Scanning tunneling microscopy and spectroscopy at very low temperatures

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Abstract. During past decades, an increasing number of laboratories is using cryogenic scanning tunneling microscopy and spectroscopy (STM/S) to probe different kinds of electronic systems. Measurements in a dilution refrigerator are particularly useful to study superconductors, because temperatures of order of 100 mK are well below most critical temperatures and effectively reduce thermally excited quasiparticles. The local electronic density of states is then obtained at atomic level with a resolution in energy of some tens of µeV. Visualizing spatial variations of the local density of states allows characterizing vortex cores and the vortex lattice. Vortex core electronic features provide the anisotropy of the superconducting properties, and help understanding the influence of competing orders such as charge density waves. Here we will review results in dichalcogenide superconductors, in the magnetic borocarbide TmNi$_2$B$_2$C and in thin films, discussing in some detail a few relevant aspects of thermal depinning and melting in thin films.

1. Introduction
Tunneling spectroscopy at zero field can be used to obtain the electronic density of states of a superconducting sample. The resulting tunneling conductance vs bias voltage is the temperature smeared density of states, integrated over the reciprocal space volume occupied by electrons participating in the tunneling process [1]. In a STM experiment, tunneling is made by using an atomically sharp tip, providing images of the spatial variation of the tunneling conductance at the surface of the sample [2–4]. STM spectroscopy of superconductors at the surface has been proven to successfully provide a good measure of the bulk electronic density of states in many materials [2–14].

In Fig.1 we plot the superconducting tunneling conductance obtained in several materials in our laboratory [5–14]. We also show its position in a plot of the critical temperature $T_c$ vs the Fermi temperature $T_F$ (Uemura plot [15]). Systems with nearly free electrons have very low $T_c/T_F$ and are located on the bottom right part of the plot, whereas systems with strong electronic correlations have $T_c/T_F$ approaching $10^{-2}$-$10^{-1}$. It is obvious that materials showing bulk properties at the surface are spread over the whole Uemura plot. STM is thus a very powerful technique to obtain the superconducting density of states in many superconductors of topical interest.
Figure 1. Tunneling spectroscopy of several superconducting materials, obtained with the STM at temperatures well below the superconducting $T_c$. We also show the position of each compound in a plot of the critical temperature $T_c$ vs Fermi temperature $T_F$ (dashed line is $T_c = T_F$). The shape of the superconducting density of states is very different in each compound, showing the variations of the superconducting gap over the Fermi surface. Finding low energy excitations does not necessarily show strong electronic correlations, but can be also due to large gap anisotropy or pair breaking. Data adapted from Refs. [5–14].
The vortex lattices of several of these compounds have been measured using STM. They are more difficult to obtain than just the superconducting density of states, because scanning conditions must be good over areas spanning many hundreds of nm. Here we will discuss results about the vortex core of the anisotropic superconductor 2H-NbSe$_2$, about vortex lattice symmetry in crystalline superconductors, and about the vortex lattice in an amorphous W-based thin film.

2. Scanning tunneling microscopy at very low temperatures

![Diagram of scanning tunneling microscope](image)

**Figure 2.** In a we show a simple arrangement of a cryogenic tunneling microscope. The microscope (A) is posed or suspended on the bottom of a dilution refrigerator cryostat or another cooling machine, eventually at the center of a superconducting solenoid. The microscope may be isolated from vibrations using fiber glass, as discussed in Ref. [16]. In b we show a schematic picture of one microscope. Piezostacks (B) are used to move a prism with a scanning tube inside (C). The tip (D) is located on top of a sliding sample holder (E). The sample holder is moved using a wire (not shown, see Ref. [16]) that goes up to ambient temperature. Eventually, a beam (F) is located in the way of the sample holder to break the sample. The whole microscope is made of Ti, using non-magnetic springs. We have built microscopes using similar designs with diameters ranging from 30 mm up to 50 mm.

Successful microscopy requires an environment free of vibrations, which is inherently rather difficult to achieve at very low temperatures in a dilution refrigerator [16]. In Fig.2 we describe schematically a cryogenic system with a scanning microscope inside (Fig.2a) that is shown in more detail in Fig.2b. Useful tricks to reduce damping out mechanical noise from pumps are
sandboxes, silicone tubing (even for the helium 3 pumping lines), increasing the rigidity of the cryostat as a whole, and surrounding the dewar with acoustic foam (such as for example Copopren®). Designs of a microscope often include an exchangeable sample and tip holder, which can be moved back to ambient conditions using a long stick. Eventually, a vacuum preparation chamber is attached to the top or the bottom of the cryostat to allow for sample and tip preparation [17–20]. In Ref. [16] we discuss in detail a procedure consisting of in-situ sample preparation by cryogenic cleaving or breaking a surface using a sliding sample holder. We have applied this technique successfully in dichalcogenides, pnictides, heavy fermions and borocarbide materials. We prepare our tips using soft metals as Au, Al or Pb, and clean them in-situ by controlled repeated indentation in a sample made of the same element [21].

Temperatures of order of 100 mK and below can be achieved by using high conductivity wires between the cold plate and the microscope, and carefully thermalizing them [4]. To meaningfully measure at these temperatures, however, the resolution in energy should be below a few tens of µeV, which is difficult to achieve. The best method, in our view, to test the energy resolution of the system is to measure the tunneling spectroscopy of superconducting tip and sample of a simple s-wave superconductor as Al [22]. At low enough temperatures, the sharpness of the quasiparticle peaks should be independent of temperature if sample and tip are far enough to avoid Andreev reflection, and only shows smearing due to jitter of the voltage source [6].

3. The vortex core and superconducting anisotropies induced by charge order in 2H-NbSe₂

In a superconducting vortex, the superfluid density, or the square of the pair wavefunction, decreases towards the core, and vanishes exactly at the vortex center. In the clean limit, in-gap quasiparticle excitations appear throughout the core due to multiple Andreev scattering [25]. The density of states at each position and energy inside vortex core is the result of the combination of the different quantized states at the core weighted by their corresponding probability density. At the vortex center the probability density is higher for the fundamental state which gives a peak located usually very close to zero bias (actually, at the zero point energy Δ²/2EF, with EF being the Fermi energy and Δ the superconducting gap). At a distance of the core center, higher energy states present a larger probability density and thus contribute more to the tunneling conductance. The first observation of vortex core in-gap states was made by Hess et al in 2H-NbSe₂ [26–28], who carefully followed the spatial dependence of these states. In particular, they found that the tunneling conductance is highly bias voltage and position dependent. At the center of the vortex core (Fig.3a), there is a pronounced peak which splits in energy when leaving the core (Figs.3 and 4). The splitting depends on the position, and we can identify two length scales, shown by the dashed lines in Fig.3a. These length scales correspond to the vortex core extension along the red lines (insets of Fig.3a) and provide the anisotropic star shaped vortex core.

We can discuss this behavior by assuming that the extension of core states is given by an effective coherence length ξ_{eff} = ℏv_F / 2πΔ where v_F is an average over all Fermi velocities of possible Fermi surface orbits at a plane perpendicular to the magnetic field, weighted by their respective contribution to the density of states, and Δ depends on the reciprocal space dependence of the superconducting gap at a plane perpendicular to the magnetic field direction [29]. The anisotropy of the vortex core observed in 2H-NbSe₂ shows that superconducting gap and Fermi surface have a strong six-fold modulation [30, 31]. This anisotropy is absent in 2H-NbS₂, a compound having a very similar T_c than 2H-NbSe₂, but no charge density wave, suggesting that the charge density wave provides the six-fold anisotropy in ξ_{eff}.

We can understand the vortex as a perturbation of the superconductor, and the surrounding patterns in the density of states show the anisotropy of the superconducting properties in reciprocal space. The patterns are wakes of extended quasiparticle excitations and their length
Figure 3. a) shows spatial variation of superconducting density of states in vortex cores of 2H-NbSe$_2$ at 0.03T and 0.1K in two different directions: along the ray (top panel) and in between rays (bottom panel). At the vortex center, we find a zero bias peak due to Andreev quasiparticles at the lowest quantized energy state. When moving out of the vortex core, the zero bias peak splits into two peaks that move to higher bias voltages (blue dotted lines in figure). The way the central peak evolves with energy and position is different along the two directions. b) shows Andreev localized states inside vortex cores of 2H-NbS$_2$ at 0.1 K and 0.15 T. Similarly to 2H-NbSe$_2$, we find a zero bias peak at the core center and higher energy states when moving out of the vortex core. But the tunneling conductance is radially symmetric. Data adapted from Refs. [23,24].

depends on the shape of the Fermi surface and on the anisotropy of the superconducting properties [29,32,33]. Similarly, other point like perturbations, as a point defect in a metal, also produce anisotropic scattering processes which provide real space image of the reciprocal space electronic properties [34,35]. By imaging the electronic properties in real space around a vortex, we obtain a picture of the anisotropies of the superconductor over the Fermi surface [23,24].

Note that the six-fold modulation is found at all energies in 2H-NbSe$_2$ (Fig.4), but the rays present at zero bias split and rotate when approaching the bias voltage of the quasiparticle peak. In 2H-NbS$_2$, the cores remain radially symmetric at all energies. The splitting in 2H-NbSe$_2$ has been reproduced by introducing a six-fold cosine modulation to the superconducting gap [31], suggesting that the anisotropy in $\xi_{eff}$ is mostly due to the superconducting gap anisotropy. Band structure calculations, on the other hand, show that there is a maximum in the non-interacting susceptibility along the directions where $\xi_{eff}$ is largest [36].

Conductance maps for several magnetic fields are shown in Fig.5. In 2H-NbSe$_2$, the in-
Figure 4. Conductance maps showing the spatial evolution of vortex core states at different energies in 2H-NbSe$_2$ (top panels) and 2H-NbS$_2$ (lower panels), both at 0.1K and 0.15 T. The changes in the conductance are given by the color scale at the right part of the figure. In 2H-NbSe$_2$ we find rich patterns with strong sixfold anisotropy. In 2H-NbS$_2$ we find rounded vortex cores at all energies.

The plane sixfold anisotropy is more pronounced at low magnetic fields although it is also present at magnetic fields as high as 1T. This is likely due to overlapping effects between neighboring vortices which become significant at high fields where intervortex distance is within the same order of magnitude than the superconducting coherence length. For instance, the vortex image at 0.03T, where the intervortex distance is 290 nm, does not show overlapping between vortex core states of neighboring vortices. When increasing the magnetic field in 2H-NbSe$_2$ we observe overlap effects already at relatively low fields. At the middle point between three adjacent vortices we find a local enhancement of the density of states which increases (Fig.5a) due to overlap between the rays of the sixfold star shape of vortices at the point where they intersect. Note that the intersection is not located along the line of nearest neighbor vortices, but along the line where the core states have a larger extension. Similar vortex core merging effects have been predicted to occur in nanostructured superconductors [37].

At high magnetic field, we observe that the contrast rapidly decreases, due to pair breaking effects. In multiband superconductors such as 2H-NbSe$_2$ or 2H-NbS$_2$, the multigap structure is affected by the magnetic field, with a destruction of smaller sized superconducting gaps [38], and an extension of associated core states.

Thus, the spatial and magnetic field variations of superconducting vortices provide a useful probe of multigap structure and spatial anisotropies of the superconducting state. Here we have
illustrated the application of this technique to the case of 2H-NbSe$_2$, and shown that the charge order significantly shapes its superconducting anisotropy. The multigap features are shared with 2H-NbS$_2$, and are probably related to the similar bands crossing the Fermi level in these materials.

Band structure calculations indicate that the charge density wave in 2H-NbSe$_2$ is not related to Fermi surface features but rather to electron-phonon coupling [36]. The charge density wave gives spatially modulated normal state density of states along the [100] direction. Core states in 2H-NbSe$_2$ have largest extension along the [110] direction of the crystalline lattice. Further work is needed to determine the relationship between the anisotropy of the normal state density of states and of the superconducting gap with the vortex core features. Interestingly, superposed to the spatial variation of the Andreev core levels, we also find an atomically modulated superconducting density of states. The atomic size modulation follows the charge density wave. Calculations show that similar results at zero field can be explained by an anisotropic tip and an anisotropic superconducting gap [23]. It is yet unclear how to relate the atomic size modulation with the vortex core features. Similarly shaped tunneling conductance maps are found in the cuprate superconductors, where vortex cores show a checkerboard pattern [39]. A comparative study between 2H-NbSe$_2$ and 2H-NbS$_2$ could bring light into the actual relationship of the charge density wave in the atomic size superconducting density of states inside vortex cores.

Figure 5. Zero bias conductance maps at 0.1 K and for different values of the magnetic field in 2H-NbSe$_2$ (a) and in 2H-NbS$_2$ (b).
Figure 6. Zero bias conductance maps at 0.1 K and 0.03 T showing the spatial changes of the superconducting density of states at $E_F$ on the scale of the coherence length around an isolated vortex and at atomic scale in the small region marked by a black square in the figure. Lower right panel shows the tunneling conductance measured when the tip is on top of a Se atom (green) and in between Se atoms (blue). Data adapted from [23].

4. The vortex lattice and crystalline anisotropies

Anisotropic superconducting properties also influence vortex arrangements and can modify the vortex lattice symmetry with respect to the conventional hexagonal configuration. In superconductors with hexagonal crystalline lattice as $2H$-NbSe$_2$ and $2H$-NbS$_2$, the vortex lattice remains hexagonal and is always oriented with the atomic lattice. In superconductors with different crystal symmetries such as tetragonal or orthorhombic, the vortex lattice may form a square or distorted hexagonal lattice. In the nickel borocarbide superconductors STM and neutron diffraction measurements have shown hexagonal to square transitions in the vortex lattice as a function of temperature and magnetic field [41–43]. In TmNi$_2$B$_2$C such a transition has been observed by STM when increasing the magnetic field parallel to the c axis [44]. When the magnetic field is parallel to the basal plane, the anisotropy induced by the tetragonal crystal structure is weaker and the transition to the square vortex lattice occurs at higher magnetic fields [44]. The hexagonal vortex lattice is nevertheless oriented with one of its high symmetry axis parallel to the c-axis, as shown in the inset of Fig.7.

The vortex arrangement is not only influenced by the crystalline and Fermi surface symmetries but also by other existing kinds of order which break symmetries of the superconductor. A relevant case is found in magnetic superconductors where ferromagnetic or antiferromagnetic ordering have a significant influence in the superconducting properties [41, 45]. Excellent
Figure 7. Internal magnetic field estimated from the intervortex distance as a function of the applied magnetic field for different superconductors: TmNi$_2$B$_2$C (black circles) and 2H-NbSe$_2$ (white circles). Continuous black line represents $B_{int} = B_{ext}$, which means that the intervortex distance follows the expected magnetic field dependence $d = (\frac{4}{3} \frac{\Phi_0}{B})^{1/2}$. Dashed line gives $B_{int}$ in TmNi$_2$B$_2$C estimated from magnetization data from Ref. [40]. Inset shows hexagonal vortex lattice obtained at 0.1 K and 0.23 T with the field applied within the plane of the tetragonal structure of TmNi$_2$B$_2$C, i.e. perpendicular to the c-axis. Red arrows show the direction of the crystalline axes a and c. Data adapted from Ref. [44].

examples are the nickel borocarbides which remain the only family of magnetic superconductors where vortex lattice has been studied in depth.

In TmNi$_2$B$_2$C we observe that the intervortex distance is slightly lower than expectations, indicating that the internal magnetic field is higher than the applied magnetic field. Fig.7 shows the intervortex distance in TmNi$_2$B$_2$C compared with the intervortex distance found in 2H-NbSe$_2$ and in 2H-NbS$_2$. Clearly, magnetism of TmNi$_2$B$_2$C gives slightly denser vortex lattices, possibly inducing anomalous behavior in other macroscopic properties such as the upper critical field.

5. Thermally activated vortex lattice
A generic phase diagram of type II superconductors is shown in Fig.8. When fluctuations become important, a vortex liquid phase appears at the boundary between the Shubnikov phase and the normal and Meissner phases (Fig.8b). The liquid occupies a large region of the phase diagram in many superconductors [3, 46–49].
Figure 8. Generic phase diagrams of type II superconductors where thermal fluctuations close to the transition to the normal state $B_{c2}(T)$ are negligible (a) and relevant (b) giving a vortex liquid phase. In b) $B_m(T)$ is the melting line which separates the vortex solid and liquid phases.

Vortex lattice melting in amorphous W-based thin films deposited using focused ion beam [50] has been observed in Ref. [51]. A good measure of the strength of the action of fluctuations in the vortex lattice is provided by the Levanyuk-Ginzburg parameter $LG = \frac{1}{2} \left( \frac{k_B T_c(0)}{4\pi \mu_0 B_{c2}^2(0) \xi^2(0)} \right)^2 \approx 10^{-7} \kappa^4 T_c^2(0) \frac{B_{c2}(0)}{B_{c2}(0)}$ ($\kappa$ being the Ginzburg-Landau parameter) [52, 53]. $LG$ compares the condensation energy in a volume of order of the coherence length with the thermal energy at critical temperature. In conventional superconductors $LG$ is very low, around $10^{-11}$ to $10^{-14}$ and the vortex liquid eventually exists in an extremely narrow region. The melting line $B_m(T)$ practically coincides with $B_{c2}(T)$. Cuprate high $T_c$ superconductors have large values of $LG$, around $10^{-1}$ to $10^{-3}$, due to their high critical temperatures, small coherence length and large anisotropy. These superconductors are characterized by the presence of a extended region in the phase diagram B-T where thermal fluctuations induce the formation of a vortex liquid (Fig.8b). Thin films of amorphous superconductors which are often extreme type II superconductors with relatively low values of $T_c$ have values of $LG$ rather close to those found in cuprate superconductors, with $LG \approx 10^{-4}$ and have showed a small but sizeable range close to $B_{c2}(T)$ with a vortex liquid phase [50].

Before melting takes place, the thermal de-pinning transition may occur. De-pinning happens when the vortex lattice is frozen into a distorted hexagonal lattice by interaction with defects. The depinning transition in the W thin films has been directly obtained by imaging the changes in vortex arrangements induced by temperature at different magnetic fields. Fig.9a shows the
Figure 9. a) Shows the angular distortion of the hexagonal vortex lattice in W amorphous superconducting thin film across the de-pinning transition, taken at a magnetic field of 1T. The de-pinning transition occurs sharply in the temperature interval between 1.5 K and 1.6 K. b) shows a sequence of vortex lattice images taken during the de-pinning transition where blue and magenta hexagons highlight respectively the low and high temperature vortex arrangement. Data adapted from Ref. [51].

thermal de-pinning of the lattice at 1T. At low temperatures the hexagonal lattice is distorted due to pinning. When increasing temperature, thermal energy increases above pinning energy and vortices arrange forming an undistorted hexagonal lattice. The de-pinning transition is very sharp. Here it occurs in the temperature interval between 1.5 K and 1.6 K.

Below and above the depinning transition, the lattice remains stable with time. But at the depinning transition we observe time dependent rearrangements. We have followed the behavior of the lattice during one hour, taking eight consecutive vortex images at each temperature. In the temperature range from 1.5 K to 1.6 K, the vortex arrangement changes between two well defined configurations, which are highlighted in Fig.9b by blue and magenta hexagons. These correspond respectively to the vortex arrangement found at low temperature (blue hexagon) which follows the pinning potential, and the vortex configuration observed after the de-pinning transition (magenta hexagon), freed from the local pinning potential. Most dramatic changes between both configurations involve vortices between pinning centers, always moving coherently in a particular direction. On the other hand, the mobility of the vortices close to pinning features is reduced. This indicates that the de-pinning in thin films is dominated by thermal activation of vortex bundles formed between surface pinning centers at low temperatures. Images taken during the transition at 1.5K and 1.6K show an increase in the frequency ($R$) of the vortex
rearrangement from one oscillation each hour at 1.5 K (three representative images, of the eight made at 1.5 K are shown in top panel of Fig.9b) to eight oscillations each hour at 1.6 K (images at bottom panel of Fig.9b). We can estimate the local pinning strength using an Arrhenius law given by $R = R_0 e^{-U/k_B T}$, where $R_0$ is the characteristic frequency for thermal fluctuations (typically $10^{-11} s^{-1}$) and $U$ is the activation energy associated to the pinning potential barrier. Using the frequency found in the experiments, we obtain a value for $U$ of about 50 K which is comparable to the values obtained from macroscopic measurements in superconducting thin films with similar properties. This value is also consistent with the pinning energy associated to a surface corrugation of around 10 nm, as found in the experiment.

![Figure 10](image)

**Figure 10.** a) Temperature dependence of the Fourier amplitude (white circles) and average conductance $\sigma_{\text{average}}(x,y)$ (black circles) of the vortex lattice images taken during the melting process. The formation of the isotropic liquid occurs at the temperature $T_{\text{isotropic}}$ where the Fourier amplitude becomes zero whereas the transition to the normal state is found at higher temperatures, at the point $T_c$ where $\sigma_{\text{average}} = 1$. b) Temperature dependence of the Lindemann number in the two dimensional vortex lattice. Black dotted line marks the temperature at which melting process starts with the first appearance of dislocations in the images. At the transition $c_L$ has a value around 0.2 in good agreement with the Lindemann criterion. Insets show vortex lattice images at the hexatic and smectic phases. Data adapted from Ref. [51].

In Ref. [51], we acquire conductance maps showing the vortex lattice as a function of temperature to reach vortex melting. We identify different vortex phases, from the vortex solid to an isotropic vortex liquid. A hexatic phase with orientational order and a smectic like phase showing 1D vortex arrangements were observed in between the solid and isotropic liquid.
phases. Temperature dependence of the Fourier amplitude and average conductance at zero bias of the vortex maps show the temperature region at which isotropic vortex liquid forms (Fig.10a).

Lindemann criterion in the 2D melting has been discussed for different systems [54]. Due to the continuous nature of the melting transition, as opposed to first order abrupt transition, it is natural to ask how large the amplitude fluctuations are when entering the liquid phases. In Fig.10b we show that Lindemann’s criterion is satisfied when entering the hexatic phase. We plot the amplitude of the positional fluctuations due to thermal smearing as a function of temperature. The melting transition is expected when the mean quadratic displacement with respect to the equilibrium positions ($u^2 > 1/2$) is equal to a fraction of the lattice parameter of the ordered solid ($d$, $u^2 > 1/2 \approx c_L d$, where $c_L$ is empirically found to be around 0.1-0.3 in most systems. We estimate $c_L$ from the reduction of the amplitude in the Fourier transforms of the vortex lattice images due to the arrival of disorder during the melting process ($A_{FFT}^{th}$). We use that $A_{FFT}^{th}$ is proportional to $e^{-2\pi c_L^2}$ [55]. To obtain $A_{FFT}^{th}$ we renormalize the Fourier amplitude at each temperature by a term proportional to $(1 - \sigma_{average}(0))$. This term accounts for temperature induced reduction of the superconducting gap and increased thermal smearing in the tunneling conductance, which both deplete the Fourier amplitude. Thus, the renormalization allows us to subtract from the Fourier amplitude any temperature dependence which is not due to the increased disorder during melting. We assume that $u^2 > 1/2$ is negligible at low temperatures so that $c_L = \sqrt{-\ln(A_{FFT}^{th}/A_{FFT}^{th}(lowT))/2\pi}$. As shown in Fig.10b, we find that $c_L$ is close to 0.2 when we observe the start of the melting process in our experiments. We also observe a slight increase of $c_L$ above the melting temperature which could be due to enhanced vortex motion within the hexatic phase.

6. Conclusion and outlook

We have discussed imaging experiments with the STM which provide direct measurement of the superconducting anisotropies, of the interaction between crystalline and vortex lattices, and of thermally induced processes such as vortex lattice melting. Measurements in nanostructures, as a function of the applied current and in strongly correlated electron systems are now ongoing by several groups. They will provide vivid images of vortex behavior and of the electronic structure that will improve our understanding of superconductors.

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