RAPID COMMUNICATION

Fabrication of superconducting NbN meander nanowires by nano-imprint lithography*

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Superconducting nanowire single photon detector (SNSPD), as a new type of superconducting single photon detector (SPD), has a broad application prospect in quantum communication and other fields. In order to prepare SNSPD with high performance, it is necessary to fabricate a large area of uniform meander nanowires, which is the core of the SNSPD. In this paper, we demonstrate a process of patterning ultra-thin NbN films into meander-type nanowires by using the nano-imprint technology. In this process, a combination of hot embossing nano-imprint lithography (HE-NIL) and ultraviolet nano-imprint lithography (UV-NIL) is used to transfer the meander nanowire structure from the NIL Si hard mold to the NbN film. We have successfully obtained a NbN nanowire device with uniform line width. The critical temperature (T_c) of the superconducting NbN meander nanowires is about 5 K and the critical current (I_c) is about 3.5 μA at 2.5 K.

Keywords: nano-imprint lithography, meander nanowires, ultra-thin NbN films

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1. Introduction

The superconducting nanowire single photon detector (SNSPD) has attracted extensive attention since 2001 when Gol'tsman *et al.* showed first that the superconducting NbN nanowire made from ultra-thin NbN film could achieve single photon detection.[1,2] In most reported works on SNSPD, the devices were fabricated by using electron beam lithography (EBL),[3] although some other techniques such as focused ion beam (FIB)[4] and atomic force microscope (AFM)[5,6] were employed too. However, the EBL process requires expensive equipment and cannot meet the low cost and high efficiency requirement for mass production. Nano-imprint lithography (NIL)[7] is a high resolution, low cost, and high throughput lithographic technology. The mechanical force can copy the template with nano-structure to the imprint resist in equal proportion, and then the structure is transferred from the resist onto the substrate by reactive ion etching (RIE). In a previous study, we have demonstrated the applicability of NIL in fabricating Nb nanowires.[8,9] In this study, we focus on the fabrication of NbN superconducting meander nanowires for SNSPD by means of NIL. It is now widely recognized that NbN is more suitable for SNSPD than Nb due to the high T_c and high light absorbancy. In this study, a combination of hot embossing nanoimprint lithography (HE-NIL) and ultraviolet nanoimprint lithography (UV-NIL) is used to transfer the meander nanowire structure from the NIL hard mold to a NbN film. The nano-structure is characterized by scanning electron microscopy (SEM). The resistance–temperature (R–T) and current–voltage (I–V) characteristics of the NbN nanowires are measured by a physical property measurement system (PPMS). The results show that the NbN meander nanowires can keep their good performance of superconductivity.

2. Fabrication process

In order to obtain high performance SNSPD,[10] ultra-thin NbN films[11] with good superconducting properties are of great importance. In this work, the NbN films were deposited on 10 mm×10 mm SiO_2/Si (100) substrates by direct current (DC) reactive magnetron sputtering[12] at room temperature in the mixed gas of Ar and N_2. The Nb target had a purity ≥ 99.95%. In order to obtain good quality NbN films, the base pressure of the deposition chamber was maintained at lower than 5×10^{-7} Pa before sputtering. Then high purity N_2 gas flowed into the chamber to a required pressure. After this, high purity Ar gas (99.999%) was supplied until the required total pressure was reached. In order to prevent active gas contaminations, the Ar gas was supplied through a customized built-in non-evaporable getter (NEG) purifier before flowing into the sputtering chamber. During sputtering, the power was kept at around 220 W while the total pressure in the chamber was maintained at 1 Pa. The sputtering power was chosen in order to maintain a slow deposition rate so that the thickness of the ultrathin film could be controlled. In order to further optimize the superconducting properties of the

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films, the NbN films were prepared under different nitrogen partial pressures P_{N_2}.[13,14] In the best case, 4 nm thick films with a critical temperature (T_c) of 7.8 K were obtained when the sputtering pressure was 1 Pa and P_{N_2} was 0.16 Pa.

Figure 1 shows the AFM image of a 100 nm thick NbN film. The surface root mean square (RMS) roughness is 1.2 nm. The figure shows that the NbN film is uniform, dense, and smooth. We also employed the x-ray reflectivity (XRR) measurement to characterize the ultrathin NbN films. Data obtained on one sample are shown in Fig. 2. The experimental data of the NbN film were fitted, giving NbN film thickness 3.8 nm with about 0.5 nm surface roughness and a 1.4 nm thick Nb_2O_5 oxide surface layer.

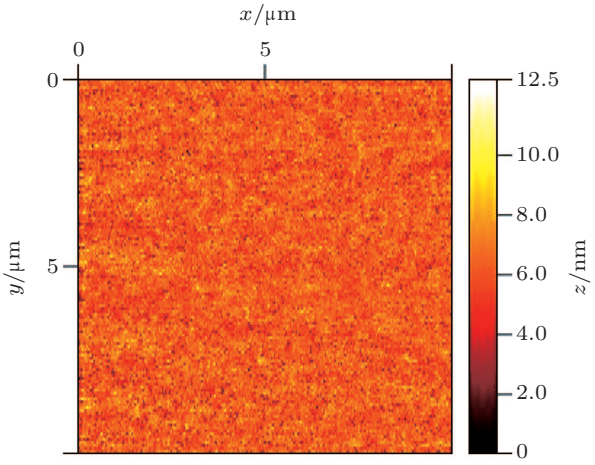


Fig. 1. (color online) AFM image of NbN film.

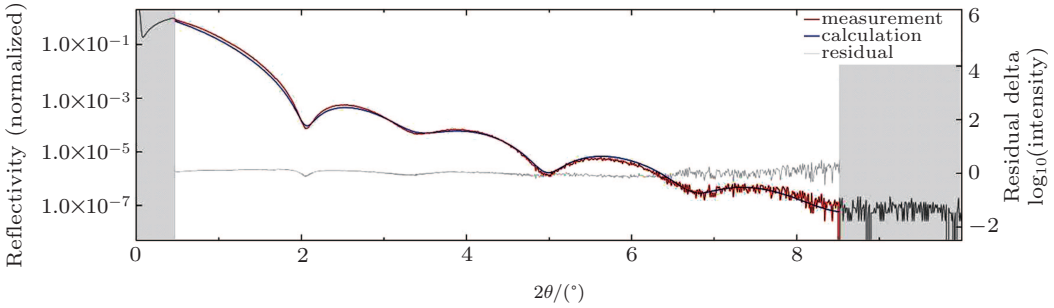


Fig. 2. (color online) X-ray reflectivity measurement data of NbN film.

NIL was used to define the meander nanowire on NbN films. The process is similar to that reported previously.[8] In order to protect the expensive hard mold and prolong the working life, the nanostructure was prepared by soft film transfer technology, which avoided direct contact between the expensive hard mold and the hard substrate. The soft film transfer technology is a method that copies the structure of the hard mold onto a soft material and then imprints with the soft material as the mold. The process of the preparation of NbN nanowire structures by NIL is as follows. At first, the hard mold was prepared on Si by using the EBL method. Then the IPS soft mold was defined using the hard mold and HE-NIL, and the structure of the soft mold was transferred onto NbN thin films coated with UV curable resists by UV-NIL. Finally, the structure on the UV curable resist was transferred to the NbN film by RIE.

For the preparation of the hard mold, EBL, UV lithography, and RIE were used to prepare the nanowire structure on a Si substrate. At first, the mender type nanowire structure was defined on Si by using direct electron-beam-lithography and RIE. As shown schematically in Fig. 3, the white area was etched away and the etching depth was about 50 nm. The nanowire structure area was protected with photoresist by ultraviolet exposure, other parts of the Si substrate were etched about 50 nm in depth by RIE. The schematic diagram of the nanoimprint template is shown in Fig. 4, in which the black part is the raised part, and the two cross marks are used for alignment in the ultraviolet exposure process. In order to avoid the residual resist on the hard mold affecting the replication of the nanowire structure, the hard mold needed to be cleaned using oxygen plasma, a microwave plasma system (PS210) was used to remove the residual resist on the surface of Si. The SEM image of the nanoimprint hard mold is shown in Fig. 5, which exhibits large area lines and relatively uniform nanowire graphics. The effective nanowire area is 10 μm×10 μm and the total length of the nanowire is 500 μm.

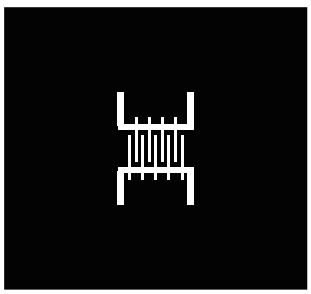


Fig. 3. Sketch of nanowire structure on Si by EBL and RIE.

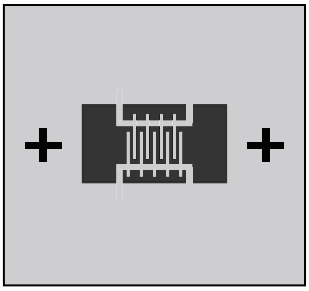


Fig. 4. Schematic diagram of the NIL hard mold.

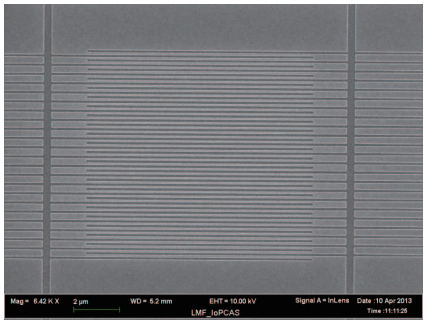


Fig. 5. SEM image of the nanowire on the nanoimprint hard mold.

HE-NIL and UV-NIL[15,16] were performed on an Eitre3 nano-imprinter system made by Swedish company Obducat. HE-NIL transferred the structure of the meander-type nanowire from the hard mold to an IPS® (intermediate polymer stamp) soft mold. IPS is a transparent polymeric material with a thickness of about 200 μm. It can be cut into different sizes according to the demand, and it can be used after peeling off the protective film on the surface. IPS has sufficient strength and toughness at room temperature, and at the temperature above 120 °C, it is in a highly elastic state. Because IPS contains fluoride, it is a good anti-adhesive material and can be easily peeled off at high temperature. In our fabrication, we cut IPS into pieces slightly larger than the size of the hard mold, then quickly and evenly placed an IPS on the hard mold. In order not to affect the graphic replication, it is important to avoid generating bubbles between the IPS and the hard mold. Figure 6 shows the sequential steps in the HE-NIL process. The hard mold and the IPS were sequentially placed on the substrate holder. After the holder was covered by a vacuum tight lid, air was pumped out. The program of HE-NIL was run. In Table 1, the parameters of HE-NIL are listed. The program began with heating the holder to 160 °C, followed by applying 40 bar pressurized air into the holder to press the IPS in tight contact with the hard mold. After 60 s, the temperature was lowered to 100 °C and held for another 20 s before cooling down to 60 °C and depressurizing. At this stage, the meander nanowire structure was transferred from the hard mold to the IPS. Peeling off the IPS from the mold, we obtained the soft mold.

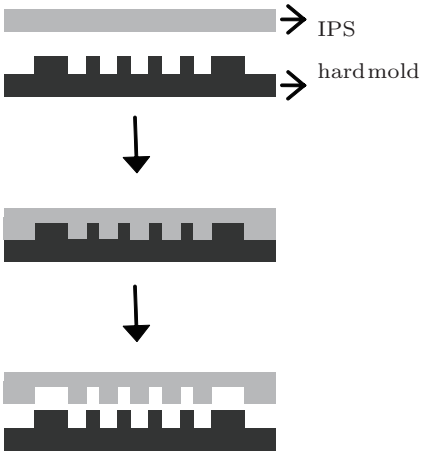


Fig. 6. Sequence of technological operations in HE-NIL process.

Table 1. The parameters of HE-NIL.

NIL type	Temperature/°C	Pressure/bar	Time/s
HE-NIL	160	40	60
	100	40	20
	60	0	0

In the process of UV-NIL, we transferred the structure of the meander-type nanowire from the IPS soft mold to the NbN film deposited on Si substrate. In this process, we chose ultraviolet curable resists TU2-60, which was spin-coated at the speed of 3000 round/min for 1 min and pre-baked at 95 °C for 3 min. The thickness of TU2-60 was about 60 nm. Due to the poor adhesion between the substrate and the UV curable resist, TU2-60 is very sensitive to the purity and humidity of the substrate surface. Therefore, it is essential to ultrasonically clean the substrate first and bake at 180 °C for 10 min. Figure 7 illustrates the schematic steps of the preparation of nanowire by UV-NIL process. The NbN film coated with TU2-60 was placed onto the substrate holder of the nano-imprinter system, and then the IPS soft mold was placed on the NbN film rapidly to ensure that the surface of IPS was not contaminated. The holder was covered by a lid with a sealing film providing a vacuum seal. After pumping out air, the program of UV-NIL was run. In Table 2, the parameters of UV-NIL are listed. Firstly, the substrate was heated to 80 °C, pressurized to 40 bar by high pressure gas and held for 240 s. Secondly, a parallel UV light was applied through the transparent IPS to cure the TU2 resist for 300 s at the same temperature and pressure. After switching off the UV light, the sample was maintained at the same temperature and pressure for 120 s. Finally, we peeled off the IPS soft mold after the sample was cooled down to 60 °C. At this point, the pattern with the meander-type nanowire structure was transferred from the IPS to the substrate coated with UV curable resist. Then RIE was performed in a mixture of Ar, CHF_3, and SF_6 gases. RIE removed the residual resist and unwanted NbN. After RIE, the NbN meander nanowire structure was formed on the SiO_2/Si substrate.

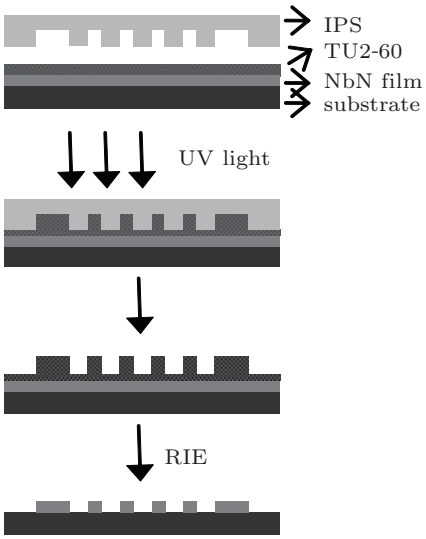


Fig. 7. Preparation of NbN meander nanowires by UV-NIL and RIE.

Table 2. The parameters of UV-NIL.

NIL type	Temperature/°C	Pressure/bar	Time/s	UV light
UV-NIL	80	40	240	off
	80	40	300	on
	80	40	120	off
	60	0	0	off

3. Measurements and results

The SEM image of the NbN meander nanowires fabricated by the NIL process is shown in Fig. 8. The meander-type nanowires are uniform in width. The width of the superconducting nanowire is about 80 nm and the filling factor is about 50%. Clearly, the NbN nanowire structure is copied from the hard mold.

The superconducting critical temperature of the NbN films sputtered at diverse nitrogen partial pressures (P_{N_2}) was determined through transport measurement in a PPMS system. The relation curve between T_c and P_{N_2} is plotted in Fig. 9 for samples of 50 nm thick and deposited under 1 Pa of total sputtering chamber pressure. It could be seen that T_c increases first and then decreases with the increase of P_{N_2}. The critical temperature of NbN reaches the maximum when the nitrogen partial pressure is about 0.16 Pa. Figure 10 shows the T_c dependence on the NbN film thickness. With the increase of the thickness, the critical temperature increases quickly and then saturates at around 60 nm. Figure 11 shows the resistance–temperature curve of the 4 nm thick NbN film when P_{N_2} is about 0.16 Pa, the critical temperature is 7.8 K at best. The T_c of the 4 nm thick NbN films deposited on SiO_2/Si substrate at room temperature was around 7–8 K,[17] and the 4 nm thick NbN thin films on MgO had T_c around 11 K.[12] In the original paper of Gol'tsman *et al.*, the T_c of the 4 nm thick NbN films on sapphire substrates was 10–11 K.[19] During their deposition process, the substrates were heated up to 900 K, leading to an epitaxial growth of the deposited films. The NbN epitaxial films deposited on MgO and sapphire substrates are single crystalline. In contrast, the NbN films deposited on Si are polycrystalline. Because of the lattice mismatch between the Si substrate and the NbN thin film, the superconducting properties of the NbN thin films grown on Si substrates are slightly worse compared with those of the NbN epitaxial films grown on MgO and sapphire substrates. The critical temperature of the NbN films deposited on SiO_2/Si substrate is relatively low. But lower T_c makes the device have a smaller superconducting energy gap, which can improve the sensitivity of the nanowires to photon. At present, the performance of the device fabricated on Si substrate is superior to that on other substrates.[18]

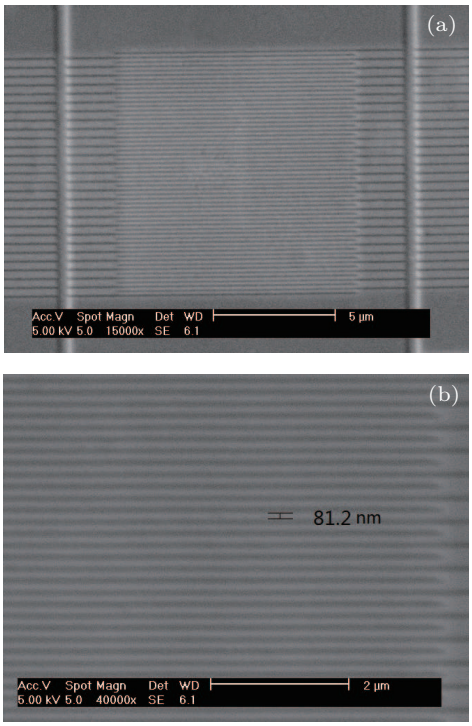


Fig. 8. (color online) SEM images of a NbN meander nanowire sample with different magnifications.

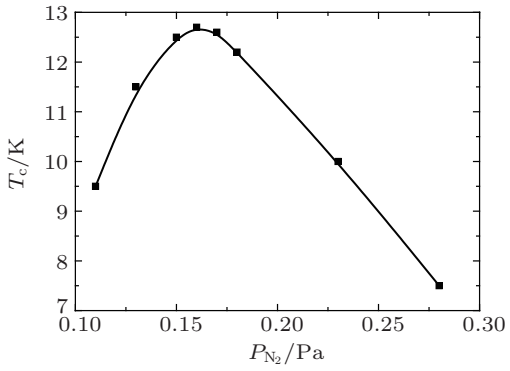


Fig. 9. The superconducting critical temperature T_c dependence on the nitrogen partial pressure ($P_{total} \approx 1$ Pa; $d \approx 50$ nm).

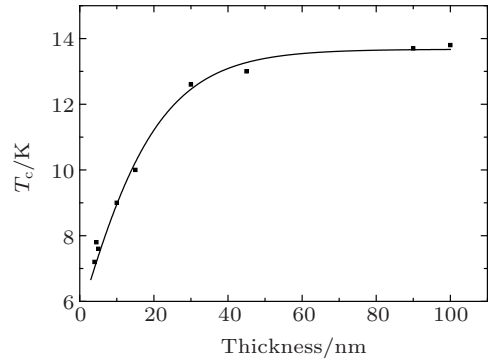


Fig. 10. The superconducting critical temperature T_c dependence on the NbN film thickness.

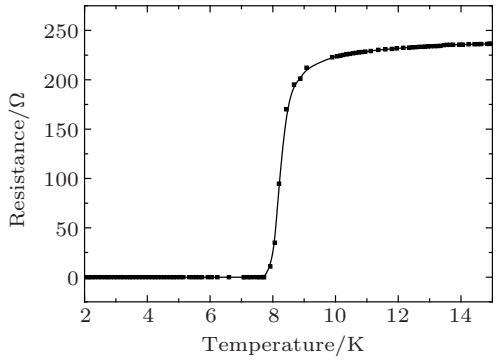


Fig. 11. Resistance–temperature curve of the 4 nm thick NbN film when P_{N_2} is about 0.16 Pa.

In order to obtain the superconducting properties of the NbN meander nanowires, electrodes were fabricated by UV lithography and the lift-off method. The superconducting properties of the device were tested by the four-wire method in the PPMS system. Figure 12 shows *R*–*T* and *I*–*V* curves of the NbN nanowire device. The two graphs illustrate that T_c of the NbN nanowire structure is about 5 K and the critical current (I_c) is about 3.5 μA at the temperature of 2.5 K. The critical current density (j_c) evaluated from the *I*–*V* curve is about 1.2×10^6 A/cm^2. After the NbN films are fabricated to meander lines, the critical temperature of the NbN films is decreased from 7.8 K to about 5 K. NbN nanowires of 100 nm wide and 4 nm thick were fabricated using direct EBL and RIE on sapphire[19] and MgO.[11] The critical temperatures of the device and the film were practically the same. This fact testifies that the technological route of SSPD manufacturing has little negative impact on the NbN film. We have prepared the NbN meander nanowire structure by the method of NIL. However the nanowire structure has lower T_c than the device fabricated by EBL.

The *R*–*T* curves measured in a series of magnetic fields are shown in Fig. 13. The magnetic fields were applied in parallel and perpendicular to the sample surface, respectively. The magnetic fields increased from 0 T to 9 T (0 T, 0.1 T, 0.3 T, 0.5 T, 1 T, 2 T, 3 T, 4 T, 5 T, 6 T, 7 T, 8 T, and 9 T from right to left). The results show clearly that the critical field is strongly anisotropic for the sample, which is very similar to what was observed previously in Nb meander nanowire samples.[8]

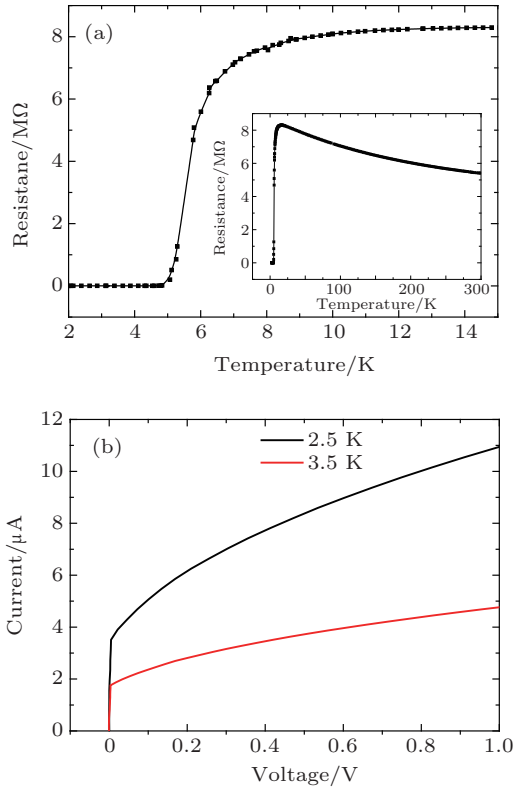


Fig. 12. (color online) (a) Resistance–temperature and (b) current–voltage characteristics of the NbN nanowires.

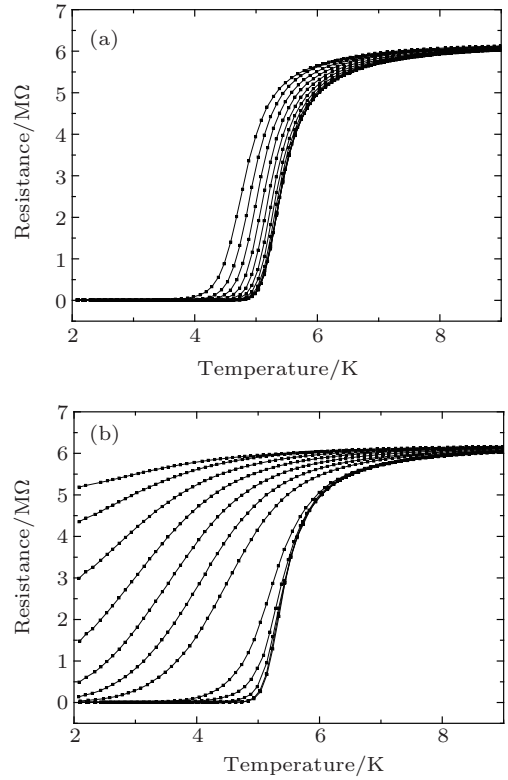


Fig. 13. The *R*–*T* curves measured in a series of magnetic fields (a) parallel (b) and perpendicular to the sample surface.

4. Conclusion

By using HE-NIL and UV-NIL, we have successfully prepared an NbN meander nanowire structure with lines of 80 nm width covering an effective area of 10 μm×10 μm. The NbN meander nanowire is relatively uniform and the critical temperature of the NbN nanowire is about 5 K. We believe that the devices fabricated by NIL technique can be used for single photon detection.

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