Nanofabrication of superconducting and ferromagnetic structures for operation in TEM

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Abstract

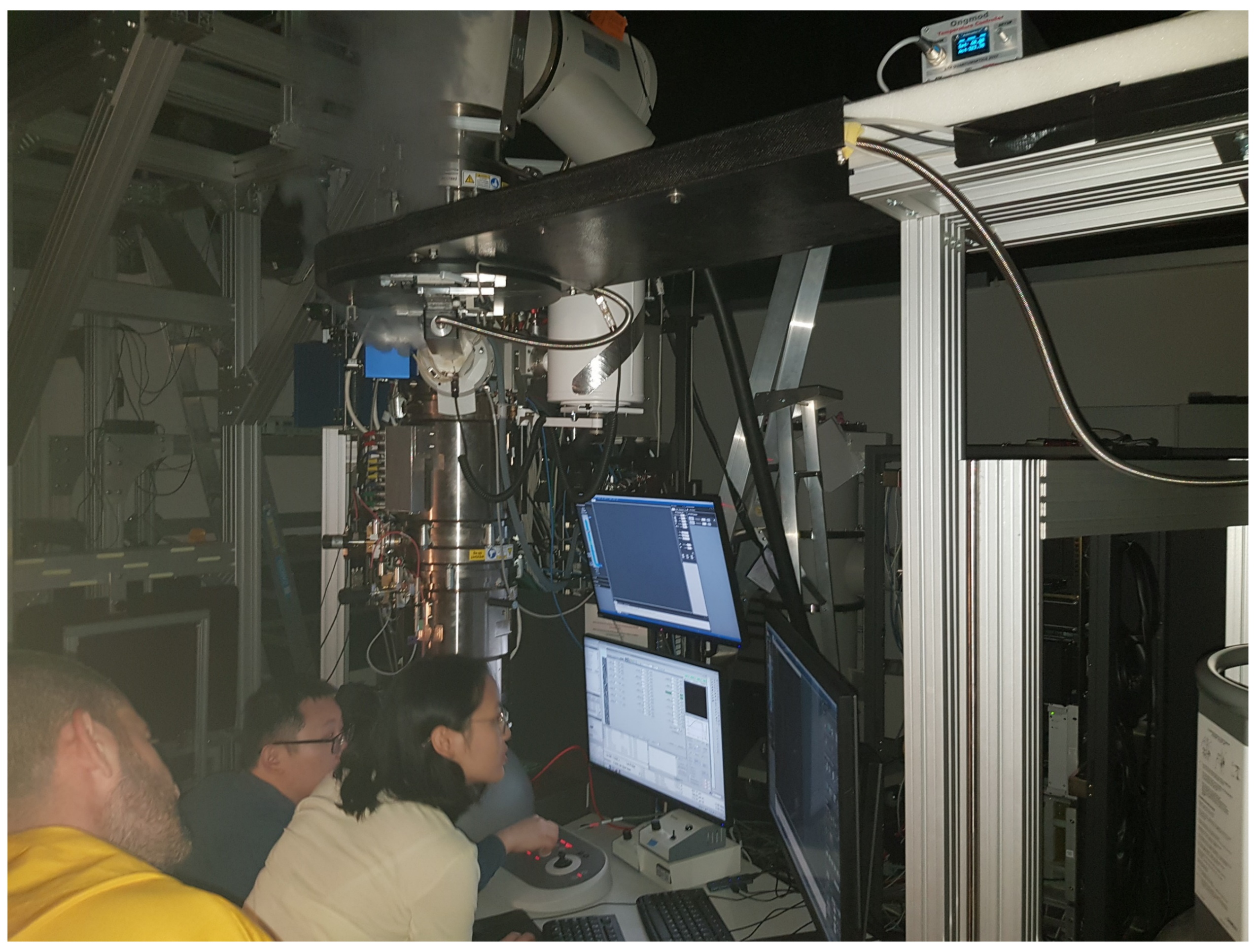
Nanoscale superconducting (Nb, TiN, NbN and BSCCO) and ferromagnetic Permalloy (Py) structures were prepared on SiN, SiO_2 and SiC membranes for experiments in a TEM. Exfoliated e-beam transparent BSCCO flakes were fixed on 4-contact chips to study correlations between temperature, electron transport properties and behavior of Abrikosov vortices. Metals Ti and Nb or nitrides TiN and NbN were combined into 3-layer heterostructures for adjusting superconducting parameters through the proximity effect. An on-chip thermometer was used also as a heater, allowing the sample temperature to be changed in the range from 400 K to 5 K and back in a few seconds. NanoSQUIDs with nanobridge Josephson junctions (nJJs) were prepared at a distance of below 200 nm from the tip of a cantilever by using bulk nanosculpturing of the substrate with a focused ion beam. The nanoSQUIDs had a sub-micrometer loop size, which limited the dimensions of the nJJs to below ~100 nm. Electron beam lithography and high selectivity reactive ion etching with pure SF_6 gas were used to pattern nJJs with a width down to 10 nm that is comparable to the coherence length in thin films of Nb and NbN and provides better reproducibility in the case of a Nb functional layer and better long term stability due to enhanced corrosion resistance in the case of NbN layer. A naturally created undercut in the Si substrate was used to prepare nanoSQUIDs on a 10-nm-thick SiO_2 membrane within 500 nm from the edge of the substrate. High-resolution TEM revealed that NbN films on SiN have a columnar structure while they observe cube-on-cube epitaxial growth on SiC membranes. Towards future realization of hybrid superconductor-ferromagnetic nanostructures for spintronics experiments in TEM, Py nanodisks and triangles with dimensions down to ~100 nm were prepared on SiN membranes and studied by Lorentz microscopy (LTEM) and electron holography TEM methods.

Materials

	Superconducting transition T_c, K	Coherence length ξ_0, nm	Penetration depth λ_0, nm
TiN	5	100	500
$NbSe_2$	7	15	496
Nb	9	10	40
NbN	15	5	300
$YBa_2Cu_3O_{7-x}$	93	1	150
$Bi_2Sr_2Ca_2Cu_3O_{10}$	110	1	270

- Ferromagnetic films of Permalloy (Py) with Curie temperature $T_c \approx$ 500 K.

Using TEM with L-He^4 cooled sample



Working with liquid helium cooled TEM sample holder of the company "condenZero" [4] at ER-C-1 FZJ. The reached temperature of 4.4 K can be maintained for more than 24 hours.

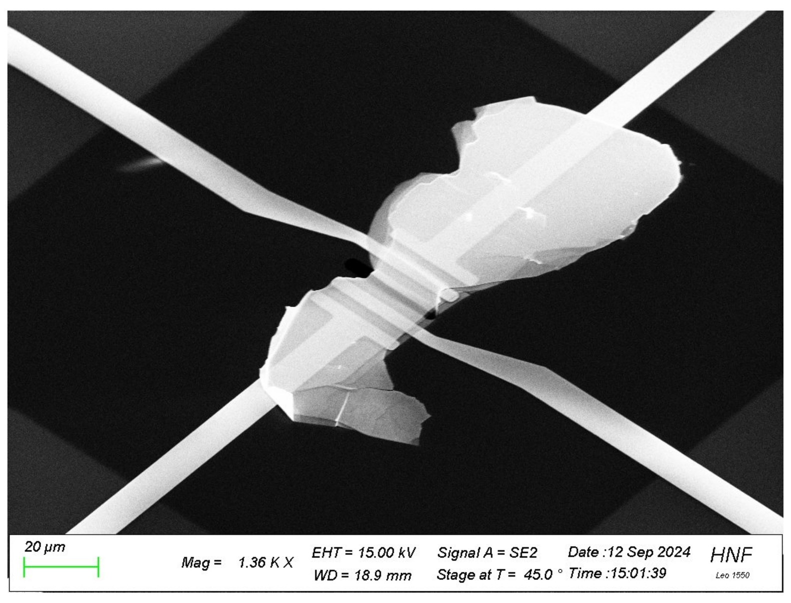
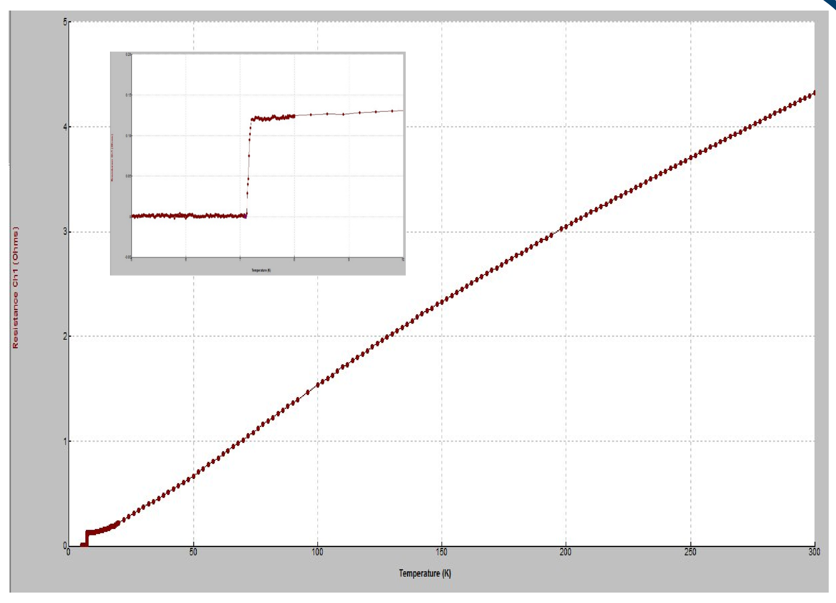
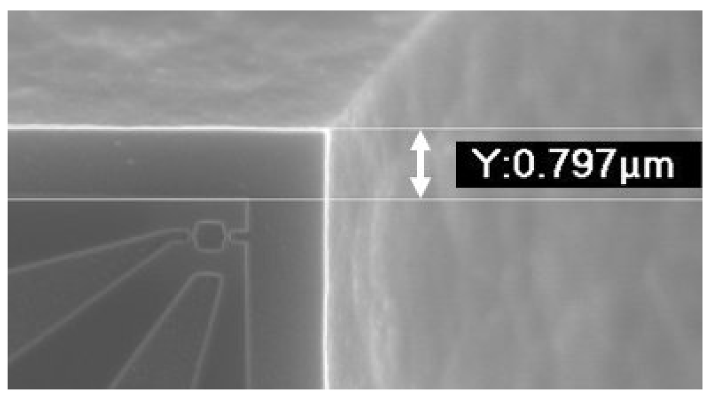


Photo of $NbSe_2$ flake with 4 contact leads on a SiN membrane.

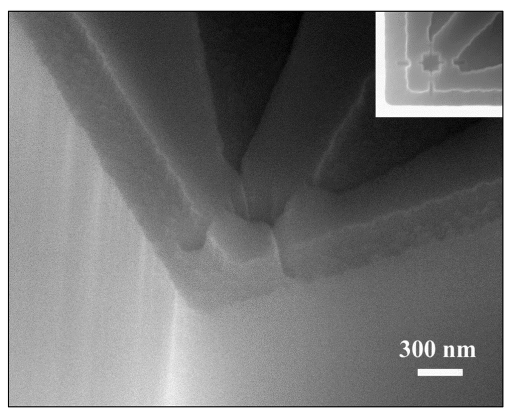


R(T) of $NbSe_2$ flake on a SiN membrane.
B_{c2} (5K) = 1.5 T → ξ_0 (flake) ~ 15 nm. λ_0(bulk) ~ 500 nm.

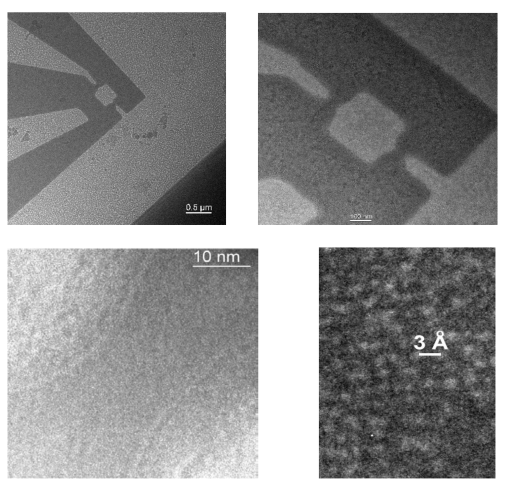
NanoSQUIDs on cantilevers and membranes



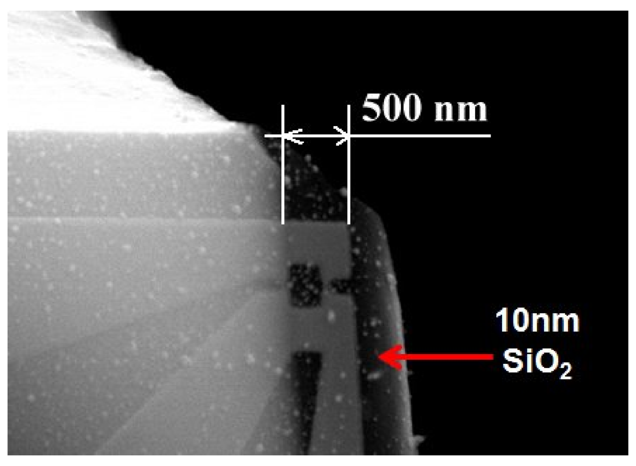
SEM image of the corner of a Si chip after ~1 hour of RIE in pure SF_6 gas at an RF power of 25 W [1].



HRSEM image of a Si cantilever with a 4JJ nanoSQUID placed within 300 nm of the corner by FIB etching. The inset shows a top view of the same nanoSQUID cantilever [2].



Plan-view TEM images of a nanoSQUID fabricated on a SiN membrane: (a) low-magnification TEM image showing position of nanoSQUID relative to the edge of membrane; (b) a higher magnification TEM image showing microcrystalline structure of the membrane and a polycrystalline structure of the Ti-Nb-Ti heterostructure film; (c) a higher magnification TEM image of one of the nJJs demonstrating width of the nJJ and HSQ resist; and (d) high resolution TEM image of Ti-Nb-Ti heterostructure in nJJ in nanoSQUID showing the ~0.3 nm interatomic distance corresponding to the lattice constant of Ti and Nb..



SEM image of a nanoSQUID placed within a distance of 500 nm from the corner of a Si cantilever chip on a 10-nm-thick SiO_2 membrane [1].

Spin resolutions

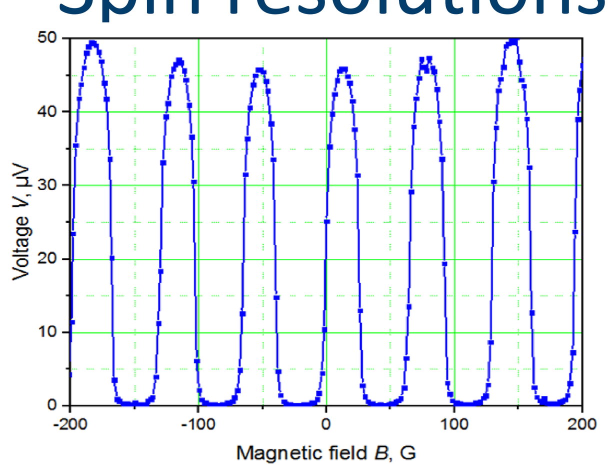
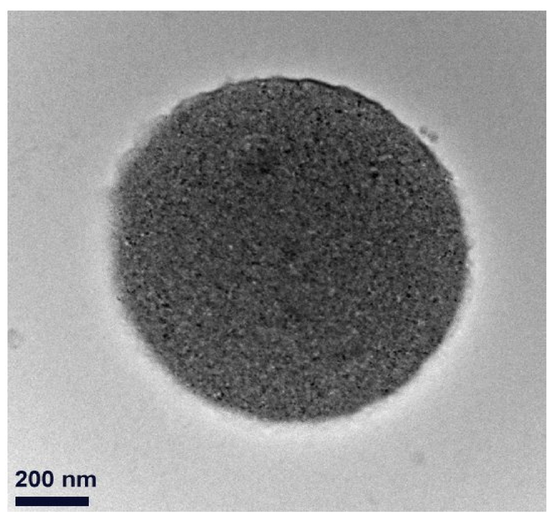


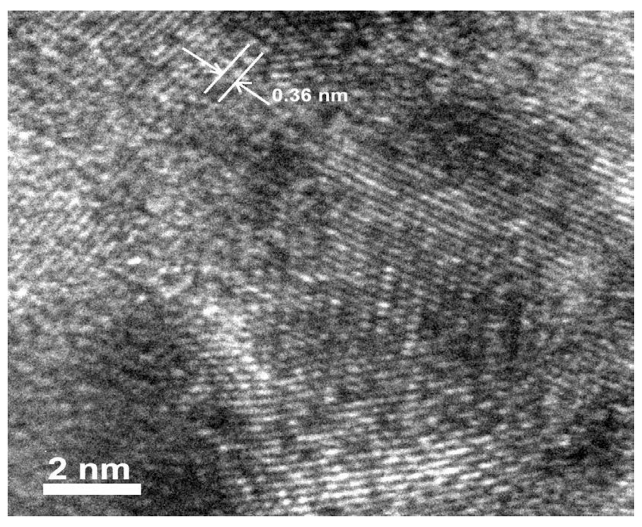
Fig. 11. *V(B)*-dependence of the planar nanoSQUID with NbN-TiN-NbN nJJs measured with a bias current of 39 µA at 4.2 K [2].

The noise level of the nanoSQUID-based measurement system is determined mainly by the white noise input level $\sqrt{S_V}$~1 nV/√Hz of the preamplifier at room temperature. The magnetic flux resolution of the present measurement system $\sqrt{S_\Phi} \cong \sqrt{S_V}/(\partial V/\partial \Phi)$ ~1.9 $\mu\Phi_0$/√Hz, where the derivative $\partial V/\partial \Phi \cong$ 525 µV/Φ_0. The magnetic field resolution $B_n = (\partial B/\partial F)\sqrt{S_F} \cong$ 12 nT/√Hz and the spin resolution $\sqrt{S_n} = (r/r_e)\sqrt{S_\Phi} \cong$ 417 μ_B/√Hz, where r_e = 2.82 × 10^{-15} m is the classical electron radius. Using a SQUID array preamplifier and a nanoSQUID of smaller size can improve the spin resolution to ~ 1 μ_B/√Hz.

Py nanodots on SiN membranes

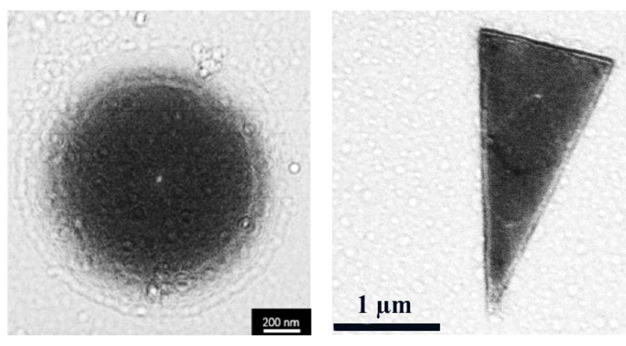


TEM image of a 60 nm thick Py disk with a diameter of ~900 nm manufactured using lift-off with a bilayer resist [2].

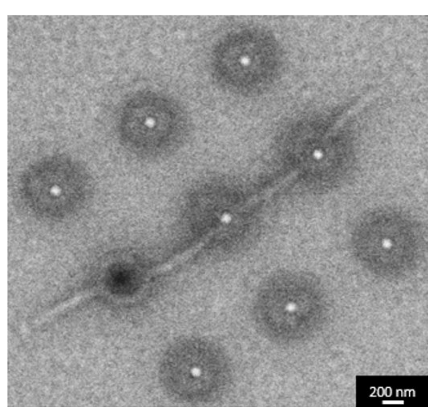


High-resolution TEM image of a small part of one of the Py nanodisks [2].

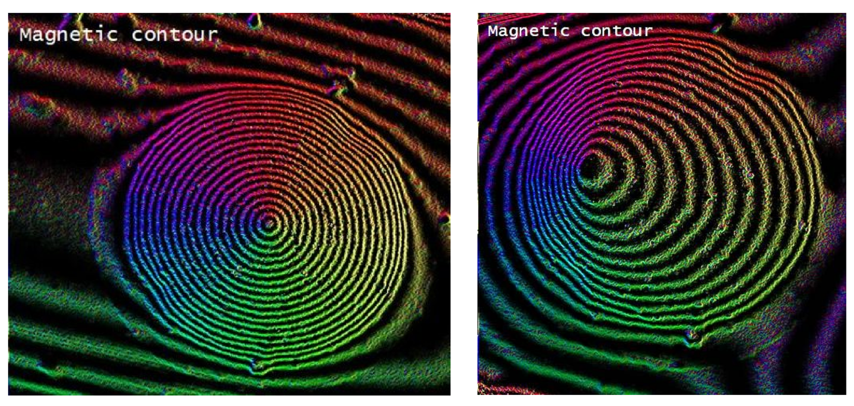
LTEM and electron holography of Py nanodisks



LTEM image of a 1 µm Py disk [6] and a Py triangle.



LTEM image of array of 400 nm Py nanodisks [6].



Quantitative magnetic imaging of the Py nanodisk. In-plane magnetic induction maps detected at room temperature using electron holography at different external in-plane magnetic fields: 0 mT (left) and 33,5 mT (right). The in-plane magnetic fields were obtained by variation of magnetic fields of the objective lens and 16° tilt of the sample. The contours outside the Py disks are mainly due to the presence of stray magnetic fields and the inhomogeneous electrostatic charge of the SiN membrane. [6]

References

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6. Williams J., Faley M.I., Vas J.V., Lu P.-H., Dunin-Borkowski R. E. *Beilstein Journal of Nanotechnology*, **15**, 1 (2024)