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A Novel Approach for Thin 4H-SiC Foil Realization Using Controlled Spalling from a 4H-SiC Wafer

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Abstract. Porosifying the surface of a single crystalline silicon carbide (4H-SiC) wafer with the means of metal assisted photo chemical etching (MAPCE) promotes the adhesion of an electroplated nickel (Ni) layer. By utilizing a mechanical peel-off process, a Ni layer with tailored mechanical stress is peeled off such that also a thin layer of 4H-SiC is teared apart from the wafer as well.

1. Introduction

Silicon carbide (4H-SiC) is a very promising material for both high temperature and power electronic applications due to its outstanding material parameters such as its wide band gap of 3.26 eV, its high electrical breakdown field of 3 x 10^6 $\mathrm{V\,cm^{-1}}$ and its high thermal conductivity of 490 $\mathrm{W\,m^{-1}\,K^{-1}}$ [1]. The state-of-the-art growth process used to manufacture high quality 4H-SiC wafers involves advanced sublimation growth techniques, which makes 4H-SiC wafers not only expensive (about 40 times more than a comparable silicon wafer) but also a scarce material in the semiconductor industry due to the high demand of this single-crystaline material with a high purity and low concentration of defects [2].

Furthermore, due to various processing steps during device manufacturing such as sawing, grinding or polishing up to 80% of the precious base material is lost [3]. For a more efficient use, some promising methods were developed in the recent decades like Smart Cut™ [4], especially Smart SiC™ which is based upon Smart Cut™ [5], epitaxial layer lift-off (ELO) [6], and their respective variants where surface layers were successfully detached from one mother wafer several times sequentially. But, the described procedures require significant modifications of the substrate, as well as advanced process equipment. Even more, in some cases their application to 4H-SiC is limited [7].

In this work the so-called controlled spalling technology for 4H-SiC is introduced as a straightforward and potentially cost–effective alternative in comparison to the above-mentioned techniques, similar to *Sonic lift-off* [8]. Therefore, it is the goal to achieve controlled spalling via an electro deposited nickel (Ni) stressor layer.

2. Experimental Details and Setup

The working principle. The working principle of controlled spalling is based on the integration of a stressor layer onto the substrate and by using a handle layer mechanically connected to the stressor layer. With the help of this handle layer, one can peel off the stressor layer which results in the detachment of a surface-near portion of the substrate material, when the latter layer shows a high substrate adhesion. For this purpose, the stressor layer is applied in this study by an electrochemical deposition technique. To ensure sufficient adhesion of the stressor layer to the wafer surface for controlled spalling, metal assisted photo chemical etching (MAPCE) is conducted as a pre-conditioning step.

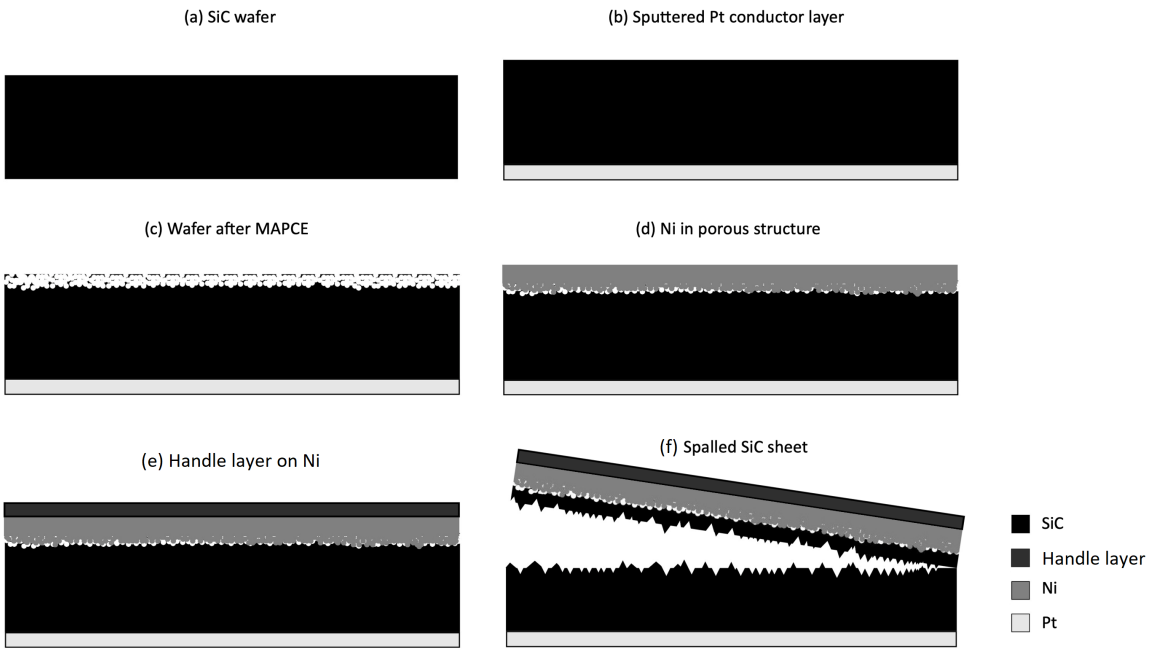


Fig. 1: Overview of the steps used for controlled spalling from (a) to (f). (a) shows the raw 4H–SiC substrate. Sputtering a Pt layer on the backside is done to create a catalytically active layer for further processing as illustrated in (b). MAPCE creates a sponge-like structure in the surface-near region of the 4H-SiC substrate as shown in (c). During electrochemical deposition, nickel (Ni) penetrates into the porosified surface of the substrate as depicted in (d). By attaching a handle layer to the Ni layer as displayed in (e), the substrate is prepared for controlled spalling. A successful spalling attempt can be seen schematically in (f), where a part of the SiC substrate is peeled off.

The individual steps for sample preparation are schematically depicted in Fig. 1 while the above-mentioned methods are explained in further detail in the following.

MAPCE. Metal assisted photochemical etching is utilized to porosify the surface of the 4H-SiC wafer [9]. For this purpose, a platinum (Pt) layer which serves as back side electrode is sputter deposited with a ”Von Ardenne” sputter equipment (LS730S). The etching solution, into which the sample is immersed, consists of hydroflouric acid ($1.31\,\mathrm{mol\,L^{-1}}$ HF) and hydrogen peroxide ($0.15\,\mathrm{mol\,L^{-1}}$ H_2O_2) as oxidation agent and with a UV source, electron hole pairs are generated in the semiconductor material. The Pt noble metal serves as a cathode, whereas, the surface of the wafer is the anode. Finally, the oxidizing agent is reduced at the cathode (Pt) and the surface of the SiC wafer is oxidized (anodic reaction). The oxide generated from this reaction is dissolved in HF [9].

Electrochemical deposition. Electrochemical deposition is carried out with a nickel electro plating bath consisting of nickel sulfate hexahydrate and boric acid. Within this arrangement, nickel stripes are used as anode, whereas the sample is cathodically polarized such that nickel deposition takes place at the side where no Pt was sputtered. The current density and plating time define the thickness of the deposited Ni layer onto the substrate which in turn also determines the intrinsic tensile stress of the Ni layer. As 4H-SiC has a lower intrinsic tensile stress than the Ni layer, exerting force onto it will peel off the Ni layer, ideally cleaving off a thin foil of SiC. This procedure is called controlled spalling and is being demonstrated with gallium nitride [10].

Controlled spalling. To achieve controlled spalling, an external load is necessary to introduce a force into the substrate which is high enough to initiate a crack that propagates through the material. When the system is in a steady state condition, this crack will propagate parallel to the surface at some specific depth as soon as the stress intensity factor K_{II} reaches zero which in turn also defines the theoretical thickness of the spalled layer [11].

Sample preparation. For this study, production grade 4-inch 4H-SiC wafers from SiC-Crystal with a thickness of 350 µm were used and cut into 20 mm x 20 mm pieces by using standard dicing equipment.

These wafer pieces received a Pt contact on the backside and underwent MAPCE for porosification. To study the impact of time on the porosification process the samples were immersed in the etching solution for 15 min, 30 min, 45 min and 60 min respectively. Varying the etching time impacts both the degree of porosification and the etched depth which in turn also affects the adhesion of the Ni layer to the wafer surface (anchor effect).

A custom designed wafer holder was used for Ni plating in the nickel electro plating bath where a circular area of the sample with a diameter of 11 mm is exposed to the solution. The bath itself consisting of nickel sulfate hexahydrate and boric acid had concentrations of 300 g/L ($NiSO_4 \cdot 6\,H_2O$) and 35 g/L (H_3BO_3). With the application of Faraday's Laws, an expression for the nickel thickness s can be stated as in Eq. 1, whereas a is the current efficiency ratio, I/A is the current density and t is the time the current flows [12].

$$s = \frac{12.294aIt}{A} \tag{1}$$

Spalling construction. Spalling experiments were conducted with a self-designed and 3D printed ramp with a length of 121 cm and adjustable height to influence the velocity of the rolling cylinder as seen in Fig. 2a). The wafer is fixed at an appropriate location with an adhesive tape, as shown with the orange circle in Fig. 2b). To ensure that the spalled film is not damaged by this procedure, a double-sided pressure sensitive adhesive tape is selected for flexible force transfer. This tape is partly applied onto the wafer piece and rolled out onto the ramp so that the rolling cylinder adheres to it and strips off a thin foil while passing the wafer, as depicted in Fig. 3. Three different cylinder designs with different mass and diameter were evaluated as described in Table 1. Additionally, three different double-sided sticky tapes were tested.

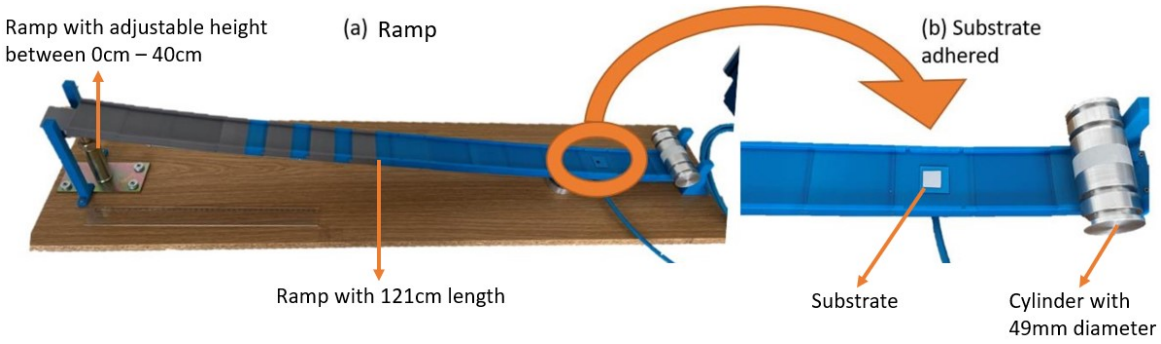


Fig. 2: Double-sided pressure tape prepared on the ramp and adhered to the substrate (a). When the cylinder rolls from the left side down the ramp, the handle layer adheres to the cylinder and peels off a thin foil from the wafer (b).

Table 1: Cylinder parameters

Cylinder #	Mass in [g]	Diameter in [mm]
1	228.5	30
2	589.0	49
3	921.0	60

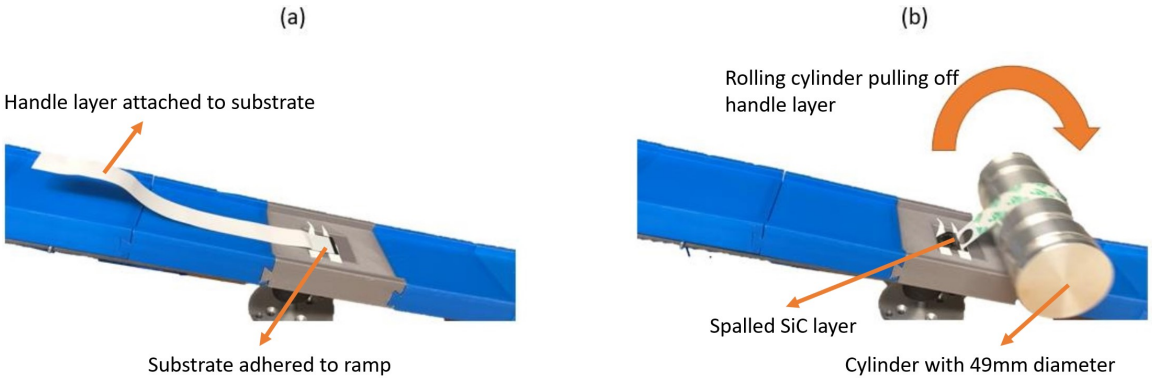


Fig. 3: Picture of the designed and manufactured ramp with a length of 121 cm used for controlled spalling experiments in (a). Close view on the position (b), where the sample to be spalled is placed, while spalling takes place by pulling off the handle layer with a thin foil attached to it. These handle layers have distinct peeling strengths as reported in Table 2. With each of the parameters described, a total number of 24 trials were analyzed to find the most suitable combination of variables as shown in Table 4.

Table 2: Double sided sticky tape parameters

Tape model	Peeling strength of tape in [N mm^{-1}]	Width in [mm]
3M 9087	1.55	19
3M 904	0.50	19
3M 98010LVC	1.16	12

Table 3: Profilometer measurements

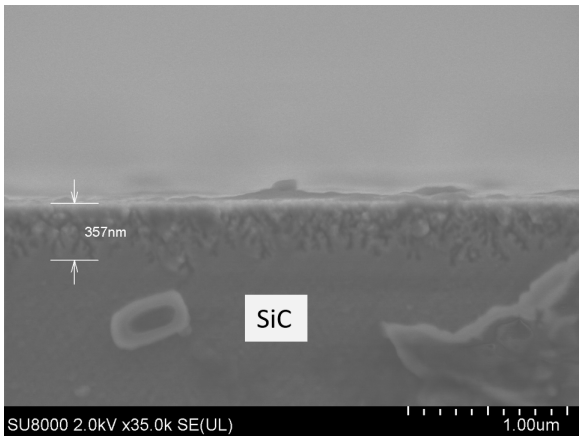
MAPCE time in [min]	Approximate Ni thickness in [µm]
15	16
30	21
45	21
60	18

Scanning electron microscope (SEM). After modifying the surface with MAPCE, several SEM micrographs (Hitachi SU8030) were made to analyze the porous microstructure with an accelerating voltage of 2 kV.

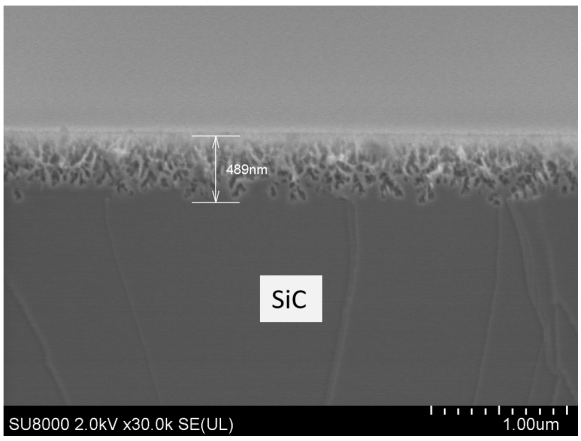
Stylus profilometer. Following up the MAPCE treatment and Ni plating, the surface profile is measured with a stylus profilometer (Bruker Dektak XT) to check on the uniformity in thickness of the plated layer.

3. Results

Increasing the MAPCE time increases the degree of porosification and it is important to find a compromise between etching time and etching depth as total dissolution of the porous 4H-SiC takes place if the wafer undergoes MAPCE for a longer period of time [9]. Spalling experiments in combination with SEM analysis showed that porosification times between 15 min and 60 min are sufficient to guarantee enough adhesion between the Ni film and the SiC substrate. In Fig. 4a - 4b, the different porosification depths are observed after etching for 15 min (357 nm) and 30 min (489 nm) respectively. To restrict the parameter space, a current density of 73.66 mA/cm^2 was selected for Ni plating according to Eq. 1. Some preliminary tests have shown that increasing the current density up to 105.23 mA/cm^2 did not have any impact on the results of the spalling experiments within the used parameter range. To keep



(a) SiC after 15 min MAPCE



(b) SiC after 30 min MAPCE

Fig. 4: Porosification result of 4H-SiC after different MAPCE times

the number of variables in an acceptable range, this parameter is kept constant. Furthermore, the exposed diameter of 11 mm in the wafer holder was selected to keep a solid frame around the porosified area for easier sample handling. With the profilometer the thickness of the Ni layer was measured, as seen in Table 3.

Table 4: Experimental parameters

Experiment #	Cylinder #	Tape	MAPCE time in [min]	Plating current in [mA]	Height in [cm]
1	1	3M 9087	30	70	20
2	1	3M 904	30	70	20
3	1	3M 98010LVC	30	70	20
4	1	3M 9087	30	70	40
5	1	3M 904	30	70	40
6	1	3M 98010LVC	30	70	40
7	1	3M 9087	15	70	40
8	1	3M 9087	45	70	40
9	2	3M 9087	30	70	20
10	2	3M 904	30	70	20
11	2	3M 98010LVC	30	70	20
12	2	3M 9087	30	70	40
13	2	3M 904	30	70	40
14	2	3M 98010LVC	30	70	40
15	2	3M 9087	15	70	40
16	2	3M 9087	45	70	40
17	3	3M 9087	30	70	20
18	3	3M 904	30	70	20
19	3	3M 98010LVC	30	70	20
20	3	3M 9087	30	70	40
21	3	3M 904	30	70	40
22	3	3M 98010LVC	30	70	40
23	3	3M 9087	15	70	40
24	3	3M 9087	45	70	40

Different heights of the spalling ramp with a length of 121 cm were tested, showing that a height of less than 20 cm was not enough for any of the cylinders in Table 1 to peel off the handle layer due to the lack of kinetic energy. Three different double-sided sticky tapes with different peeling strengths were selected for the spalling tests to evaluate the minimum strength needed.

From Table 4, cylinder 1 with the least mass was mostly caught by the sticky tape in experiment 1 - 8 and stopped rolling without pulling off the handle layer. The only case where the peeling of the tape successfully took place was with experiment 5 (tape *3M 904*) as the peeling strength was low in comparison to the other tapes. But unfortunately, no spalling took place. A similar situation occurred with cylinder 3, but in this case the length of the ramp was not sufficient to provide the kinetic energy necessary to peel off the tape as the cylinder just got stuck at the tape. Also here there was only one observation where the peeling took successfully place and that was with experiment 21. In this case also no spalling occurred due to the low peeling strength of the tape. Cylinder 2, however, allowed to successfully peel off all three types of tape as soon as the ramp reached a height of 40 cm. A sweet spot is found with tape *3M 9087* in combination with a ramp height of 40 cm like in experiments 12, 15 and 16. Before conducting spalling attempts, a SEM imaging is performed on a wafer with porosified

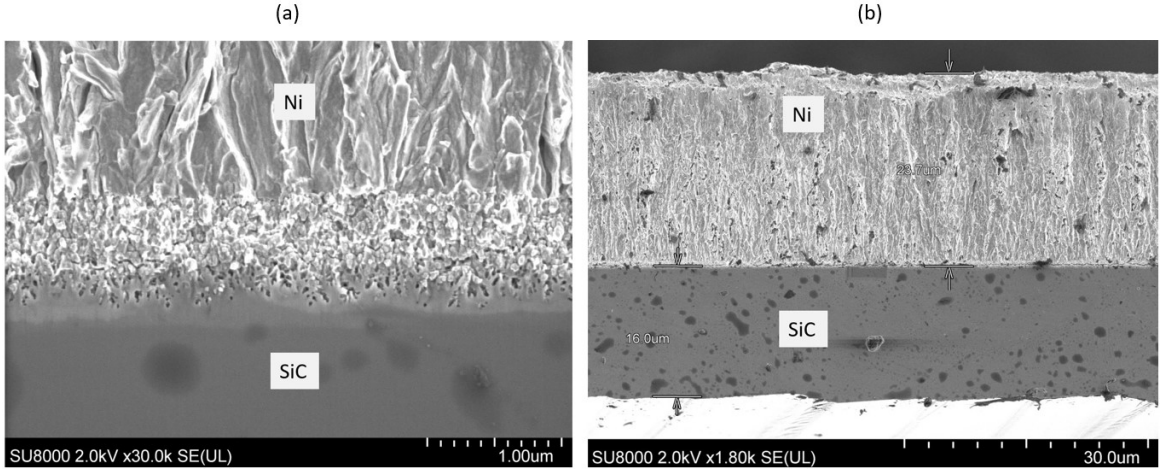


Fig. 5: SEM micrographs of a 4H - SiC wafer after electro deposition (a) and spalling (b). (a) shows a cross-sectional view of 4H-SiC after Ni deposition. A clear penetration of Ni into the open porous structure is observed. 4H-SiC foil with Ni stressor layer attached on the top, as depicted in (b). (SEM micrographs taken from reference [13]).

surface where Ni is deposited, as shown in Fig.5a [13]. After the layer was spalled, the produced foil underwent a SEM analysis showing a successful attempt in creating a 4H-SiC layer with a thickness of about 16 µm, as depicted in Fig. 5b [13].

4. Conclusion

First attempts to create thin foils of 4H-SiC using controlled spalling are reported. To prepare the SiC samples for electro deposition, MAPCE is applied for a specific time to improve adhesion of the Ni layer to the wafer. This chemical etching process should be conducted such that the etching solution is not depleted of oxidizing agent as otherwise the generated porous structure is dissolved completely, rendering the preparation useless. With all the samples tested, it is important to use a handle layer with an appropriate peeling strength and to use a ramp setup where the cylinder with its properties can exert enough force to the SiC sample surface to initiate controlled spalling with the sticky tape. Doing so, thin foils can be detached from a 4H-SiC wafer with a low-cost technique. Fig. 5b clearly illustrates that the thin foil comprises a fairly large portion of single crystalline 4H-SiC material in comparison to the porosified structure which further supports utilizing the controlled spalling technique to create thin 4H-SiC foils.

In the near future, the focus is to study the spalling process in more detail as the exact mechanism is yet unclear. By modifying the electro deposition parameters, the tensile stress of the stressor layer can be changed to initiate the spalling process more reliably. When knowing the necessary peeling force, further research effort can be invested to get further insights about the controlled spalling process. Additionally, further investigations on removing the Ni stressor layer will be required once the process is better understood so that the devices produced from the spalled films are not affected.

5. Acknowledgement

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