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# Surface engineering for SiC etching with Ni electroplating masks

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*Abstract—This study reports research on Nickel etch mask used for deep plasma etching of silicon carbide (SiC) substrates, aiming to fabricate power integrated devices. Ni masks with significant thicknesses are requested when performing deep etches of SiC, which are less compatible with classic thin film physical vapor deposition (PVD) methods. Uniform and smooth plasma etched surfaces of SiC were obtained without micromasking phenomena upon using Ni masks deposited by electroplating. Detailed here is this Ni masks based technology we developed beyond its limitation, in order to obtain a high-etched thickness of silicon carbide substrates.*

*Keywords - SiC, APTES, etching, Ni electroplating*

## 1. Introduction

Etching of silicon carbide (SiC) substrates is necessary in manufacturing electronic devices such as power transistors and microelectromechanical systems (MEMS), that enable less power losses, higher switching frequencies, and a reduction of the size of integrated components, such as capacitors and transformers [1]. Classical plasma etching of only a few micrometers of silicon carbide is already solved and reported in previous studies [2]. New technologies require the fabrication of microelectronic systems having deeper etching up to 100  $\mu\text{m}$ , and even more, as for example, to insulate the components in power monolithic integrated systems.

The use of Ni as a mask during SiC plasma etching has been found to reduce the micromasking effect, in addition to its good selectivity for SiC up to  $\sim 50:1$  in  $\text{SF}_6/\text{O}_2$  reactive ion etching (RIE) plasmas compared to other metals such as aluminum [3]. Consuming the Ni mask during RIE and inductive coupled plasma (ICP) etching could be a rate-limiting step in SiC etching. High thickness Ni layers are needed, which are

difficult to produce by classic physical vapor deposition (PVD) methods, as evaporation or sputtering. Ni electroplating of a few micrometers on different substrates has been reported in the literature [4]. In our study, we will report the formation of micrometric layers of Ni on SiC substrates using an electroplating process, and detail how to stabilize this layer through growing active catalyzers. Besides, we performed the plasma etching obtaining a considerable etched thickness of SiC.

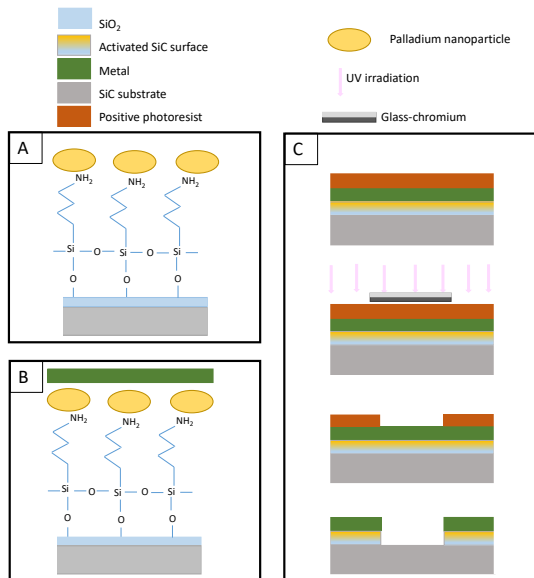
## 2. Experimental part

Commercial n-type silicon carbide, 4H-SiC, off-axis substrates have been utilized for this study on Si-face.

### *A. 3-Aminopropyl triethoxysilane (APTES), functionalization step, growth of Pd nanoparticles and Ni electroplating*

3-Aminopropyl triethoxysilane (APTES), was grafted on the  $\text{SiO}_2$  thin layer natively present on the SiC surface, through the hydroxyl group formed with  $\text{O}_2$  plasma treatment. This method was investigated previously in order to adsorb APTES on oxidized aluminum[5]: 1mL of APTES, was dispersed in 20 mL of a toluene solution. The SiC substrates were immersed in prepared toluene solution containing silane precursor for 24 hours. The resulting amino silane functionalized SiC surface were finally washed with toluene, air dried, and kept in glove box for the next step.

The amine functional group on the other edge is now free to participate in further surface modification reactions (figure 1A).



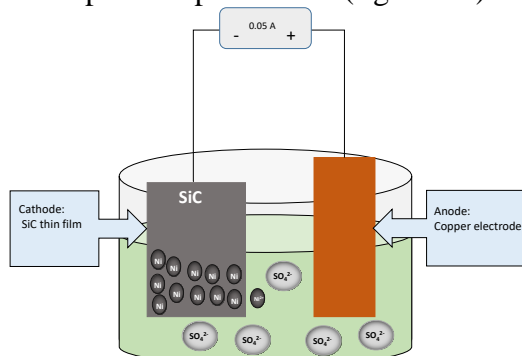
**Fig. 1** A) APTES functionalized SiC substrate. B) Ni electroplated on a functionalized SiC substrate. C) Photoresist deposition on the Ni layer, photolithography, and wet etching to obtain the target pattern.

The activation of the substrate covered with APTES was performed by immersion into an aqueous solution that contained 0.5 g/L of PdCl<sub>2</sub> and 20 mL of 37% HCl at room temperature (RT) for 10 min.

The electroplating solution consists of 1750 mg of (Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O), 1500 mg of (NiSO<sub>4</sub>·6H<sub>2</sub>O) with 2600 mg of boric acid in 500 mL water. The activated samples were coated via an autocatalytic reaction forming a metallic layer deposited from the dissolved Ni ions in the solution at RT for 2 min with an applied current of 0.05 A (figure 2).

### B. Ni mask patterning and SiC etching

To pattern the Ni layer mask, the seemingly simple process of spin coating accomplishes a thick and uniform coating of AZ9260 positive photoresist (figure 1C).



**Fig. 2** Schematic representation of nickel electroplating of silicon carbide substrate.

After coating, the resist film was annealed at 115°C for 15 min. UV photo exposure was done using optical lithography photomask alignment machine (SUSS MicroTec MJB4), for 75 seconds at hard contact with a glass chrome mask. Post-exposure: the substrate is dipped within AZ 726MIF developer. The uncovered metal area was etched using a mixed of orthophosphoric, nitric and acetic acid solution, by dipping the substrate at 60°C for 2 hours, and then the photoresist removed with acetone.

Silicon thin layers are also deposited on the Ni layers to increase the volatility of the Ni by-products, avoiding thus micromasking during SiC plasma etching process [3]. The deposition of silicon was done by electron-beam gun evaporation with Plassys MEB400 machine, at low pressure of  $1.6 \times 10^{-6}$  mbar.

The patterned SiC substrates were etched using ICP/RIE Plassys MU400 reactor. The utilized reactor is composed of two power sources: RIE to form the plasma potential at the substrate surface and ICP to enhance the density of reactive species. The etching gases used are SF<sub>6</sub> and O<sub>2</sub>. Fluorine ions strike the SiC surface and dissociate the Si-C bond, creating SiF<sub>x</sub> and CF<sub>x</sub> volatile species. Oxygen atoms enhance the fluorine atoms generation and interact with the carbon layer creating also volatile CO<sub>x</sub> species.

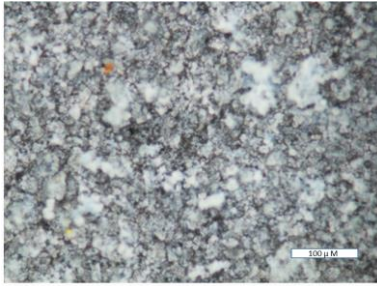
### C. Optical and SEM characterization

The morphology, the patterned masks and the etched trenches were studied using optical microscope (Nikon eclipse) and by field emission scanning electron microscopy (SEM-FEG) with Hitachi SU8030 equipment.

## 3. Results and discussion

### A. Surface activation

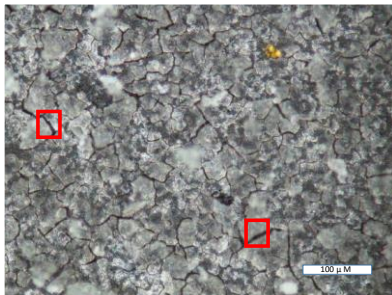
APTES treatment was applied on SiC substrate to graft silane with the hydroxyl group formed through O<sub>2</sub> plasma treatment on the native silicon oxide layer on the surface. The APTES easily reacted with the hydroxyl group forming a self-assembled monolayer (SAM) of organic molecules [6].



**Fig. 3** Optical image of Ni electroplated on a Pd activated SiC substrate.

The binding mechanism of Pd (II) to the amine-terminated SAM was already applied by Guo et al [6] on polyester. We followed the same procedure to create Pd colloids on the SiC surface as active catalyzers for Ni electroplating. The Ni coating is formed from the dissolved Ni ions in the solution (figure 3).

We applied Ni electroplating successfully, directly on the SiC without adding APTES/Pd catalyzers. Nevertheless, we realized that the metal is not stable on the bare SiC surface compared to the functionalized substrate as shown in figure 4. Crakes can be observed clearly on the SiC surface compared to figure 3 where the metal layers appear denser.

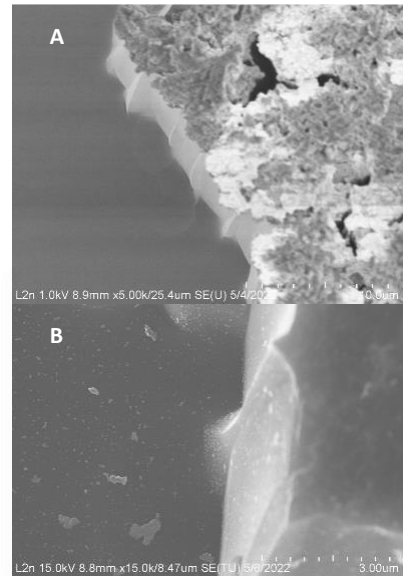


**Fig. 4** Optical image of Ni electroplated directly on SiC substrate.

### ***B. Etch mechanism and surface morphology***

Following Ni deposition on the surface of SiC substrate, the desired Ni pattern has been obtained using  $\sim 12\mu\text{m}$  AZ9260 photoresist, through defining the uncovered metal area needed to etch SiC as shown in figure 1 C.

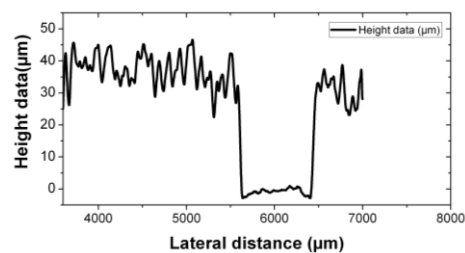
The etching parameters used in ICP/RIE plasma reactor are composed of 8 mTorr work pressure, 25 sccm  $\text{SF}_6$  and 7 sccm  $\text{O}_2$  gas flow, 1000W ICP power and 180W RIE power ( $\sim 430\text{V}$  bias). These parameters were adapted to our ICP/RIE reactor from our previous studies done on RIE reactors [3,7].



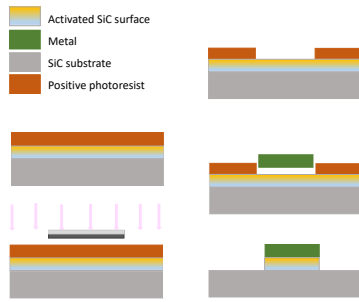
**Fig. 5** Scanning electron microscopy (SEM) images of SiC substrate etched with Ni electroplated masks observed before (A) and after (B) removing the Ni electroplated metal layer.

Figure 5 A and B correspond to an etched SiC surface before and after metal removal. Figure 5 B showed an average etching depth of  $12\mu\text{m}$  obtained after 30 min etching and thus with a  $0.4\mu\text{m}/\text{min}$  etch rate. The jagged profiles at the SiC etched trenches come from the lithography process of our positive resist which has an unusual high thickness but necessary to well cover the Ni electroplating layer.

The thickness of the Ni deposited on the SiC surface remained with approximately the same size,  $\sim 3\mu\text{m}$ , after SiC etching. These results allow us to perform more plasma etching steps to obtain higher etching thickness. A new step of Si e-beam evaporation has also been added before a new plasma etching in order to avoid micromasking [3]. Figure 6 shows a DEKTAK Bruker stylus profile on SiC substrate, ICP/ RIE plasma etched 2 times (30 min each) resulting in a  $\sim 30\mu\text{m}$  etched thickness after Ni mask wet removed.



**Fig. 6** Film thickness profile measurements for Ni patterned Pd activated SiC substrate after two steps ICP/ RIE etching.

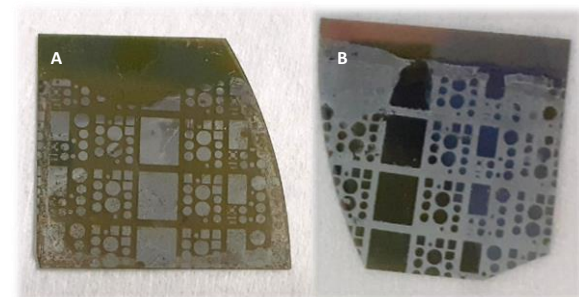


**Fig. 7** Ni electroplated on the photoresist patterned after activation SiC substrate followed by Ni electroplating and resist removing with acetone.

Other than the Ni electroplating followed by the procedure explained in figure 1, we tried to perform an electroplating process by inserting this step after the photoresist deposition and photolithography. Ni growth occurs only on the non-patterned surface with the photoresist (figure 7). Finally, the photoresist is removed with acetone.

Compared with the previous samples, in this case, we obtain a negative image (figure 8B) of the pattern obtained following the procedure detailed in figure 7.

Nevertheless, we noticed that the metal layer was less stable compared to the metal layer deposited directly on the whole activated substrate, and we attributed this to the dissociation of the activation layer through dipping the substrate in the AZ 726 MIF developer then in and acetone.



**Fig. 8** Images of SiC plasma etched substrates A) with Ni growth on the entire surface and patterned after. B) Ni electroplated only on the non-patterned surface with the photoresist.

#### 4. Conclusion

A Ni electroplating procedure was applied on activated SiC substrate. Activation of SiC using APTES, followed by Pd treatment, allows for having a more compact and rigid Ni metal on the surface of the SiC substrate. The Ni layer stays rigid and stable even after

the plasma etching process, without affecting its thickness except at a few hundreds of nanometers. A considerable SiC etching thickness measured around 30  $\mu\text{m}$  was obtained by applying two 30 min plasma etching process, using  $\text{SF}_6/\text{O}_2$ . This study opens perspectives for having larger etching thickness for SiC, by applying a longer plasma etching process conserving the Ni-metal mask on the surface of the SiC substrate.

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