Proximity effects in superconducting spin-valve structures

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Chapter 3

Experimental details

3.1 Sample specifics

For this work the standard geometry of our devices are elongated S/F bi-layer and F/S/F tri-layer strips. The lateral dimension are either in the macroscopic regime, with a typical dimension of 100 μm $\times$ 1000 μm, or in the mesoscopic regime, with a typical dimension of 2 μm $\times$ 40 μm. These strips are contacted with non-magnetic, non-superconducting microscopic contacts which lead to large (1 mm $\times$ 1 mm) contacting pads where connections to the measurement electronics can easily be made. The contact geometry to the strip is a 4-probe type with 10 μm respective 1 mm spacing between the voltage probes for the microscopic and macroscopic samples. For the superconductor we use Niobium (Nb), for the ferromagnets we use Copper-Nickel (CuNi) and the Nickel-Iron alloy Permalloy (Py = $Ni_{80}Fe_{20}$) to investigate respectively the weak and strong exchange limit. The contacting material is Gold (Au). The substrates used for our devices are all Silicon (Si) cut along the [100]-plane with a low p-type doping concentration of 10^{13-16} dopants per cm^3. The devices are made in a three-step process: 1) depositing the layers for the strip, 2) defining and etching down the strip and 3) defining and growing the contacts. The details of these three steps are written down below.

In the first step, the metallic layers that comprise the strip are grown onto the Si(100) substrates by DC magnetron sputtering in a ultra high vacuum chamber with a background pressure of 10^{-9} mbar, using Argon (Ar) as plasma. Specific sputtering parameters for the different materials are as follows:

target	**Ar pressure**	**sputtering rate**
Nb	4.0 μbar	0.12 nm/sec
$Cu_{50}Ni_{50}$	4.0 μbar	0.04 nm/sec
$Ni_{80}Fe_{20}$	2.5 μbar	0.20 nm/sec

No additional cooling was used during sputtering, meaning the sputtering temperature starts at room temperature and slowly increases with some tens of degrees. For the sample with Py layers, a specially made magnetic sample holder was used to induce a homogeneous in-plane magnetic field. This sets the direction of the magnetic easy axis and also improves its switching properties. The orientation of the strip is defined parallel to this induced easy axis. Unless specifically mentioned, a thin Nb capping layer (about 2 nm thick) is added on top of the layer package to prevent oxidation of the top ferromagnetic layer. The purity of the Nb target is 99.95 % which yields a T_c of 9.1-9.2 K. The ratio Cu/Ni in the target is 50/50 (atomic percentage), which results in an approximate 43/57 ratio at the sample. The Curie temperature of the

$Cu_{43}Ni_{57}$ is around 150 K and it has a degree of polarization close to zero. The (corresponding) exchange energy is some tens of meV ($\ll E_F$) making it a model weak ferromagnet. For the Py the Curie temperature is much higher, around 950-1000 K, and it has a degree of polarization of about 45 %. The (corresponding) exchange energy is some hundreds of meV, which is a sizable fraction of the Fermi energy, making it a strong ferromagnet.

In the second step the strips are defined by standard lithographic techniques and afterwards etched down. First, a MaN 2405 (a negative-tone resist) is spin coated at 6000 RPM (during 60 seconds) on top of the layer package. Afterwards it is baked for 10 minutes at 90 ^oC. Standard electron beam lithography, using a JEOL JSM 820 Scanning Microscope, was used to pattern the strips and several alignment markers. The beam current was 30 pA and the dose 45 μC/cm^2. As developer, the MaD 332 was used to dissolve the non-exposed areas (in about 30-35 seconds). The thus obtained

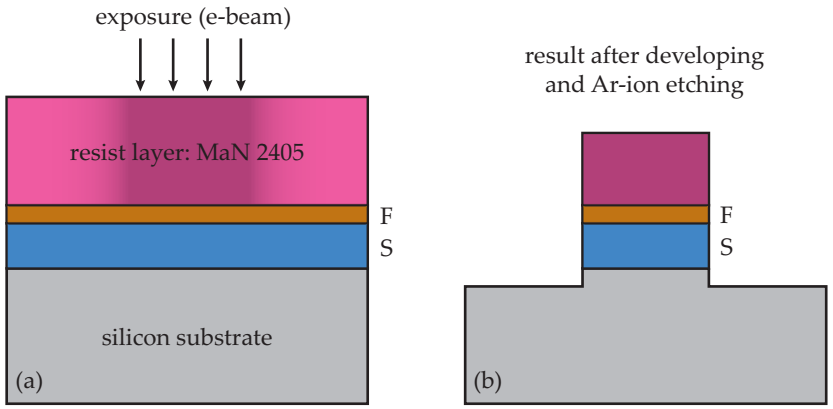

Figure 3.1: Sketch of the side-view of the substrate after (a) spin coating the MaN 2405 resist, and (b) etching down the strip. In (a), the darker-colored resist areas will we sufficiently exposed by the electron beam, and after developing that is what remains.

resist pattern serves as a protective mask for the following Ar ion-etching (see Fig. 3.1) which is performed at 2.5 μbar Ar pressure in a background pressure of 10^{-6} mbar. Etch times were ranging from 5 to 15 minutes depending on the specific layer thicknesses and to prevent burning and hardening of the resist, the sample was continuously cooled with Nitrogen gas (N_2). Additionally, the sample normal makes a small angle with the incoming ions and the sample is rotating in plane at a speed of order 10 Hz. In this way, the unwanted re-

deposition (of the sputtered material) at the sides of the strip is continuously etched away. Finally, the remaining part of the resist mask was cleaned-off using boiling acetone (at 70-75 ^oC).

In the last step, the contacts are lithographically defined using a lift-off geometry after which the contacting material is sputtered. The used lift-off geometry consist of a double positive-tone resist layer, where the in-plane development of the bottom resist expands further then the top layer. A PMGI/PMMA double resists layer is spin coated. First the PMGI at 4000 RMP (during 60 seconds) followed by a post-bake of 90 minutes at 200 ^oC, next the PMMA at 4000 RMP (during 60 seconds) followed by a post-bake of 30 minutes at 160 ^oC. Contacting pads and leads towards the strips were pat-

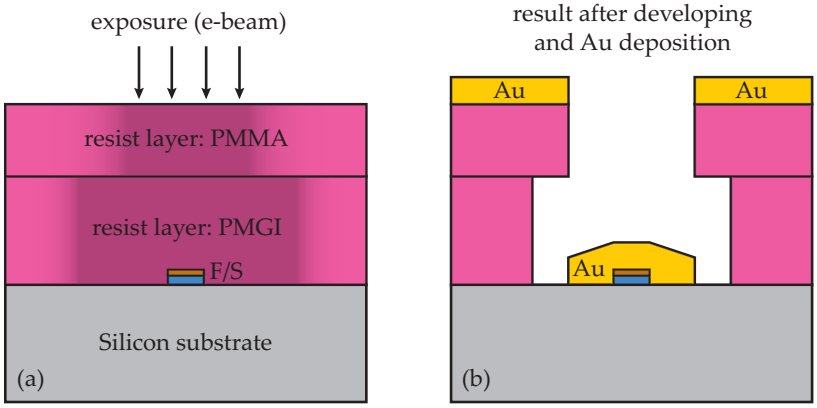

Figure 3.2: Sketch of the side-view of the substrate after (a) spin coating the PMGI/PMMA double resist layer, and (b) sputtering the contact material. In (a), the darker-colored resist areas will we sufficiently exposed by the electron beam, and after developing that is what dissolved. In (b), all the remaining resist (with Au on top) is dissolved using developer, completing the lift-off technique.

terned using the JEOL Microscope, where the alignment markers are used to align this new pattern with the strip. The used beam current was 10 nA and the dose 200 μC/cm^2. The development is now a two-step process. First the top PMMA layer is developed in diluted acetone for 35 seconds (diluted with demi-water, roughly at a 50/50 volume ratio). Second is the development of the PMGI layer in MF 322 for 5 minutes, where the last 2-3 minutes are used to create an undercut. A short cleaning in an Oxygen plasma is performed to remove some resist residues from the contacting areas on the strips (to lower

the interface resistance). Finally the Au contacts are sputtered (see Fig. 3.2) in a ultra high vacuum chamber with a background pressure of 10^{-7} mbar using an Ar plasma. Additionally, an initial few monolayers of Titanium (Ti) are sputtered *in situ* as adhesion layer for the Au. The Au contacts are aimed at a thickness of 100 nm and sputtered at an approximate rate of 0.17 nm/sec with an Ar pressure of 4 μbar. After sputtering the sample is immersed in NMP (N-methyl-2-pyrrolidone) to complete the lift-off by dissolving all resist.

3.2 Measurement setup specifics

The three main types of transport measurements on our samples are field dependent resistance measurements $R(H)$ at low temperatures, temperature dependent resistance measurements $R(T)$ and field dependent critical current measurements $I_c(H)$ (I_c is determined from measuring the current-voltage characteristics). These measurements are all performed using the same setup, which consist of a standard ^{4}He cryostat with a home-built insert which is connected to the measurement electronics (see Fig. 3.3). The cryostat is equipped with magnetic shielding to provide a low-noise environment and a superconducting coil (NbTi wire) to provide the magnetic field (up to 1 T). The cryostat insert is a closed type, where the sample chamber is shielded from the Helium bath inside the cryostat. The inner sample space is continuously pumped during measurements and reaches a pressure around 0.1 mbar. An Agilent DC power supply was used for the coil magnet. It provided a smallest current step of 1 mA, which corresponds to an induced field of about 0.1 mT. A heater (Cu wire) is used to increase the sample temperature which, for the given pressure, can go up to at least 10 K. The temperature itself is measured using a carbon glass resistor which has a low temperature sensitivity of about 200 Ω/mK. The desired (low) temperature is PID-regulated by an isolated GPIB circuit to increase its response time, resulting in a good temperature stability with fluctuations of 0.1-0.3 mK at the sample, in the range 4.2 K to 10 K. A Keithley K2400 was used for the heater current source. To measure the resistance of the thermometer a Keithley K220 current source and a Keithley K181 nanovoltmeter were used. By pumping the Helium bath directly the temperature can be lowered down to 1.7-1.8 K, which is necessary for the $I_c(H)$ measurements. All the $R(H)$ and $R(T)$ measurements are performed in the range 5-10 K, as the superconducting transition temperature for our samples is between 5 K and 7 K.

All measurements are current biased and for the $R(H)$ and $R(T)$ measurements we perform bipolar measurements (measuring the voltage difference

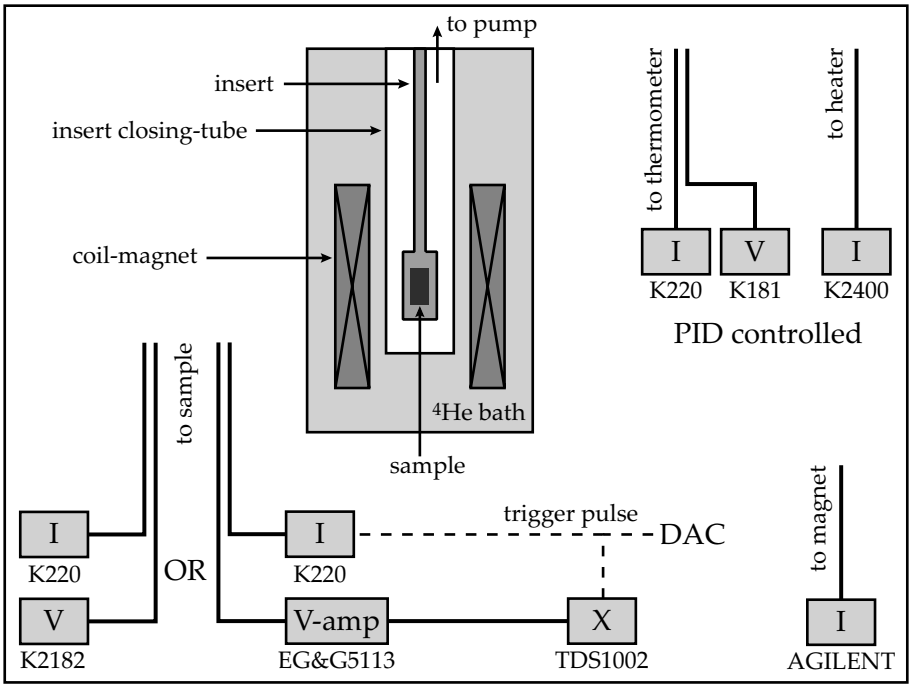

Figure 3.3: Schematic of the measurement setup.

for both positive and negative current and taking the average), with a current density of the order 2.5 $\times 10^8$ A/m^2. The sample is measured using a Keithley K2182 nanovolt meter and Keithley K224 programmable current source. For the critical current measurements we make use of a pulsed current technique. By applying short current pulses, Joule heating of the sample is minimized and the critical current can be determined much more accurately. We used 3 ms current pulses, with an interval of several seconds between the pulses and an increasing amplitude until the critical current is reached and the superconductor is driven into the normal state. The samples are initially cooled down in zero field condition and the first measurement at a fixed temperature always starts in zero applied field. The current pulse generates a voltage pulse across the voltage probes of the sample, which is amplified and measured with an oscilloscope. An external trigger pulse is used to synchronize the current pulse with the measurement time-window of the oscilloscope to catch the voltage pulse. The voltage amplifier is an EG&G 5113 differential amplifier with incorporated band filters and the oscilloscope is a digital Tektronix TDS1002. The external trigger is generated by a standard DAC-interface and all equipment is GPIB controlled.