAs flux to avoid contamination by other elements [Wang: 2009], show a sharp resistive transition at $T_c = 25.3$ K with width $\Delta T = 0.5$ K. Our results are fully documented in two published papers [Yin: 2009a, 2009b] and a PhD thesis [Yin: 2009c].

In summary, the main results are:

(1) At zero field, a single gap with coherence peaks at $\overline{\Delta} = 6.25$meV is observed in the density of states. The gap Δ shows nanoscale inhomogeneity with fractional variation $\sigma/\overline{\Delta} \sim 12\%$.

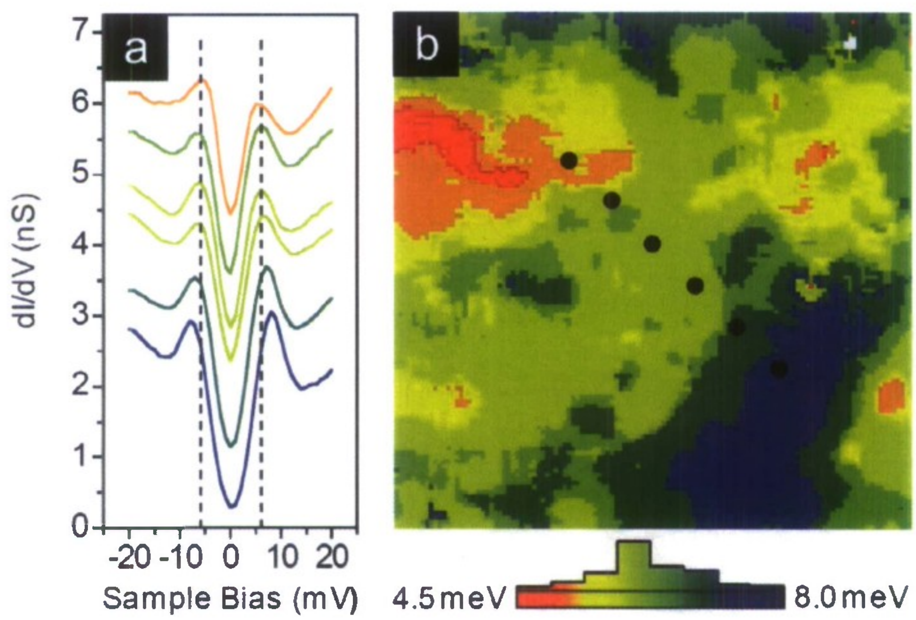

Figure 3: (a) A series of dI/dV spectra taken along an 11 nm line, illustrating the shape of the superconducting gap and the low differential conductance at the Fermi encrgy. **(b)** A 20 × 20 nm^2 gap map, revealing the spatial variation of the gap magnitude Δ. The gap has $\overline{\Delta} = 6.25$ meV and fraetional variation only 12%. A color-eoded histogram of Δ is shown below the gap map. The six spectra in (a) (from top to bottom) are taken at the loeations of the blaek points indieated in (b) (from upper left to lower right). Data were aequired at T = 6.25K in zero magnetie field.

(2) At 9T and 6 T, we image a disordered vortex lattice, consistent with isotropic, single flux quantum vortices. Vortex locations are uncorrelated with strong scattering surface impurities, demonstrating bulk pinning.

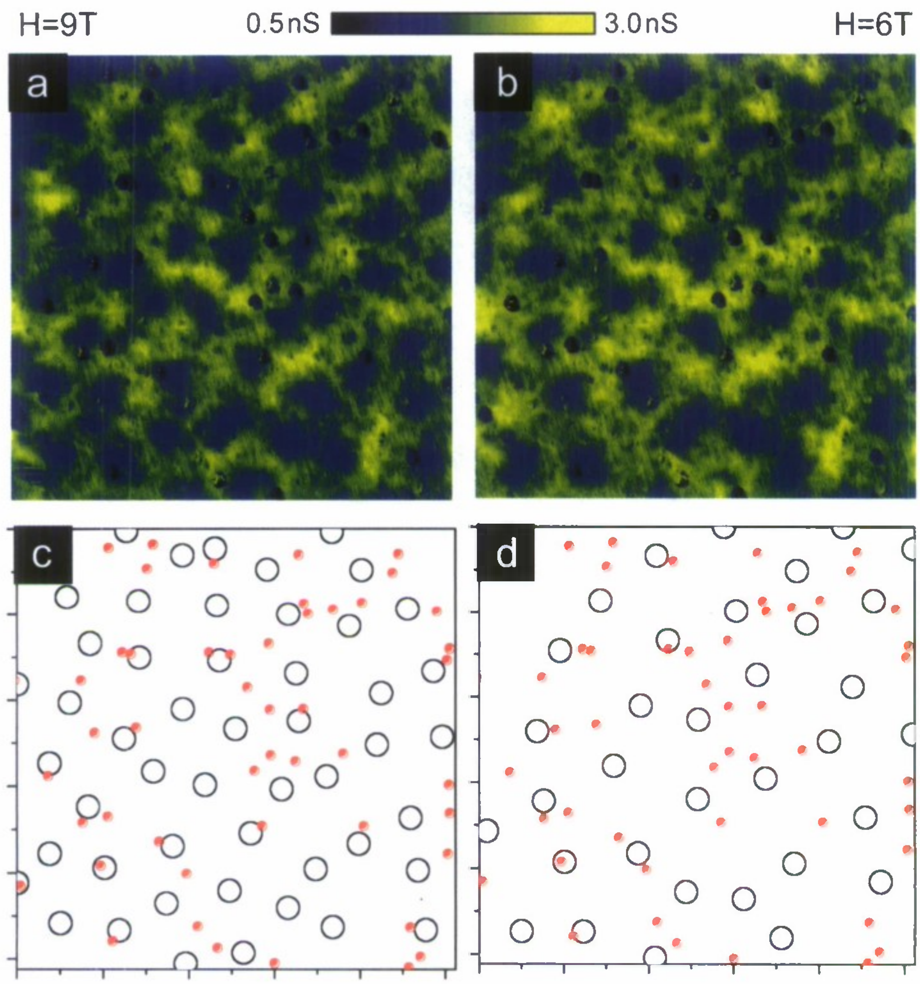

Figure 4: 100 × 100 nm^2 differential conductance maps recorded in **(a)** 9T and **(b)** 6T magnetic field, revealing the sample DOS at 5 meV. Vortices appear as dark features due to the suppression of the coherence peaks inside the vortex core. Vortices form a disordered lattice. The average per-vortex flux values of $\bar{\phi}(9T) = 2.05 \times 10^{-15}$ T·m^2 and $\bar{\phi}(6T) = 2.17 \times 10^{-15}$ T·m^2 are in good agreement with the expected superconducting magnetic flux quantum $\Phi_0 = 2.07 \times 10^{-15}$ T·m^2. In addition to the vortices, impurities are visible in (a) and (b), appearing as smaller, deeper coherence peak depressions. There is no statistically significant correlation between vortex and impurity locations. Idealized data in frames **(c)** and **(d)** depict an idealized version of the same data showing both vortices (open black circles) and impurities (filled red circles).

(3) The vortex-induced sub-gap density of states fits an exponential decay from the vortex center, from which we extract a coherence length $\xi = 27.6 \pm 2.9$ Å, corresponding to an upper critical field $H_{c2} = 43$ T.

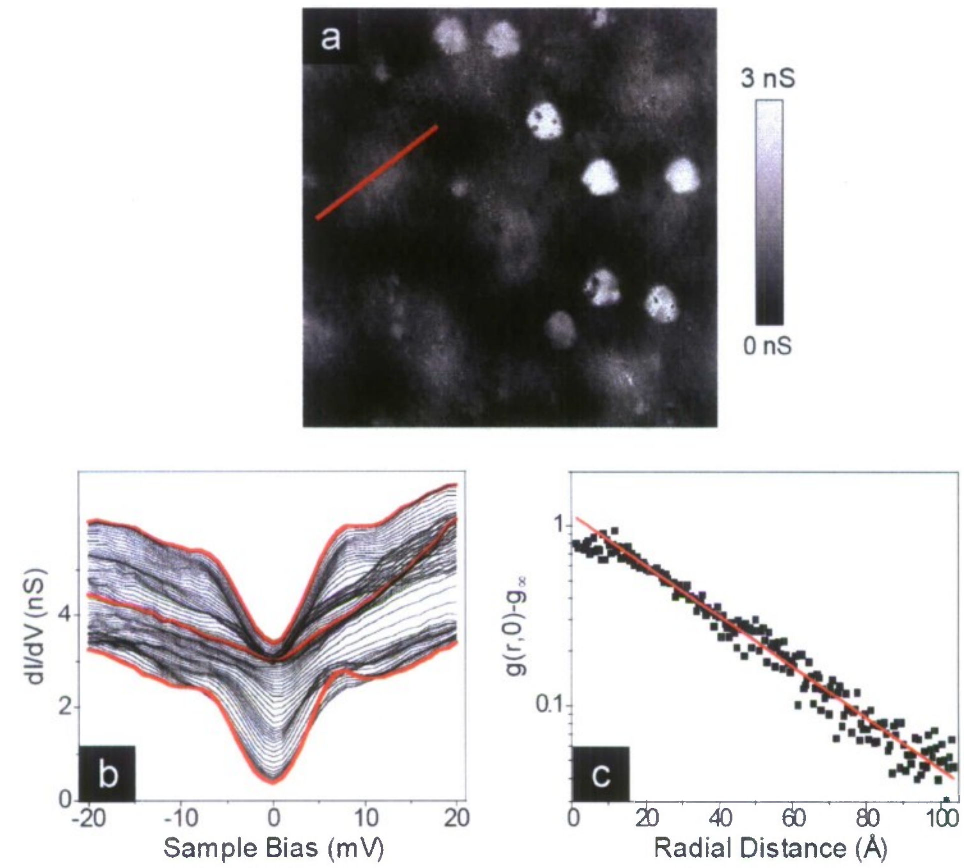

Figure 5: (a) A 40×40 nm^2 map of the zero bias conductance in 9T magnetic field at T = 6.15 K, showing ~8 vortices (broader, lighter objects) and 8 strong-scattering impurities (sharper, brighter objects). **(b)** A series of spectra along a 14.5 nm trajectory through one of the vortices, indicated by the red line in (a). The superconducting gap is completely extinct inside the vortex core; only the V-shaped background remains. The three thick red lines emphasize the spectra at the vortex core and far from it on both sides. The 75 spectra shown are offset by a total of 3 nS for clarity. **(c)** Azimuthally averaged radial dependence of the differential conductance g(r, 0) around a single vortex as measured (black squares) and fit by an exponential decay (red line). The constant background g_∞ has been removed in order to emphasize the exponential decay on the logarithmic scale of the y-axis. The exponential fit leads to a coherence length of $\xi = 30.8 \pm 1.4$ Å for this vortex and to an average of $\overline{\xi} = 27.6$ Å with standard deviation 2.9 Å for all vortices investigated.

Conclusions:

In conclusion, a new high resolution, low temperature scanning tunneling microscope system has been designed, constructed, and successfully tested. The overall project was slowed by Harvard's two year delay in constructing the laboratory to house the experimental system, however important results on a vortex liquid in $Bi_2Sr_2CuO_{6+x}$ and on vortex pinning in the new iron pnictide superconductor $Ba(Co_xFe_{1-x})_2As_2$ have been obtained with the new system. The door is now open for quick turn-around, high resolution, high field studies of vortex pinning in any promising new high-T_c superconductor.

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