Manipulation of superconductivity in van der Waals materials and thin films
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Experimental Set-Ups

This thesis focuses mainly on research related to conventional phonon-mediated s-wave superconductors. This category of superconductors has transition temperatures below liquid nitrogen temperature. To study their properties near the phase transition, transport measurements are carried out at temperatures down to 2 K. To get a complete picture it is necessary to study the interaction between magnetic field and superconductivity. To do this, a tunable uniform magnetic field in the sample space is required. Apart from this, some studies described in the thesis require small injection currents (down to 1 μA) to protect the sample or measure relatively small critical current densities close to the phase transition. With those conditions in mind, a stable cryostat with a tunable magnetic field and tunable electronics is required.

On the sample fabrication side, apart from conventional types of device-fabrication equipment, a very important setup for van der Waals materials manipulation is the 2D transfer system. This special setup allows precise (re-)placement of small and thin van der Waals flakes, making it possible to create heterostructures with one or more types of materials. This chapter will give a brief overview of the cryostat and the 2D transfer system that were used in most of the studies included in this thesis.

2.1. Cryostat for Transport Measurements

The cryostat in which most of the experiments described in the thesis are performed is an Oxford Instruments Teslatron closed-loop cryostat (shown in Figure 2.1). The cryostat has the ability to cool down the sample space to 1.5 K. The cooling system constitutes two helium closed loops; a high pressure circuit and a low pressure circuit. The high pressure circuit contains 16.5 bar of helium gas and connects the Sumitomo F-70 compressor with the RP-082x2S cold head. The pulse-tube-based cold head has two stages, the first stage is cooled down to between 40-70 K, whereas the second stage is kept at below 4 K.

The Variable Temperature Insert (VTI) is not in direct physical contact with the cold head. Instead, it is in thermal contact with the second stage through the helium gas contained in the lower pressure circuit. A needle valve is in place to control the pressure of helium gas flowing between the cold head and the VTI. The gas in the lower pressure circuit carries the heat load from the VTI to the cold head, thereby, providing cooling power to the system. The cooling power is directly related to the amount of helium gas in the circuit, 10 mbar of helium is sufficient to cool down the system to 1.5 K.

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The VTI is in direct thermal contact with the sample through 0.5 bar of helium exchange gas in the sample chamber. There is a thermometer and a heater directly below the sample space for regulating the temperature of the sample as accurately as possible. Surrounding the sample space is a superconducting Nb_3Sn coil magnet capable of providing a uniform magnetic field up to 8 T. It is in physical contact with the second stage of the cold head by a copper braid. And it is kept at temperatures below 4 K, i.e. below the critical temperature of Nb_3Sn.

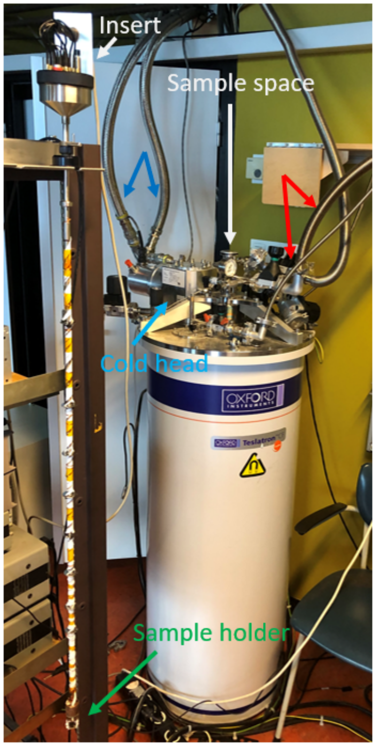

Figure 2.1: A photo of Teslatron in the lab. Key elements are marked in the photo. The blue arrows indicate the high-pressure helium circuit, whereas the red arrows point to the low-pressure circuit.

The magnet can operate in two modes; power supply mode and persistent mode. In power supply mode the magnet is connected with an external power supply, and therefore the field can be freely varied by varying the supplied running current. After the magnet is energised, it can be switched into the persistent mode, where the coil is shorted from the power supply. The supercurrent running in the magnet can run without resistance and power supply for an indefinite amount of time providing a stable magnetic field.

The switching between the two modes is done by a heater that heats a switching wire in the magnet coil. While the heater is on, the superconducting wire is in the resistive stage, so the magnet is not shorted. If the heater is switched off, the wire transits into the superconducting stage and therefore the coil is shorted, allowing the supercurrent to run persistently in the magnet.

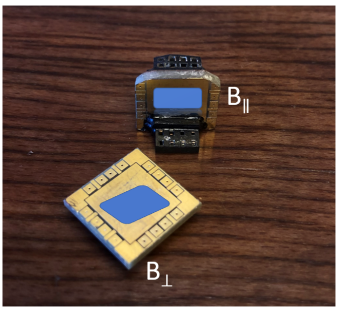

Figure 2.2: A photo of the two types of sample holders. The orientation of the magnetic field related to the sample surface is indicated in the photo.

Both the temperature and the magnetic field can be intelligently controlled with MercuryiTC controller and MercuryiPS magnet power supply. Full automation of a specific measurement process can be realised by remote control of the two systems. As such, Teslatron is a fully automated, closed-loop, liquid-free cryostat that can be continuously operated with minimum maintenance.

The sample is placed into the cryostat sample space by an insert. The insert is wired with 12 coaxial cables (87 pF m^{-1}, 170μm core diameter), which are soldered to a classic sample holder. The coaxial cables are visible in figure 2.1 as white wires around the insert.

Two types of sample holders are available; one places the sample perpendicular to the magnetic field lines, and the other keeps the sample surface parallel to the field lines (home-made by Peter van Veldhuizen). A photo of the two sample holders is shown in figure 2.2. The holders have a space of roughly 8×8 mm^2 for the sample. To ensure good thermal conductivity, the samples are glued on the sample holder with silver paint and electronically connected to the holder by wire bonds. On the outside of the cryostat, the 12 coaxial cables are connected to a room temperature breakout box where the sample can be safely switched between measurement and ground. The current injections in most of the transport experiments described in this thesis are done with a Yokogama GS200 DV voltage/Current source whereas the voltage signals are measured with Keithley 2182A Nanovoltmeter. In its full capacity, the cryostat can cool down and measure 3 devices simultaneously on one or more substrates.

2.2. 2D TRANSFER SYSTEM

The devices based on van der Waals materials in this thesis are made using a commercially available manual 2D transfer system provided by HQ graphene, developed specifically for manipulations of exfoliated van der Waals flakes to build quantum devices.

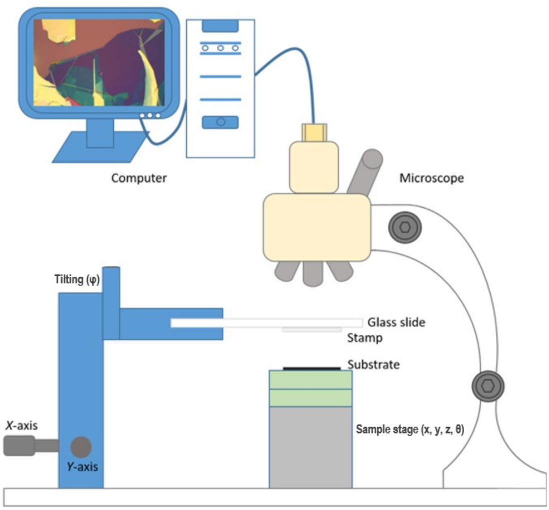

Figure 2.3: A schematic of the stamping set up used for fabricating van der Waals heterostructure devices.

A schematic of the transfer setup is shown in figure 2.3. The set-up itself has two major parts; the sample stage and the stamping stage. The stamping stage can move in the xy-plane, and is kept in place with 4 different screws at each corner. The 4 screws can be moved in the z-direction independently of each others. By moving them with respect to each other, the stamp, which is kept in place by a glass slide, can thus be tilted in the desired manner.

The substrate stage has four degrees of freedom. It can fine rotate along the z-axis to adjust the rotational angle with respect to the rest of the set-up and move in the x-y plane. The stage can also move in the z-direction to bring the sample into contact with the stamp. The stage has a heater that can go up to 200°C, and the sample is kept in place by a dry membrane vacuum pump. The stages are designed such that manual operation can also achieve μm position placement of the stamp relative to the substrate.

There is a microscope directly above the sample stage, so that fine alignment of the stamp to the substrate can be directly observed while operating on the setup. The maximum magnification available for this particular system is 500 times, and it is well sufficient for its purposes.

Specific polymer substances are used for making the stamp depending on which of the van der Waals materials is in question. Detailed stamping processes for each experiment will be discussed in their own chapters. A photo of the stamping stage is also shown in Chapter 6.