A tunneler’s view on correlated oxides and iron based superconductors
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APPENDIX

C

Temperature calibration measurements

Before conclusions can be drawn from temperature dependent measurements such as the ones presented in chapters 6 and 9, the temperature of the sample in the STM should be calibrated. In the first section of this appendix, the sample temperature will be discussed which was measured with a thermometer mounted on a sample holder and inserted into the STM. The response of the sample temperature to a heater mounted on the base of the STM is shown. In the second section, the settings of the PID temperature controller are briefly discussed. In the third section, the effective temperature during a measurement at various sample temperatures is discussed using temperature dependent measurements of the superconducting gap of the BCS superconductor niobium. The last section briefly discusses the temperature dependence of the piezo constants.

C.1 Sample temperature

As has been shown in chapter 2, Fig. 2.3, the STM head is equipped with a silicon diode thermometer and a diode heater to regulate the temperature. Even though the sample holder and sample are thermally connected to the main STM block where the two diodes are fixed to, they could in principle still have a differing temperature due to radiative heating through one of the viewports directed at the sample area. In order to determine if the sample temperature deviates from the temperature measured by the thermometer on the STM block, a thermometer has been mounted on a sample holder and placed in the STM. For a given set temperature (of the STM block), the temperature of the sample was monitored. At the base temperature of 4.6 K, both the STM block and sample gave identical readings.

Upon increasing the temperature by heating, or decreasing by pumping on the He cryostat, both the sample and STM block require a certain amount of time to come to a new equilibrium. An example of such an exponential reaction to the equi-
Figure C.1: (a) Relaxation of the sample- and STM block temperature as a function of time after a change in temperature from 20 K to 25 K. The sample temperature is fitted with an exponential of the form $y = y_0 + \exp(-x/\tau)$, where $\tau \sim 9$ seconds. In the fit, only data points are considered from the moment the STM block has reached its equilibrium temperature. The higher the difference between initial and final temperature and the higher the absolute final temperature, the higher the relaxation time $\tau$. (b) Difference between the sample- and STM block equilibrium temperature as a function of temperature. For temperatures up to 300 K the difference is always less than a few Kelvin.

The difference in temperature between the set temperature (i.e. STM block temperature) and sample temperature for various set temperatures is shown in Fig. C.1b. In this instance, the sample was held at a constant temperature of 20 K before the heater was set to 25 K. As can be seen, the STM block temperature reaches the set temperature very rapidly, due to a proper setting of the PID controller (see section C.2). As there are several mechanical interfaces between the sample and the block that contains the heater and temperature sensor, the relaxation of the sample temperature is somewhat slower. A fit to the sample temperature from the moment where the STM block has reached its equilibrium temperature (solid line in Fig. C.1a) gives a decay constant of approximately 9 minutes. For any temperature step, the decay constant is on this order of magnitude, although it increases for increasing absolute temperature and temperature difference between original and final temperature. In any case, after approximately half an hour, the system is more or less in its new equilibrium and tunneling contact can be established again.

The difference in temperature between the set temperature (i.e. STM block temperature) and sample temperature for various set temperatures is shown in Fig. C.1b. In the temperature range used in chapter 6, the deviation is always less than 0.8 K, ensuring that the conclusions drawn from the temperature dependence are valid. For temperatures up to 300 K, the difference has been checked to be less than a few K.

\(^1\) due to the temperature dependence of the piezos, it is not safe to remain in tunneling contact during heating as the tip-sample distance will change as a function of temperature.
C.2 PID settings

The Neocera LTC-21 temperature controller used to regulate the temperature of the STM uses a PID algorithm to stabilise the temperature. This algorithm adjusts the heater settings using three parameters: (i) the Proportionality term, which is proportional to the difference between the actual and set temperature (ΔT) times a constant $P$, (ii) the Integral term, which is proportional to the time integral over the temperature difference over a time $I$ (i.e. $\int_0^I \Delta T dt$) and (iii) the Derivative term, which is proportional to the time derivative of the temperature difference ($\frac{dT}{dt}$) times a constant $D$. Using the Ziegler-Nichols tuning method [281], the optimal settings for this temperature controller and STM were found to be $P = 100000$, $I = 9$ sec. and $D = 2.25$ sec.

C.3 Temperature dependence of a Niobium superconductor

Having established that the sample temperature is nearly identical to the set temperature, the question remains what the effective temperature is of the STM, and how broadening due to experimental noise affects the spectra. The main motivation for this investigation, aside from a general characterisation of the system, is that the gaps seen on the ‘122’ pnictides always have a rather shallow gap minimum. This apparent filling of the gap could be intrinsic to the ‘122’ pnictides, for instance due to large scattering effects or a non-superconducting band at the Fermi level. However, it could also be an experimental issue, where the spectra are smeared by either an effective temperature that is different from the sample temperature or a large experimental broadening factor $\Gamma$ which could furthermore be temperature dependent.

An ideal system to do characterisation measurements on is the BCS superconductor niobium (Nb), which has the largest transition temperature of all pure elements - $T_c = 9.2$ K for very pure Nb - well above the STM base temperature. The gap size $\Delta$ has been shown to be 1.52 mV [282], similar to the smallest gaps reported in literature on the pnictide superconductors [283]. Therefore, detection of the Nb gap would show that if gaps of such size would be present in the materials studied, they would have been picked up. Furthermore, by fitting the s-wave gap of Nb (if detected) with the BCS equation for the density of states, i.e. Equation (3.4), the effective temperature and broadening can be extracted as a function of sample temperature.

As there was no Nb single crystal available at the time, a Nb tip was used to measure the Au(788) characterisation sample, which should give identical results. Fig. C.2a shows the tunneling spectrum thus obtained at a measurement temperature of 5 K. From the BCS fit, the superconducting gap was found to be $\Delta = 1.47$ mV, with an effective temperature of $T = 7.1$ K, and a broadening of $\Gamma = 0.17$ mV. Although the spectra are thus slightly more broadened due to an effective temperature that is larger than the sample temperature, the experimental broadening is rather small and the resolution overall is rather good. The slightly lower value for the superconducting gap is not unusual (see for instance Ref. [153]), but not well understood. The
C. Temperature Calibration Measurements

Figure C.2: (a) Tunneling spectrum of a Nb tip on an Au(788) sample taken at 5 K, with $\Delta = 1.47 \text{ mV}$, $\Gamma = 0.17 \text{ mV}$ and $T = 7.1 \text{ K}$ from the fit. (b) Temperature dependence of a tunneling spectrum taken with a Pt/Ir tip on a polycrystalline Nb sample. The spectra are normalised to a spectrum taken at 9 K, the lines are BCS fits to the experimental data, giving $\Gamma = 0.02 \text{ mV}$ for all spectra, $T = 13 \text{ K}$ for the 2 K spectrum and $T = 17 \text{ K}$ for all other spectra. The extracted gap sizes are plotted in (c) and are in excellent agreement with BCS theory (solid line), giving $\Delta_0 = 1.50 \text{ mV}$. (d) Spectra taken on (Pb,Bi)$_2$2212, both gapped (red = 4.3 K, orange = 20 K) and peaked around zero bias (blue = 4.3 K, green = 20 K). Thermally broadening the spectra taken at 4.3 K to 20 K (black dashed lines) matches the high temperature spectra perfectly. As the gap itself is nearly constant over this temperature range ($T_c = 90 \text{ K}$), experimental thermal broadening alone can account for the difference between the temperatures.

Measurement shown in Fig. C.2a has been performed using the ‘old’ z-piezo, where a single magnet is used to attach the tip to the piezo (see section 2.1.3 for details).

During the coarse of the research, the z-piezo that holds the tip has been replaced, and in the new design of the z-piezo, the electric contact to the tip has also been changed from a ring-electrode contacting the entire tip, to three point-like contacts. While indeed reducing the magnetic field at the sample position, the new holder...
turned out to have much poorer thermal contact to the piezo. This fact went undetected for approximately a year and only at the end of this Ph.D. research it was decided to characterise the STM once again on a Nb superconductor.

This time, it proved impossible to see a gap using a Nb tip on the Au(788) sample. Therefore, a finely polished polycrystalline piece of Nb was repeatedly sputtered with Ne and annealed to \( \sim 800 \text{ K} \) in vacuum before insertion into the STM underneath a regular Pt/Ir tip. In this configuration the tunneling spectra did show the typical signature of a superconducting gap, and its temperature dependence was investigated. Spectra taken at various temperatures on the same position, normalised to the spectrum taken at 9 K, are shown in Fig. C.2b. Note that the spectrum taken at the lowest temperature (i.e. 2 K) is achieved by pumping on the Helium cryostat from which the STM is suspended by springs. This not only cools the STM itself but also the inner radiation shield, and therefore reduces the radiative heating on the STM itself.

The superconducting gap extracted by fitting the spectra is plotted as a function of temperature in Fig. C.2c and shows excellent agreement with the behaviour predicted from BCS theory given in Eqn. (3.2) (shown in Fig. C.2c as a solid line), from which follows that \( \Delta_0 = 1.50 \) and \( T_c = 7.6 \text{ K} \). From the fits it follows that the experimental broadening, \( \Gamma \), for all temperatures is on the order of 0.02 mV, which is an improvement with respect to the ‘old’ z-piezo. The effective temperature on the other hand is for all temperatures, except the lowest, as high as 17 K. The spectrum taken at 2 K has an effective temperature of 13 K. The only possible explanation is that the tip is at a much higher temperature than the sample: it is well above the transition temperature of Nb explaining the inability to see a gap with a Nb tip, and causes significant broadening of the spectra. It must be noted that the pnictides have also been measured with the ‘old’ z-piezo, also resulting in rather broad spectra, indicating thermal broadening due to a warm tip is not the only factor playing a role. Notwithstanding, the large effective temperature of the current z-piezo is clearly affecting the spectra.

The main reason why the tip is warmer than in the old configuration is probably the poor thermal contact to the tip (by three point-like contacts instead of a ring). As the cooling power is therefore reduced, radiative heating through the view-ports directed at the tip effectively warms the tip up. To counter this effect so as to ensure the tip is at the same temperature as the sample, gold plated window shutters (as radiation shields) are currently in the process of being installed, which in the near future will hopefully lower the effective temperature of the system and increase the resolution.

In closing this discussion, it should be mentioned that the fact that gaps on the order of 1 mV were observed, shows with the current status of the machine, detection of very small gaps is possible. Also, from a temperature dependent measurement on (Pb,Bi)2212 shown in Fig. C.2d, a 86 K superconductor, it can be seen that there is no broadening of the spectra other than thermal broadening as a function of temperature, i.e. the experimental broadening \( \Gamma \) is not temperature dependent. All of these
findings strongly support that if there would be a pseudogap in the pnictides, the temperature dependent study described in chapter 5 would have detected it, even though the effective experimental temperature was higher due to the sub-optimal cooling of the tip.

### C.4 Piezo constants

Exact determination of the piezo constants is essential for proper analysis of the topographic data. Before each measurement, the z-piezo constant is checked on the stepped Au(788) calibration sample. As atoms are difficult to detect on an Au surface, other samples have been used to calibrate the x- and y piezo constants, namely highly oriented pyrolytic graphite (HOPG) and well studied systems such as Bi2212 and (Pb,Bi)2212 (see appendix A). Approximately twice a year the piezo constants were checked using one of these samples and were not seen to vary throughout this research even though piezoelectric crystals are known to age. The x- and y piezo constants at base temperature (4.2 K) are 15.1 Å per Volt, while the z-piezo constant is 7.5 Å per Volt. Increasing the temperature of a piezo increases its piezoelectric response, meaning that the piezo constant increases as a function of temperature. By measuring the size of a particular field of view at base temperature, and comparing this to the same field of view at elevated temperatures, the x- and y piezo constants were determined for various temperatures. Fig. C.3a plots these values versus temperature.

According to Ref. [284], the temperature dependence of the deformation of a
piezoelectric material can be described by:

\[ \frac{\Delta \ell}{\ell_0} = \alpha(0) + cT^n + \beta \ln(t), \]  

where \( \Delta \ell \) and \( \ell \) are the length deformation and undeformed total length respectively, T is the temperature and t is the time. As the slow relaxation given by \( \beta \ln(t) \) is small at low temperatures this term can be omitted. The dashed line in Fig. C.3a is a fit of the data points with Eqn. C.1, whereby n has been fixed to 0.75, giving \( \alpha(0) = 14.1 \) Å/V and \( c = 0.35 \) Å/V. These values are similar to those obtained in a temperature dependent study on various piezo electric materials that includes two piezos from the family of materials used in this study, lead zirconate titanate (PZT)\(^2\) [284]. The summary plot from Ref. [284] is shown in Fig. C.3b for comparison.

Using the parameters from the fit, and assuming the piezo constants have not changed considerably since the last calibration, a good guess of the piezo constants at any given temperature below 40 K can be calculated.

\(^2\)The piezos used in this study are PCC255 by PI Ceramics