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Overcoated diamond tips for nanometer-scale semiconductor device characterization

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ABSTRACT

Micromachined diamond tips have become the ultimate choice for the electrical probing of semiconductor devices at the nanometer scale because of the required high pressures in the GPa range. Although state-of-the-art full diamond tips (FDTs) show an ultra-high spatial resolution of 1 nm, they are suffering from the limited electrical conductivity of the interfacial diamond layer resulting in an overall lower dynamic range. Conventional coated diamond tips (CDTs) on the other hand show a higher electrical conductivity but their core Si tips are prone to breaking off close to the apex due to the high lateral forces during scanning. Therefore, we developed in this work so-called overcoated diamond tips (ODTs) which are combining the advantages of the high mechanical stability of molded FDTs with the higher conductivity of CDTs. The key is the local underetching of a first molded diamond tip and the subsequent growth of a 50–150 nm thin boron-doped diamond layer onto the interfacial diamond layer. The resulting ODT structure is attached to a metal cantilever and is used for electrical atomic force microscopy (AFM) measurements. The fabricated ODTs are mechanically stable and show a higher conductivity than FDTs. This work presents the probe and fabrication scheme, shows manufactured probe devices and demonstrates their performance in electrical AFM measurements.

1. Introduction

Boron doped diamond tips allow for ultra-high pressures in electrical atomic force microscopy (AFM) and are therefore commonly used for carrier profiling of semiconductor devices by scanning spreading resistance microscopy (SSRM) [1]. In SSRM, a diamond tip scanning at a high pressure of 8–12 GPa transforms the Si into the so-called ß-tin phase which shows a metallic behavior and allows to probe the local spreading resistance which is linked to the carrier concentration. There are basically two types of diamond tips being used: full diamond tips (FDTs) [2,3] with a pyramidal shape made by molding, and coated diamond tips (CDTs) [4] consisting of Si tips which are coated with a thin diamond layer. Although state-of-the-art FDTs are showing a spatial resolution of 1 nm in SSRM measurements [5], they are still suffering from a limited electrical conductivity resulting into an overall lower dynamic range. It has been shown recently that this is due to the lower active doping of the interfacial diamond layer [6]. CDTs on the other hand show a higher electrical conductivity but their core Si tips are prone to breaking off close to the apex due to the high lateral forces during scanning. A diamond tip configuration is hence desired which combines the advantages of the high mechanical stability of molded FDTs with the higher conductivity of CDTs. Therefore, we have developed the concept of overcoated diamond tips (ODTs). In this tip configuration, we first define a Si mold by anisotropic etching and fill it with boron doped diamond (like in the existing FDT approach). The novel element in our surface micromachining approach is the local underetching of this diamond tip followed by the overcoating of a thin boron-doped diamond layer on top of the interfacial diamond layer. Note that this second diamond growth is done into a confined micrometer-size cavity in an upside-down manner. The resulting ODT structure is then bonded to a metal cantilever and the resulting ODT probes can be used for electrical AFM measurements. Our paper presents the probe scheme, explains the fabrication and assembly process using state-of-the-art Si wafer technology and discusses manufactured probe devices. Their performance in electrical AFM measurements is demonstrated.

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2. ODT concept and fabrication

Fig. 1 shows a scanning electron microscopy (SEM) (Hitachi SU8000) image illustrating the typical problem of the CDT (Fig. 1a) configuration; part of the tip is typically breaking off at the apex region due to the high lateral forces while scanning (Fig. 1b). It is mostly an inter-crystalline cleavage through the hard but fragile mono-crystalline Si material. Diamond pyramidal tips (Fig. 1c) are mechanically stable (Fig. 1d) but are showing a lower conductivity than CDTs (about 50–100 kΩ for FDT and 5–10 kΩ for CDT). Fig. 1e shows schematically how both concepts can be merged into the ODT configuration. For the proof-of-concept, we overcoat existing diamond pyramid arrays with thin diamond layers with a thickness ranging between 50 and 150 nm and evaluated nanocrystalline (NCD) versus microcrystalline (MCD). The diamond deposition is done using a hot-filament chemical vapor deposition (HFCVD) system (model 655 by sp3 Diamond Technologies) in a CH_4/H_2 flowing gas ratio of 2.4% for NCD and 1.5% for MCD. The pressure is about 6 Torr for NCD and 25 Torr for MCD and the substrate temperature is about 850 °C. The boron doping is achieved by adding trimethylboron (TMB) into the gas phase with a TMB/CH_4 gas flow ratio of 1% for NCD and 0.4% for MCD. Fig. 1f shows a diamond pyramid array which has been uniformly coated with a 150 nm thick MCD layer. It should be pointed out that the underlying interfacial pyramid diamond layer serves as seeding layer in this diamond growth. Note that the presence of sharp nanocrystals (inset of Fig. 1f) at the apex region is preferred over a smooth coating as these sharp crystals can form small contact points in the electrical AFM measurements. Although sharp nanocrystals were observed for both NCD and MCD layers, the MCD coating showed a lower resistivity (1.8E−5 Ωm) than the NCD coating (3.2E−5 Ωm). We therefore selected the 150 nm thick MCD layer for further experiments.

For the use of ODTs as tips in electrical AFM, a fabrication process is required which yields individual tip units. For this, we developed the surface micromachining approach shown in Fig. 2. First, we are molding a diamond pyramid in a conventional manner. A 100-mm diameter (1 0 0)-oriented Si wafer covered by a 200 nm thick SiO_2 layer (Fig. 2a) is patterned by a lithography step with square openings ranging from 10×10 to $40 \times 40\ \mu m^2$. The pattern is transferred into the SiO_2 layer by wet etching in buffered HF (BHF). Pyramidal etch pits are then formed by anisotropic etching in 30% KOH at 70 °C (Fig. 2b). The remaining SiO_2 layer is etched away by BHF. The wafer is then cleaned in 4% HCl to remove Fe-particle precipitations originating from the KOH solution [7]. The substrate is then seeded with 5 nm diamond nanoparticles as described in detail elsewhere [8] and a 1 μm thick doped NCD layer is grown (Fig. 2c). A 500 nm thick Al film is sputtered onto the diamond film and the ODT area is patterned by a lithography step. The Al film is structured by wet etching and the diamond is etched by reactive ion etching (RIE) in an O_2:SF_6 plasma (Fig. 2d). The remaining Al film is then removed by another wet etching step. The diamond pyramidal area is then locally underetched in 30% KOH at 70 °C followed by another 4% HCl cleaning step (Fig. 2e). Note that the diamond bridge structure holds the diamond tip in place after being released. A 150 nm thick doped MCD diamond layer is finally grown on the NCD film on both topside and the recessed interfacial side (Fig. 2f).

Fig. 3 shows fabricated ODT structures ranging from 10×10 to $40 \times 40\ \mu m^2$ with tip heights from 7 to 28 μm respectively inspected by SEM. Note the presence of sharp diamond nanocrystals at the apex similar in size and shape compared to the proof-and-concept pyramid structures (Fig. 1f). This proves that high-quality diamond films can also be grown onto shadowed structures (the tip apex is rotated 180° away from the substrate

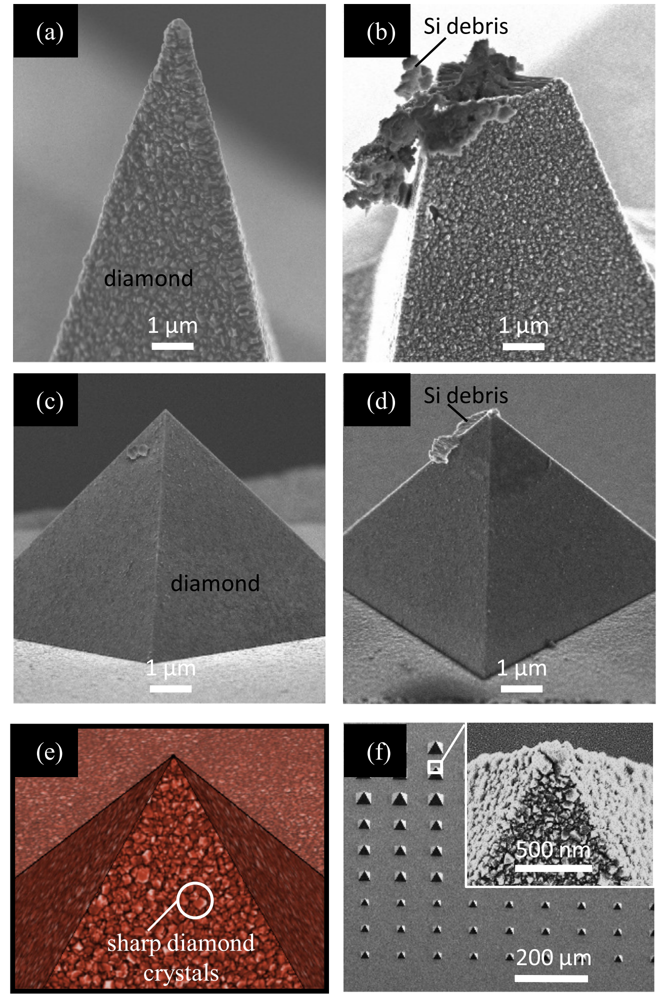

Fig. 1. CDTs (a) break during SSRM measurements (b); FDTs (c) are mechanically stable (after SSRM) (d) but suffer from lower conductivity; ODT concept (e) merging FDT and CDT; proof-of-concept for ODT approach (f).

growth surface) embedded into recessed areas. One typical artifact, which we refer to as 'shark-bite', we noticed during these depositions are smaller areas on the pyramid sidewalls which have been etched instead of overgrown. We attribute this to a local passivation of these areas which prevented the nucleation and growth process at these areas. However, as these shark-bite structures were never noticed on the apexes themselves, they do not affect the functionality of the scanning ODT tips. Besides ODT structures having a pyramidal shape, the developed overcoating approach inside recessed areas can also be applied onto other tip shapes. We fabricated, for example, also ODTs with a knife and triangular shape. The latter are preferred for nanoprobing experiments [9] as the region of interest is visible during probing.

The use of ODT structures as scanning probe tips requires their integration into cantilever beams. As the existing cantilever process developed for FDTs [5] cannot be directly applied on top of recessed areas, we opted for fabricating cantilever and ODT structures on separate wafers and bonding cantilever and ODT modules together afterwards as schematically illustrated in Fig. 4. This approach allows also for a very high ODT integration density onto a single wafer. Fig. 4a shows the tip-less cantilever probe which consists of a Ni beam (305 μm long, 50 μm wide, 5 μm thick) with Ni holder membrane which is fixed to a metallized Si holder chip (3.4 mm long, 1.6 mm wide, 0.35 mm thick). We describe the cantilever probe fabrication in detail elsewhere [10]. For assembly, a