Composition Characterisation of SQUID Chips with SEM and EDX

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1. Motivation - The Role of SEM and EDX in Industry

- **S**canning **E**lectron **M**icroscopy (SEM) allows for detailed view of micrometre structures (figures 2 (a) and 3 (a)).
- Can adjust many free parameters to obtain crisp images at high magnification.
- **E**nergy-**D**ispersive **X**-ray (EDX) spectroscopy allows for identification of materials using Mosely's law: $\nu_{X-ray} \propto Z^2$ (figures 2 (b) and 3 (b)).
- One can reverse engineer technology by combining SEM and EDX data.
- Crucial for Technology Push research and driving the rapid development of technology in industry.
- This is demonstrated here on an example SQUID chip.

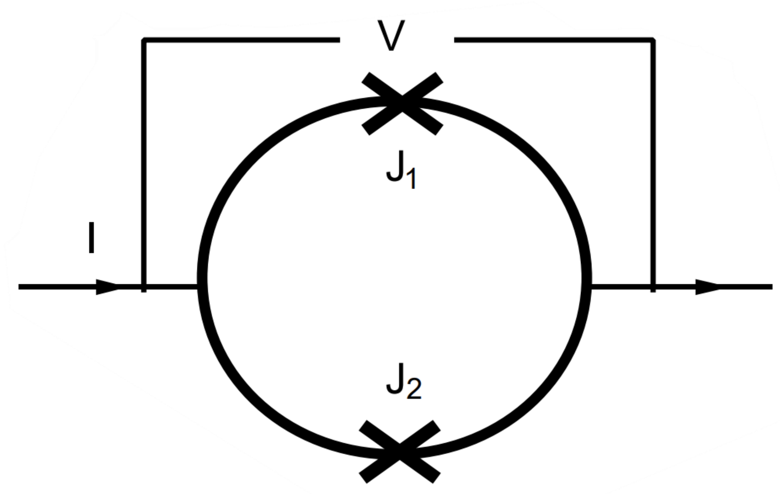
2. What are SQUIDs?

A SQUID is a **S**uperconducting **Q**uantum **I**nterference **D**evice that can measure extremely weak magnetic fields. They can:

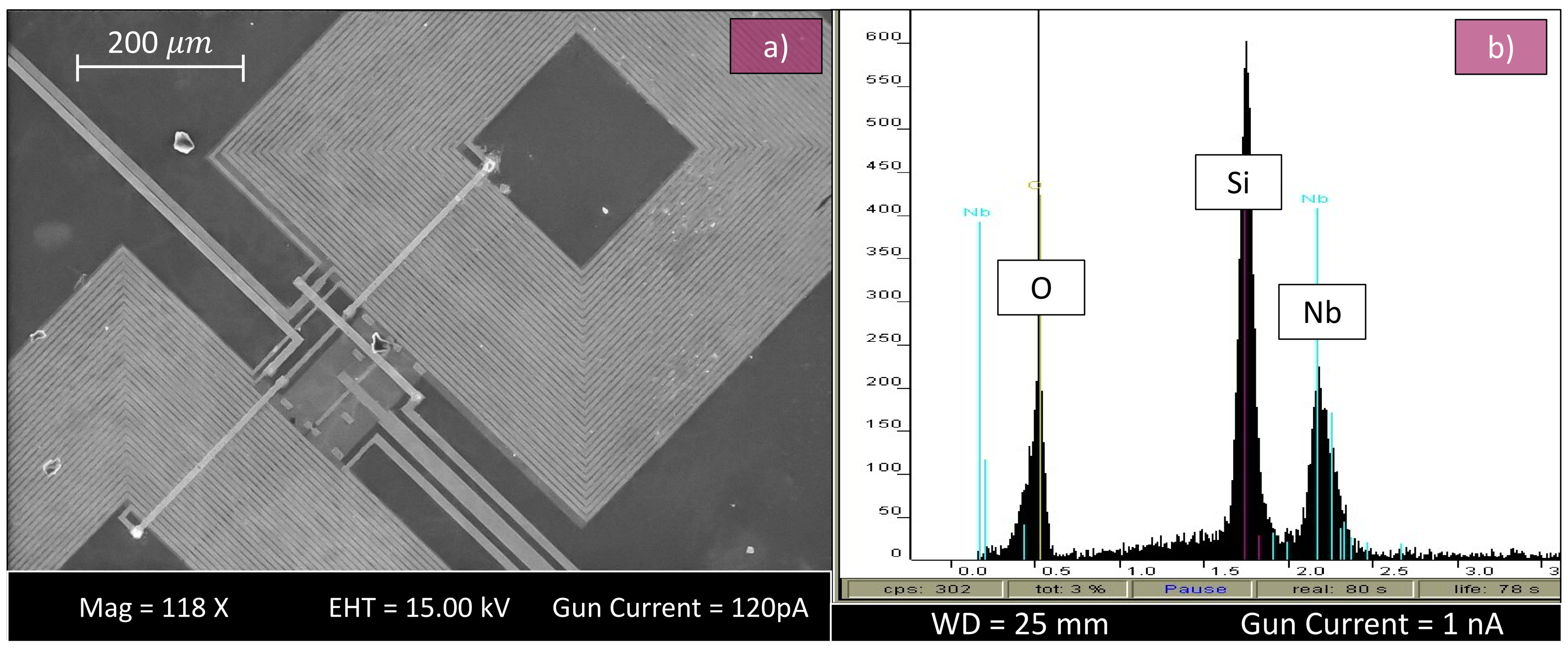
- Monitor neural activity of the human brain by detecting weak magnetic fields from neural currents.
- Detect magnetic fields of the heart in cardiology.
- Equip AFM probes with a SQUID to examine surface roughness[1].
- Perform MRI scanning with micro-tesla fields[2].

3. The Physics behind a DC SQUID

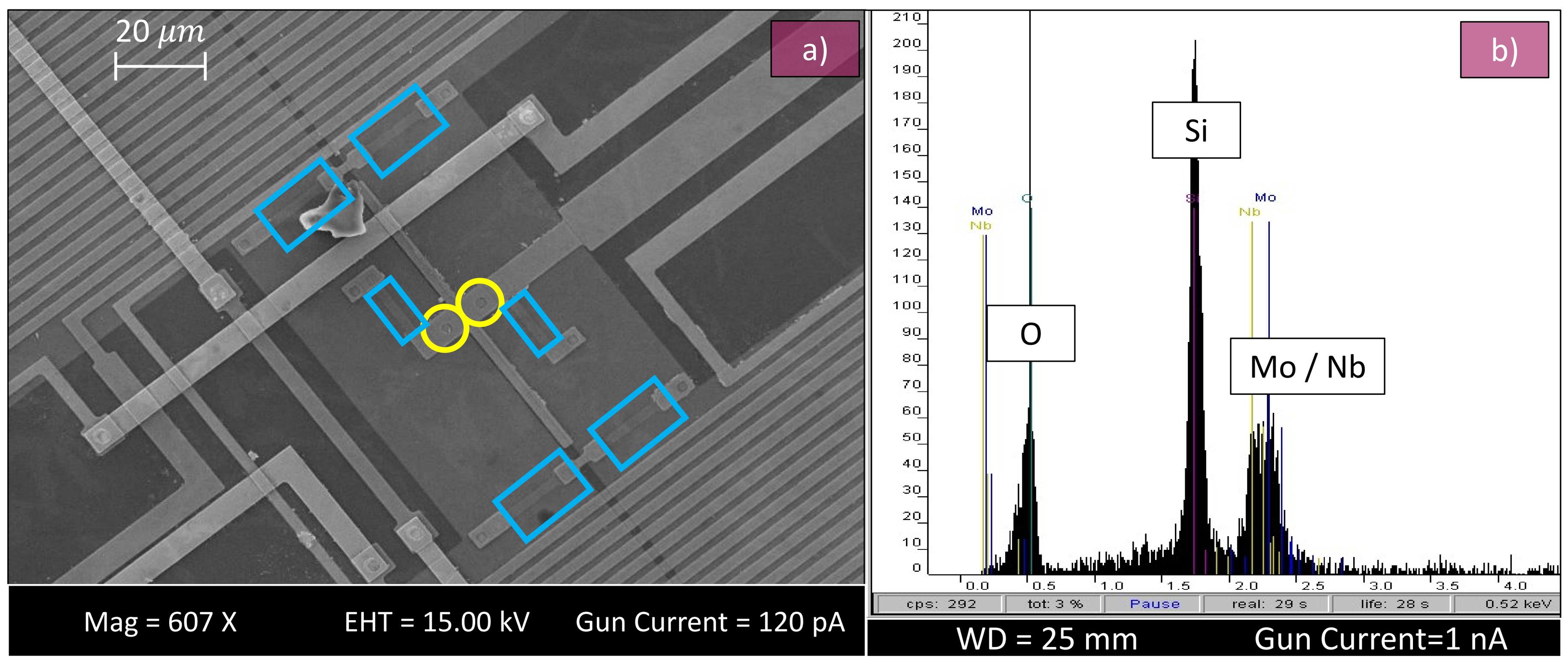
- Direct-current SQUIDs utilise the Josephson effect.
- Josephson effect occurs when two superconductors are in proximity: currents can flow between the two with zero resistance through quantum tunneling[3].
- The zero-resistance current is called the Josephson current.
- Two Josephson junctions lie parallel in a superconducting loop (labelled J_1 and J_2 in figure 1)
- Current splits evenly in the absence of a magnetic field.
- Small magnetic field generates a screening current and hence magnetic field which opposes the flux of the environment.



[Figure 1: Schematic of a DC SQUID, with two Josephson junctions in parallel to one another, with an input bias current. V represents the voltage response to the flux threading the superconducting coil.]



[Figure 2: (a) Central SEM image of SQUID chip illustrating two Josephson junction in parallel with two superconducting coils. Superconducting metal contacts enter at the centre, connecting to the coils. Figure 2(b) illustrates the corresponding EDX spectrum identifying the presence of niobium (the superconductor) and silicon and oxygen (from the silicon base and SiO_2 insulation).]

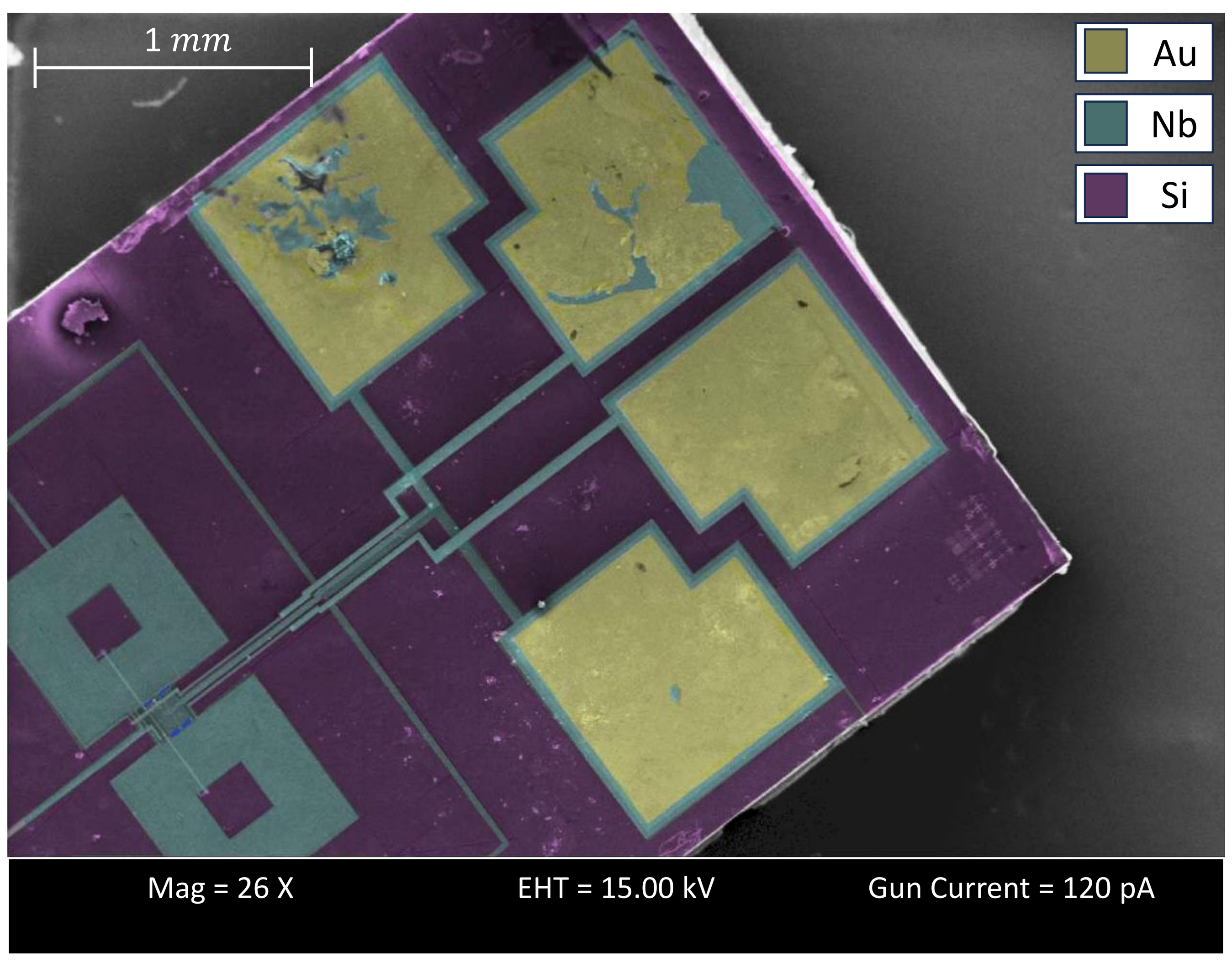


[Figure 3: (a) Enlarged view of centre of SQUID chip with Josephson junctions circled and thin resistors highlighted in rectangles. (b) EDX demonstrates precision accuracy in identifying molybdenum as the material of the thin layered resistors. Further detection of Si, O and Nb as expected.]

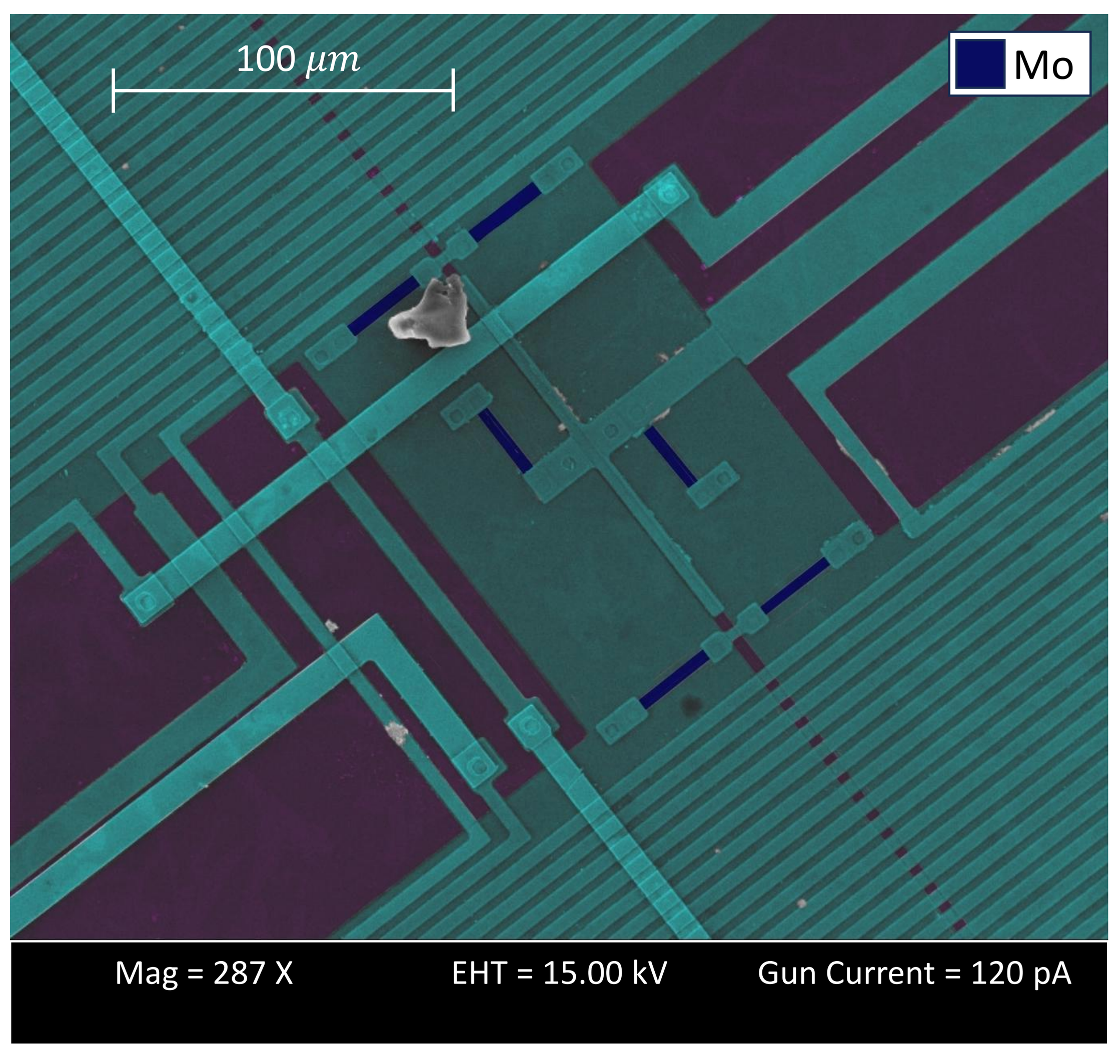
4. Imaging Method

- Distance between SQUID chip and electron gun was reduced for improved quality at high magnification.
- Work distance was reduced below 10 mm at high magnification and increased above 30 mm to achieve single digit magnifications.
- To achieve greater image brightness, filament current was set to 2.6 A at low magnification, and 2.9 A at large magnifications.
- Contrast was varied on a frame-by-frame basis and ranged between 10 – 20%.
- Brightness was adjusted with contrast and ranged between 50 – 60%.
- Frame averaging was increased to reduce noise in final images. This averaging increased for higher magnifications, and typically exceeded 10 frames totalling 5 minutes of averaging.

5. Results – Combining SEM and EDX Data with Colour



[Figure 4: Combination of SEM image of a SQUID on a large scale with EDX data for colour mapping of composition. Some anomalous white contrast due to charging at the border.]



[Figure 5: Central magnification illustrating molybdenum resistors and the two Josephson junctions, bridging the middle gap between the two superconductors, that cause the Josephson effect.]

Figure 4 illustrates gold contact pads on the outskirts of the silicon substrate, with niobium (superconducting at 9.2 K) forming the metal connections. Three layers of niobium have been identified, connected by vias of $\sim$ 3.6 μm diameter. Many vias are used for interconnection of layers at the centre as seen in figure 5. Superconducting coils are formed of niobium turns, with each line $\sim$ 4 μm in width. Silicon dioxide has been detected as an insulator material between metal layers and is hence not visible in either figures 4 or 5. Thin, molybdenum resistors (3.8 μm x 22 μm) can provide respectably high resistance in a compact space. The separation between the superconducting niobium in between the Josephson junctions was found to be $\sim$ 3.6 μm, that facilitates the Josephson effect. This brief characterisation demonstrates the use of SEM and EDX, from understanding how a theoretical concept can be engineered, identifying issues in defective ICs, to reverse engineering designs in a competitive and fast-paced industry.

6. References

[1] Suderow MH, Curtois MH, Hasselbach MK. Microscopie à micro-SQUID: étude de la coexistence de la supraconductivité et du ferromagnétisme dans le composé UCoGe

[2] Clarke J, Hatridge M, Mößle M. SQUID-Detected Magnetic Resonance Imaging in Microtesla Fields

[3] Anderson PW, Rowell JM. Probable Observation of the Josephson Superconducting Tunneling Effect. Phys Rev Lett. 1963 Mar;10:230-2