Nonorganic evaporation mask for superconducting nanodevices

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We describe a novel technique to produce submicron thin film structures of high melting superconducting materials (Nb). The method is based on a nonorganic evaporation mask (Si_3N_4) to avoide any outgassing of the mask material during the metal deposition which would deteriorate the superconducting properties of the Nb. The mask has a large offset from the substrate so that very clean interfaces of different materials (e.g. normal metal/ superconductor (NS)) can be achieved by angle evaporation in one single process step. By this means we have prepared narrow Nb wires with high transition temperature and NS structures with high quality interfaces.

Recently, many groups [4] have encountered the problem, that submicron niobium structures patterned with ordinary PMMA resist had a significantly suppressed transition temperature T_c compared to the T_c of a coevaporated niobium film. The most important reason is the outgassing of the PMMA during the evaporation of the high melting Nb due to radiation heating from the evaporation source. Most experiments which involve superconductors have therefore been performed with low melting materials (e.g. Al). However niobium based junctions are very interesting because of its larger superconducting gap (Δ) which allows to study a wider energy range of temperature and magnetic field dependent effects. Also for future technical applications, e.g. bolometers [1], Nb would be advantageous. The common method to fabricate junctions with well defined interfaces is the use of angle evaporation through a suspended mask [2]. By varying the deposition angle one can fabricate very clean interfaces with small overlap or wires of very narrow linewidth. The commonly used method for shadow evaporation is the lift off technique. Two different photoresists with different sensitivity provide high resolution pattern with a good undercut profile. The double layer resist of polymethylmethacrylate (PMMA) as mask and a co-polymer (PMMA-MA) as support has shown to be successful for low melting materials [3]. For high melting materials more complicated techniques were developed like a two layer metal mask which was differentialy etched [5] or a four layer resist system [4]. The first is not applicable for angle evaporation of submicron structures because of the rough edges of the mask. The latter still had the problem of deterioration of the superconducting properties. The T_c was suppressed up to 1/5 of its bulk value. Our investigation showns that not only the background pressure during evaporation but in particular the outgassing of the organic resist mask is the main source of impurities in the deposited material. This is most important for small devices where the whole structure is in the vicinity of the mask material.

We have therefore developed a nonorganic evaporation mask to avoid any outgassing of the stencil during evaporation. Figure 1 shows a chart of the process. It consists of a 800nm SiO_2 and a 200nm Si_3N_4 layer on a Si wafer. Both materials were deposited by Low Pressure Chemical Vapor Deposition (LPCVD). On the Si_3N_4 a 600nm PMMA layer is spun. The PMMA top layer is structured by conventional e-beam lithography (Jeol JSM-IC848). After development in 1:3 MibK:IPA the PMMA is used as an etchmask for the Si_3N_4. The pattern is transferred to the Si_3N_4 by a CHF_3 anisotropic Reactive Ion Etching (RIE). The etchratio of Si_3N_4 to PMMA is 1/3 at 0.03 mbar and 50W. At this step we have to accept a little widening of the structure because the width of the opening in the Si_3N_4 will correspond more to the bottom width of the PMMA undercut profile, because the ouverhanging PMMA is etched more rapidly in the plasma (see fig. 1). Now the SiO_2 support layer can be removed with a wet etchand. 15 min in buffered hydrofluoridacid (BHF) results in the freestanding Si_3N_4 with 1μm undercut.

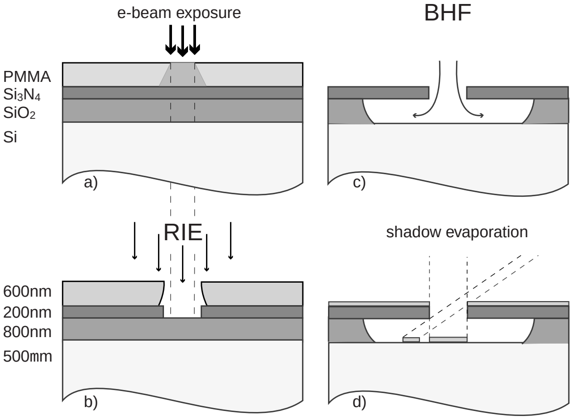

Figure 1. Process chart: a) e-beam lithography on a three layered wafer b) pattern transfer by RIE c) wet etching of the support material d) angle evaporation through the stencil

This technique allows a very controlled way to produce deep undercut profiles since only the topmost PMMA layer must be sufficiently exposed during e-beam lithography. The ordinary double layer technique with PMMA-MA relies on overexposure of the MA layer to achieve a large enough undercut. The Si_3N_4 stencil is mechanically strong enough that bridges of several micrometers can easily be realized. After evaporation a lift off is not possible. Under clean preparation conditions there is no contact between the device structure in the etched pit and the film on top of the stencil which is 800nm offset from the substrate. As a first test of the method we produced narrow (250-415nm) Nb wires to show that mesoscopic structures with good superconducting properties are feasible. The Nb wires of varying linewidth were prepared by depositing 50 nm of Nb with an electron beam gun (e-gun) at normal incidence and 5Å/s at 10^{-7} mbar background pressure (fig. 2). The material parameters for the wires are listed in Tab. 1.

width [nm]	R(10°K) [Ω]	ρ(10°K) [$\mu\Omega$cm]	mfp [nm]	T_c [K]
250	67	15	2.5	8.41
390	111	19	2.0	8.4
415	116	21	1.8	8.39

Table 1

Figure 2 shows a SEM micrograph of the Si_3N_4 mask. The undercut (white) is about 2μm. The resistive transitions of Nb wires together with a coevaporated two dimensional (2D) Nb film is shown in figure 3 . The transitions of the wires are very sharp and very close to that of the 2D film. The inset in fig 3 compares the dependence of T_c on wire width for both the Si_3N_4 and the PMMA-MA resist techniques. While in the case of PMMA-MA T_c goes down to 2 K for the Si_3N_4 hardly any degradation can be seen.

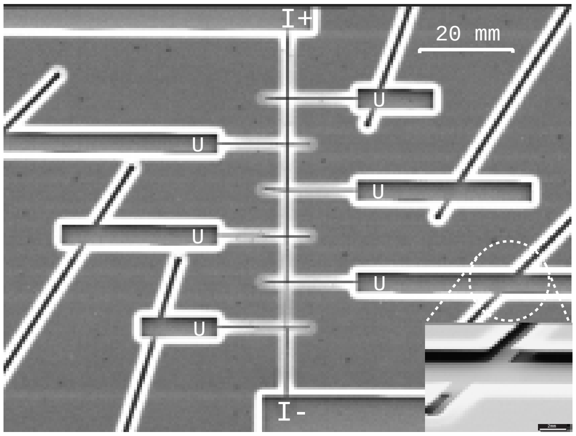

Figure 2. Series of Nb wires with current leads on top and bottom and voltage probes to the sides. The wires are 50 nm thick and from top to bottom 1μm to 250nm wide. The white areas are the free standing parts of the mask. Bottom right: a detail of the mask seen under a tilt angle.

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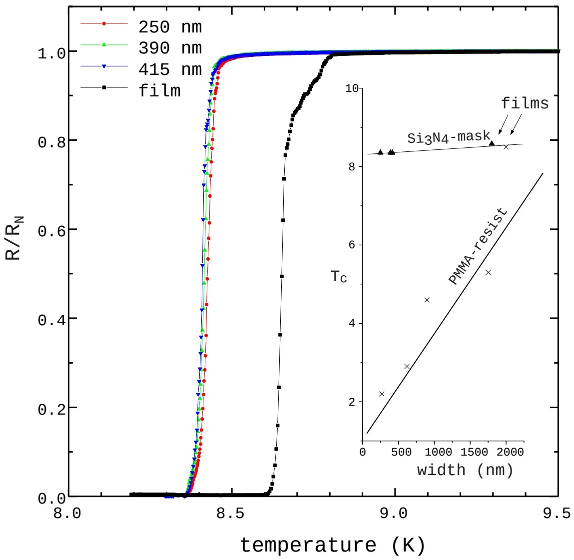

Figure 3. Normalized resistance as a function of temperature of Nb wires of varying width and a coevaporated film. The inset shows a strong decrease in T$_c$ of Nb wires fabricated with organic resist.

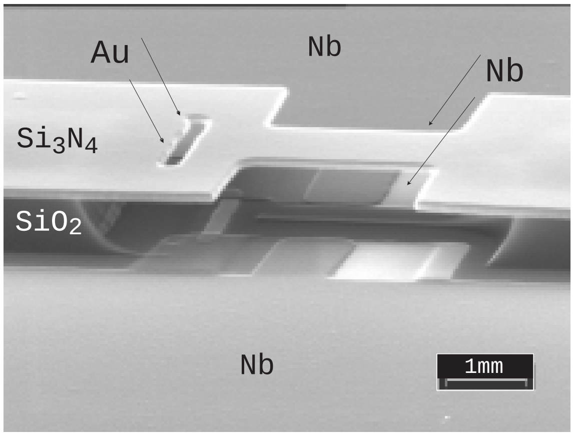

Figure 4. SNS-junction. Through the freestanding mask Au is deposited under 33° from the left and Nb under 33° from the right. The gold wire is on both sides connected to Nb reservoires

conductor/normal metal/superconductor (SNS) junctions we have prepared gold wires between superconducting Nb reservoirs. This was performed by angle evaporation where we have exploited the very large undercut and high offset from the substrate of the stencil. This allows a large horizontal shift of the shadow image of several micrometers. Fig. 4 shows the pattern in the Si$_3$N$_4$ mask and the device structure that results after evaporation under two different angles. The e-beam pattern is advantageous since the proximity effect is absent.

Under 33° tilt angle 15 nm gold were deposited at 5$\AA$/s and 10^{-6} mbar. Without breaking the vacuum 50nm Nb was evaporated at 5$\AA$/s and 10^{-7} mbar under -33° tilt angle (fig. 4). This has the advantage that we have very clean interfaces without oxidation or adsorbates at the interfaces. Our samples show a pronounced signature of Andreev reflection in the differential resistance at energies of the order of the gap energy and for wires shorter than 1μm even a supercurrent for low bias voltage . In figure 5 we have plotted the differential resistance dV/dI of a series of 12 Au wires between Nb reservoirs. At voltages slightly higher than the gapenergy of the Nb dV/dI drops considerably, which can be attributed to Andreev reflection at the NS interfaces. Since for Andreev reflection a high transmission is needed, this indicates the high quality of our interfaces. Around zero bias voltage a very sharp and pronounced dip develops in dV/dI which signals the formation of a proximity induced minigap [6] in the density of states of the normal conductor. This minigap is the precursor of a supercurrent, which develop in shorter devices when the length of the wires becomes comparable with the thermal diffusion length $L_T = \sqrt{\hbar D/k_B T}$.

In summary we have presented a novel structuring method with a nonorganic mask which is espacially suitible for high melting materials. The method is particularily useful for angle evaporation techniques because it allows very large undercuts and free standing masks. The new resist is ideally suited for the preparation of metallic Andreev intoferometers, which are under current investigation.

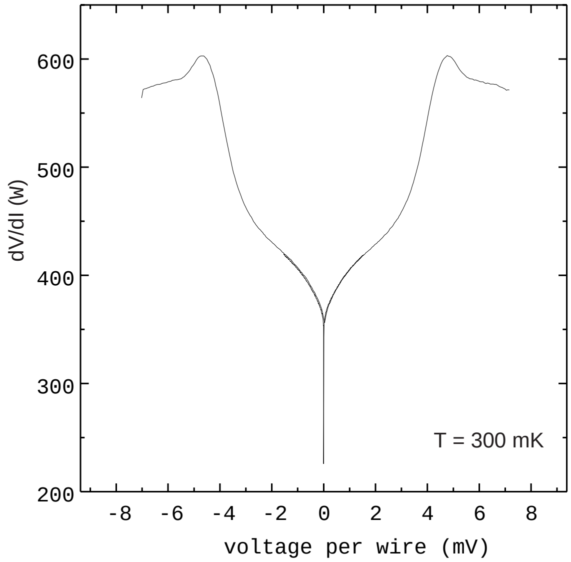

Figure 5. Differential resistance of a 1μm long goldwire connected to Nb reservoires. At the order of the gapenergie ($2\Delta = 2.6 mV$) a resistance drop occures due to Andreev-reflection. For low bias voltage the onset of a supercurrent can be seen.

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