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NV-center diamond cantilevers: Extending the range of available fabrication methods

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ABSTRACT

We present an alternative fabrication process for single crystal diamond cantilevers that consist of a platform carrying a nanopillar containing nitrogen vacancy centers close to its surface. These cantilevers are suitable for vector magnetometry by fitting them to the scanning unit of an atomic force microscope. Our methods extend existing technology to lower electron voltages (30 kV) and standard reactive ion etching. Special procedures are presented to overcome both restrictions due to charging of the diamond during electron beam lithography and limitations of the etch depth due to the low plasma density in reactive ion etching systems. The presence of nitrogen vacancy centers in the fabricated nanostructures is confirmed by photoluminescence measurements.

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1. Introduction

Nitrogen vacancy defects (NV-centers) in diamond are a powerful tool for imaging magnetism of objects on the nanoscale with both high spatial resolution and high sensitivity for small stray fields [1,2,3]. To perform magnetometry in a scanning probe experiment the NV-centers may be introduced into diamond nanocrystals that are subsequently attached to an atomic force microscope (AFM) tip [4,5,6] or into single crystal diamond (SCD) [7]. The former suffer not only from an inherent finite probability of formation of bonds between nanocrystal and sample surface and thus disabling the sensor but also from short spin coherence times due to poor crystal quality and inefficient far-field collection of the fluorescence from the nitrogen vacancy center [7]. The latter requires non-trivial fabrication of a monolithic diamond cantilever from the SCD, where the cantilever consists of a platform carrying a nanopillar. These cantilevers can readily be used as a sensor in an AFM. The nanopillar forms the tip of the microscope and carries the NV-centers close to its surface providing the opportunity to achieve close proximity between the object of interest and the NV-centers during a scanning probe procedure. Typical dimensions for the cantilevers are thicknesses up to a few μm whereas the nanopillar has a diameter of ~200 nm and an aspect ratio between 5 and 10.

The previously reported method for the fabrication of such monolithic cantilevers and their mounting to the AFM scanning unit relies on complex equipment such as high voltage electron beam lithography (EBL) and inductively coupled plasma (ICP) etching systems [7]. 100 kV EBL is carried out on both sides of a few μm thick diamond membrane. Membrane and nanopillars are etched out of the single crystal using a multi-step ICP etching recipe with alternating O_2 and Ar/Cl_2 etching to maintain a smooth surface. The alignment of subsequent lithography layers on both sides of the sample is carried out using a high precision laser interferometer stage (LIS) and markers. Finally a dual electron/focused ion beam (FIB) system is used for cutting the cantilevers out of the membrane.

Here we present an alternative fabrication method for SCD cantilevers and extend thereby the range of available techniques for diamond microstructure manufacturing.

2. Materials and methods

The cantilevers are fabricated from a CVD (chemical vapor deposition) grown slab of highest purity, single crystal (100) diamond with dimensions of $2 \times 2 \times 0.05$ mm^3 (sourced from Element Six). To allow for surface identification later in the processing, we start with creating an asymmetrical mark on one diamond surface by oxygen plasma etching using a shadow mask technique. To introduce NV-centers we implant the entire diamond surface with ^{14}N ions with an ion energy of 6 keV (done by external company) and perform subsequent annealing for 2 h in ultra-high vacuum at 800 °C. These parameters are chosen to obtain an average implantation depth of 10 nm and a density of ~500 NV/μm^2. In principle the nitrogen implantation can also be carried out locally [8] creating NV-centers only in lithographically defined areas.

To improve the handling of the samples during fabrication we use specific tweezers (e.g., COBALTIMA® Micro-fine Oblique Tip 51-CO) and whenever possible fix the sample in a carrier made from a standard

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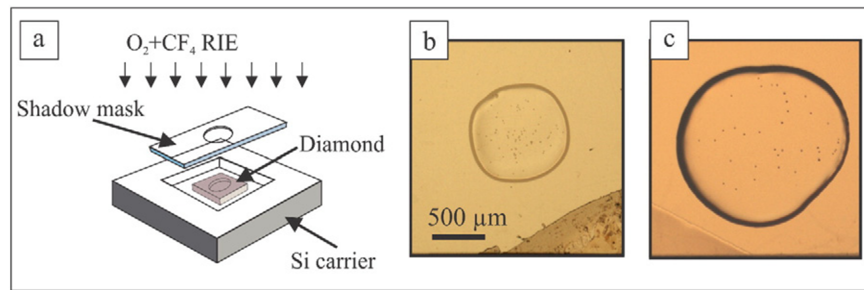

Fig. 1. Schematic of the membrane fabrication process (a) and optical micrographs of two different membranes taken from the face (b) and from the back (c) side of the diamond.

silicon wafer piece (see Fig. 1a). On the carrier we etched a pit by anisotropic wet etching using a SiO_2 mask. The dimensions of the pit are defined such that the diamond crystal can be glued inside it. The depth of the pit is set slightly larger than the sample thickness to allow fixation of a shadow mask on the carrier surface without the mask being in contact with the sample surface. For gluing we use a thermoplastic polymer (Crystalbond™). After each process the sample can easily be separated from the carrier by heating. Glue residues are removed in organic solvents. Other possible carrier materials are described in e carried out locally [9]. The diamond samples are cleaned after each process step in a boiling acid mixture (1:1:1 H_2SO_4: $HClO_4$: HNO_3 (3-acid clean)) e carried out locally [10].

EBL is carried out using a LEO 1525 scanning electron microscope (SEM) equipped with an ELPHY PLUS pattern generator from Raith GmbH. High resolution PMMA resists from AllResist GmbH and hydrogen silsesquioxane (HSQ) resists from Dow Corning Ltd. are used for electron beam lithography. For reactive ion etching (RIE) an AER 100 parallel plate RIE system from FHR Anlagenbau GmbH was used. A chemically assisted ion beam etching (CAIBE) system from Roth & Rau AG (now Meyer Burger AG) is used for argon ion beam etching (Ar^+ IBE). A Helios NanoLab™ 600 dual beam FIB/SEM workstation from FEI Company is used for separating cantilevers out of the membrane and to attach them to a quartz tip. 3-acid clean is done in a special flask connected with a water cooled condenser.

3. Lithographical patterning of the single crystal diamond

3.1. Membrane etching

A diamond membrane is created by etching the central part of the diamond slab starting from the non-implanted surface (back side) to a remaining thickness of ~5 µm using RIE. For this step the diamond sample is glued face down into a silicon carrier and covered with a sapphire shadow mask (see Fig. 1a). The shadow mask contains an aperture with a diameter of ~1 mm. During RIE etching the diamond face surface can optionally be protected by Ti/Au (5 nm/100 nm).

Reactive ion etching is carried out using a mixture of CF_4 and O_2 (1:15) with total flow of 15.5 sccm. The power density is set to 2.5 W/cm^2 and the process pressure is tuned as low as possible (0.7 Pa). To avoid strong heating of the sample we provide good thermal contact between Si carrier and the water cooled sample stage by using Fomblin® oil as a contact agent and alternate etching and cooling cycles. This process results in a low diamond etch rate of 0.033 µm/min since it is deliberately optimized for minimal surface roughness and not for high etch rates. The overall RIE process time adds up to ~37 h.

Fig. 1a shows a schematic of the membrane fabrication process next to optical micrographs of two membranes taken from the face (b) and from the back (c) side of the diamond. In both cases some hillock like structures develop during etching, which act as additional masking material and lead to the formation of membrane defects. These are visible as dark spots. In spite of the hillocks there are sufficiently large defect free areas with both a very smooth surface and very good thickness uniformity where the cantilevers are fabricated.

3.2. Fabrication of the pillars with NV-centers

After membrane fabrication nanopillars together with a set of alignment marks are processed on the NV-center carrying side (face side) of the sample. Typical pillar diameters are 200 nm, corresponding to an average number of ~70 NV-centers per pillar (NV-center ensemble).

For nanopillar fabrication the pillar shape is created in hydrogen silsesquioxane (HSQ) resist with a total thickness of ~500 nm as a first step. This resist pattern is then transferred into the diamond using again a RIE process. Subsequently the etch mask is removed using wet chemical etching. The HSQ thickness is chosen to be sufficiently large to allow for etching of pillars as high as 2 µm.

Obviously 30 kV EBL was found to lead to charging of the diamond sample during writing. Charging problems may be avoided by deposition of a conductive layer on top of the resist [11]. But instead we evaporate a thin Ti layer (5 nm) beneath the HSQ layers. This Ti layer avoids thus not only charging but also significantly improves adhesion of small resist structures. In this way arrays of nanopillars with diameters as small as 70 nm could easily be fabricated with an aspect ratio exceeding 30.

We use XR-1541 and Fox® 16 HSQ resists which are dried using a temperature controlled (120 °C) hot plate after spincoating. Exposing at 30 kV the area dose is found to be ~400 µC/cm^2 and up to 2000 µC/cm^2 for nanopillars with a diameter of 200 nm. The HSQ bilayer is developed for 17 s in a 25% TMAH aqueous solution with subsequent rinsing in deionized water for 10 min. The sacrificial Ti layer is then removed by ion beam etching using argon ions with an energy of 400 eV. The resist pattern is then transferred into the diamond by RIE using a gas mixture containing O_2 and Ar in a ratio of 2/3 at a very low pressure (0.7 Pa) and a low plasma power density of ~0.6 W/cm^2. Adding CF_4 to the gas mixture is not necessary since at etch depths of only 2 µm surface roughness is not yet pronounced. Moreover, the HSQ mask is not fully compatible with fluorine containing RIE processes. Low power density processes are used to reduce mask erosion which is caused by physical etching contributions. To achieve satisfactory results it is essential that the process pressure is kept low and sample heating is avoided whereas the exact gas composition is not as crucial.

In Fig. 2 both optical (a, b) and scanning electron micrographs (c, d) of an actual sample are shown with increasing magnification from a through d. Fig. 2a shows the complete 2×2 mm^2 sample where the total membrane area carries ~25 individual writing fields containing pillars and alignment marks. The 200×200 µm^2 area of one of these writefields is depicted in Fig. 2b. The image shows individually labeled crosses for subsequent field recognition and alignment marks. Scanning electron micrographs in Fig. 2c and d show magnified images of the pillars. Plasma conditions during etching determine the overall shape of the pillars which we optimized to combine a small diameter at the tip with a strong base providing high spatial resolution and robustness during AFM operation.

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