# Electropolishing of n-type 3C-polycrystalline silicon carbide

N. Ballarin [a], C. Carraro [b], R. Maboudian [b], L. Magagnin [a,*]

[a] Politecnico di Milano, Dip. Chimica, Materiali e Ing. Chimica Giulio Natta, Via Mancinelli, 7, 20131 Milano, Italy
[b] Department of Chemical and Biomolecular Engineering, University of California at Berkeley, USA

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## 1. Introduction

Silicon carbide has many attractive properties that make it a suitable alternative to silicon and a promising material for microsystems working in harsh environment [1–5]. Mechanical robustness, chemical and high thermal stability, and excellent wear resistance are examples of the properties that make SiC suitable for such applications. Unfortunately, these properties also entail considerable difficulties in key microfabrication steps, particularly in those involving chemical processing of SiC thin films, such as etching and planarization. After polycrystalline SiC film deposition, roughness reduction is required for further fabrication steps, such as high-resolution photolithography, as well as to take full advantage of desirable characteristics as low friction coefficient and self-lubricity [6]. Whereas planarization of single crystalline SiC is a relatively mature technology, techniques that are suitable for planarization of polycrystalline films are comparatively less developed [5]. The present work addresses the surface polishing of cubic polycrystalline SiC thin films by electrochemical etching.

Electrochemical etching of SiC has only recently been investigated and most works focus on monocrystalline SiC. Rysy et al. [7] studied the etching mechanism of both p- and n-type 6H-SiC in hydrofluoric acid HF solutions, which is driven by holes and made of a first surface oxidation step followed by an oxide layer removal due to the HF in the electrolyte. Van Dorp et al. [8] assessed that the reaction can be described by a 6-hole process with some contributions of an 8-hole process. The photoelectrochemical behavior of SiC, stressing out the importance of UV illumination to achieve electrochemical etching of n-type SiC, was discussed by Van Dorp et al. [9]. Recently, an interesting study of electrochemical properties of polycrystalline SiC (polySiC) was made by Yang and coworkers [10], who investigated electrical behavior of nanocrystalline 3C-SiC as an electrode material. Shor and Kurtz [11] developed a wet etching process with high etching rate on C-face p-type 6H-SiC, consisting of an electrochemical etching step followed by wet thermal oxidation and oxide removal, obtaining a relatively smooth surface with roughness of about 40 nm. Surface electropolishing of 6H-SiC was also studied by Chang et al. [12,13], who developed a two-step process consisting of anodic oxidation of SiC, followed by oxide layer removal with concentrated HF up to an average surface roughness of about 83 nm. Li et al. [14] combined instead anodic oxidation of 4H-SiC with chemical mechanical polishing, claiming this process to be self-planarizing. Ke and coworkers [15] investigated electropolishing effects on C-face and Si-face, achieving the best planarization on the last one by constant current etching in a mixed HF and ethanol solution. Etching rate was found to be linearly dependent on current density. Shishkin, Ke and Devaty also noticed that surface morphology turns porous as current density increases [16,17].

In this paper, results on surface polishing by means of electrochemical etching of n-type polycrystalline 3C-SiC (polySiC) are presented, demonstrating the possibility of removing a material from the surface and reducing its roughness in the absence of UV illumination.

## 2. Experimental details

The substrates used in this investigation are 4″ polycrystalline n-type 3C-SiC on Si(111); SiC layer is about 2.5 μm thick with average grain size of about 90 nm. The 3C-SiC films were deposited in a low-pressure chemical vapor deposition reactor, using methylsilane and

* Corresponding author. Tel.: +39 0223993124; fax: +39 0223993180.
*E-mail address:* luca.magagnin@polimi.it (L. Magagnin).

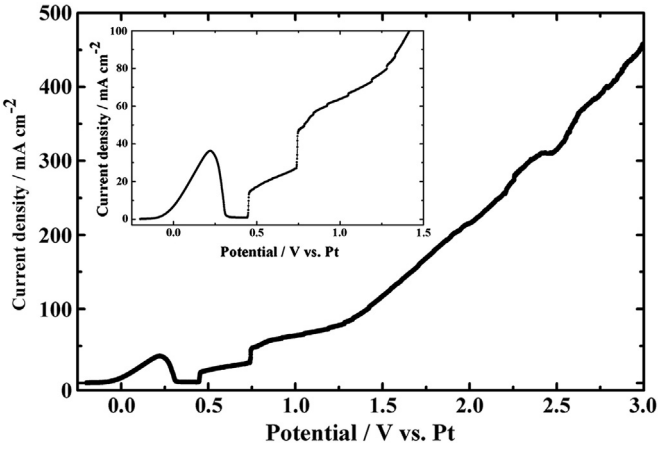


**Fig. 1.** I–V curve of polycrystalline n-type 3C-SiC in hydrofluoric acid 1 wt.%.

dichlorosilane as the precursor gases and in-situ doped using ammonia [18]. Resistivity value is about 8 mΩ cm. Etching is performed in a Teflon cell; the electrolyte is an aqueous solution of hydrofluoric acid HF, with concentrations ranging between 0.5% and 2% by weight. Platinum wires are used as counter and reference electrodes and SiC as working electrode. Potentiostat Keithley 2400 provides electric current to perform electrochemical etching. SiC samples are cut from the substrate wafer; electric contact is made using a short piece of copper wire and attaching one end to the sample surface edge by means of a conductive silver epoxy paste, resulting in an ohmic contact as verified prior to the electropolishing. Uniform etching process at wafer level can be performed if a homogeneous distribution of the anodic current is achieved. After anodic etching, samples are rinsed in de-ionized water and dried under $N_2$ stream. Electrical contact is removed by sonication in acetone for 15 to 20 min, then samples are rinsed again in DI water and cleaned with IPA in order to remove residual particles and finally dried under $N_2$. Current-potential scans are performed with a EG&G potentiostat by National Instruments. Surface morphology and roughness are inspected by Nanoscope IIIa from Digital Instruments and a Solver Pro M from ND-MDT atomic force microscopes; initial root mean square roughness was 17.2 nm in the 10 μm × 10 μm scan area. Surface potential measurements were carried out using the Kelvin probe set-up with gold coated tips. Surface imaging and microanalysis were performed with Novelx mySEM and Zeiss EVO 50 EP scanning electron microscopes.

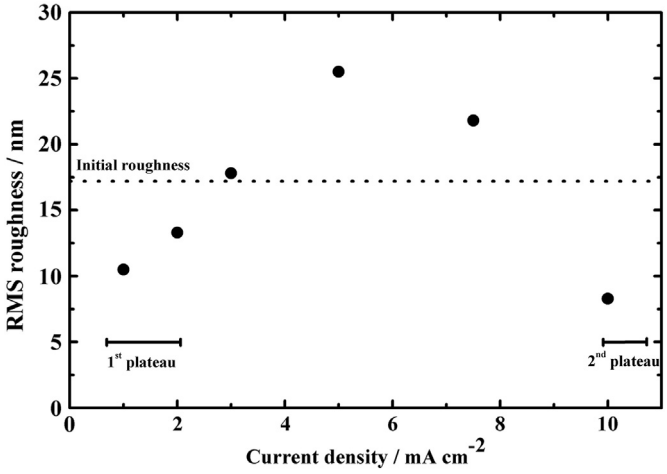


**Fig. 2.** Variation of root mean square roughness as a function of etching current density in hydrofluoric acid 1 wt.% for 30 min. (Scan area 10 μm × 10μm; initial rms roughness 17.3 nm.)

## 3. Results and discussion

Fig. 1 shows the I–V curve of polySiC in HF 1 wt.% obtained from a potential scan between −0.5 V and 3 V with a scan rate of 20 mV/s. The presence of a plateau with low current densities can be observed at potential values higher than 0.3 V vs Pt, indicating the possibility of electropolishing the SiC surface. A first set of tests was run in order to verify the effects of current density on surface morphology and roughness and to assess the optimum value for electropolishing of polySiC. Some preliminary etching runs were carried out imposing etching current density values comparable to those found in literature for monocrystalline SiC electropolishing [13–15], up to 50 mA $cm^{-2}$. Such values are not suitable in the case of polySiC, because they lead to significant SiC layer removal and surface roughening. Lower current density values, in the range 1–10 mA/$cm^2$, were then investigated in subsequent experiments. All experimental conditions apart from current density were kept constant in order to allow comparison of the results. Surface roughness has been measured with AFM and values obtained after each etching experiment were compared with those of unetched samples prior to treatment. We achieved the highest surface roughness reduction etching at 10 mA/$cm^2$ in HF 1% for 30 min: rms roughness is reduced from 17.3 nm to 8.3 nm, so more than half compared to the initial value, as shown in Fig. 2. The correlation between optimal current density and doping was not investigated, expecting minor effects due to the galvanostatic approach of this study.

Surface roughness strongly depends on etching current density: polishing is achieved for lower current density values, namely 1 and

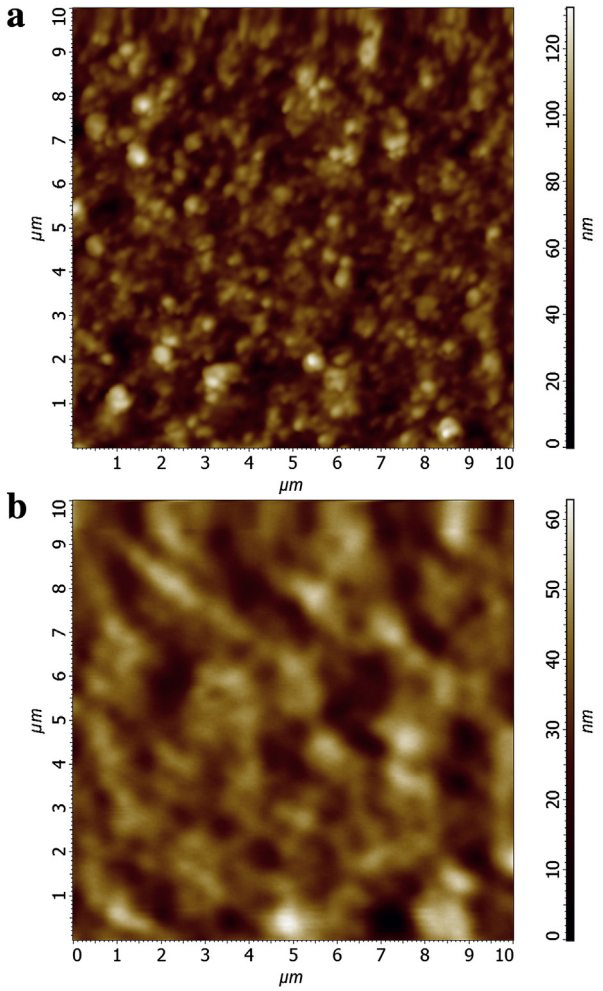


**Fig. 3.** AFM images of polySiC surface a) before (rms roughness = 17.3 nm) and b) after (rms roughness = 8.3 nm) etching at 10 mA/$cm^2$ in HF 1 wt.% for 30 min.

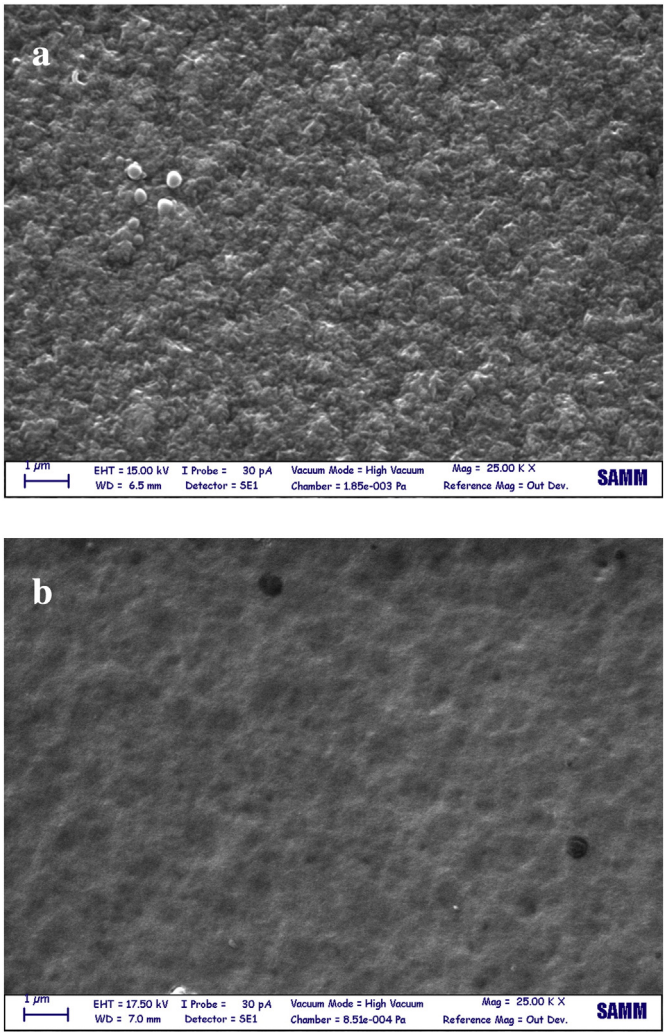


**Fig. 4.** SEM images of polySiC surface a) before and b) after etching at 10 mA $cm^{-2}$ in HF 1 wt.% for 30 min.

2 mA/$cm^2$, then the surface becomes rougher for higher current densities. This is consistent with the plateau observed at lower potential in the I–V curve, where passive phenomena can decrease the current density and favor the dissolution of asperities. A real electropolishing mechanism seems to be consistent with the presence of a second plateau at 0.45 V vs. Pt within a small range of current densities around 10 mA/$cm^2$, before the instability of the oxide interface layer during polishing, e.g. current jump at about 0.75 V, the onset of localized attacks at grain boundaries and transpassive behavior. At 10 mA/$cm^2$ the roughness drops, resulting in the smoothest surface with the lowest roughness value.

Effects of HF concentration were investigated for solutions at HF 0.5%, 1% and 2% by weight, since higher concentrations are known [16,19] to roughen SiC surface. Etching at 10 mA/$cm^2$ leads to surface roughening in HF 2 wt.% due to more aggressive conditions, while polishing is achieved both in HF 0.5 wt.% and HF 1 wt.%, the latter providing the highest roughness reduction. The effect of HF concentration on surface roughness is less critical compared to current density, leading to smaller variations of roughness values. Polishing effect has been observed to decrease with increasing etching time: experiments were run for 30 min, however even in the case of best experimental conditions (etching at 10 mA/$cm^2$ in HF 1 wt.%), the polySiC surface becomes rougher after 60 min and porous after 120 min.

Fig. 3 shows AFM images of polySiC surfaces before and after electropolishing. Surface features such as peaks and heights appear to be smoothened and flattened after the etching process, which is consistent with the measured lower roughness value. Fig. 4 shows planar view SEM images of polySiC samples before and after the etching treatment at 10 mA/$cm^2$: surface appears smoother and more featureless compared to the unetched one. No intergranular and localized corrosion or other degradation phenomena related to the polycrystalline structure were observed on polySiC samples after electropolishing.

Average surface potential was also measured by means of Kelvin Probe Force Microscopy (KPFM) on the best electropolished sample (etched at 10 mA/$cm^2$) and the one polished at 1 mA/$cm^2$; results were compared with the value obtained for the untreated material. Measured surface potential values are: 0.029 V and 0.055 V for the two electropolished samples, respectively. Untreated polySiC average surface potential is 0.196 V. Surface potential is found to decrease after the electrochemical etching treatment, suggesting a modification of the surface chemical groups at the surface, consistent with the removal of oxide species; it slightly reduces also as polishing gets more effective.

## 4. Conclusions

We demonstrated that electrochemical etching is a suitable technique to polish n-type polycrystalline 3C silicon carbide surfaces. Compared to the electrochemical etching of monocrystalline SiC, this process requires lower current density values and no need of UV illumination, and it provides smooth and polished surfaces. Surface roughness strongly depends on etching current density, etching time and HF concentration, the first being the most critical parameter. The best result in terms of roughness reduction is achieved etching at 10 mA/$cm^2$ in HF 1 wt.% for 30 min; after the treatment, surface roughness is more than halved compared to the initial value, decreasing from 17.3 nm to 8.3 nm. polySiC surface appears smooth and uniform; no sign of intergranular or localized corrosion could be observed after the treatment.

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