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Micromachining of p-type 6H–SiC by electrochemical etching

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Abstract

In order to structure p-type single-crystal 6H–SiC wafers for micro-electro-mechanical systems (MEMS) applications, electrochemical etching at a constant current density is used. In this paper, first, a suitable mask with a Poly/SiO_2/SiC scheme is used for a long-time electrochemical etching of p-type 6H–SiC in a dilute HF solution. Etching rates and etched profiles with different concentrations (1, 2, 4, 8%) of the HF solution and different current densities (50, 100, 150 mA/cm^2) are investigated in order to optimise the etching process. An HF concentration between 2 and 4% and a current density between 50 and 100 mA/cm^2 are suggested to obtain a smooth etching surface and a uniform etching profile. With these process parameters, the etching rate is found to be between 0.40 and 0.95 μm/min. A circular structure with a depth of 200 μm is demonstrated for further MEMS applications.

Keywords: p-Type 6H–SiC; Electrochemical etching; Micromachining; MEMS

1. Introduction

Silicon carbide, a wide band gap semiconductor, is believed to be the ideal material for the fabrication of electronic devices that can operate at higher power levels and operating temperatures than devices produced from Si. Not only does silicon carbide display superior electrical and mechanical properties but processing technologies using silicon carbide are compatible to silicon microfabrication technology. Also, silicon carbide has many advantages compared with silicon for micro-electro-mechanical systems (MEMS) [1–3], such as its high toughness, and its capability to work at high temperature and harsh environment. However, due to the lack of bulk micromachining techniques, it is difficult to make three-dimensional structures in a SiC substrate. To date, there are publications that mention the use of high intensity plasma such as magnetron plasma [4], ICP [5] and helicon plasma [6] in order to achieve a high etching rate of 6H–SiC. If we look for wet etching solutions for 6H–SiC, unfortunately, there is no etchant that can attack silicon carbide at room temperature. The only way to etch 6H–SiC at room temperature is electrochemical etching in fluoride based solutions [7]. For the electrochemical etching of SiC, electronic holes are required to make the electrochemical oxidation happen [9]. In p-type SiC, holes are the majority carriers. For the electrochemical etching of this material, it is not necessary to generate holes as in n-type ones.

There are only a limited number of publications, in which the application of electrochemical etching to p-type 6H–SiC is discussed. Previous research investigated the porous structure [8] or the crystal morphology of the p-type 6H–SiC [10,11] after electrochemical etching. Regarding micromachining of p-type 6H–SiC, only two works have been reported: one is by Shor and Kurtz [12], who demonstrated the electrochemical etching of p-type 6H–SiC achieving an etching rate of 2.2 μm/min, and the other is by Rsys et al. [13], who also report about electrochemical etching of p-type 6H–SiC showing an etched cavity about 60 μm deep. However, because of lacking a suitable masking layer, these works do not show any results for etching times longer than 1 h. In this paper, we will demonstrate a feasible etching method for the micromachining of p-type 6H–SiC and investigate etched profiles achieved by different etching parameters, such as the current density and the concentration of the electrolyte.

2. Experiments

In this section, the preparation of p-type 6H–SiC samples for the etching experiments and the set up for the electrochemical etching that is used in our experiments are described, respectively.

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2.1. Sample preparation

Before the electrochemical etching of p-type 6H–SiC, the etching mask and the ohmic contact to p-type 6H–SiC as a working electrode are considered. In the previous etching studies, either a noble metal mask [12] or an O-ring [13] were used for the definition of the region to be etched. However, with these techniques it is not possible to form precise structures and to etch for a longer time. In our case, undoped LPCVD polycrystalline silicon is used as an etching mask because it is not attacked by an HF solution. Before the LPCVD process, the silicon carbide wafers are thermally oxidized to grow a thin insulation layer of SiO_2. It has been reported by Johnson et al. [14] that the etching rate of thermal oxide layers on 6H–SiC is similar to that on silicon. Since the HF concentration of the electrolyte in our experiments is lower than 10%, the etching rate of the oxide layer is below 70 nm/min and is much lower than the etching rate of p-type 6H–SiC we expect to achieve. So the undercutting of the poly-silicon etching mask during the electrochemical etching will not be serious. For the ohmic contact, aluminium is used because it is the natural dopant of p-type 6H–SiC [15].

The whole sample preparation is illustrated Fig. 1 and is described as follows: 3.5° off-oriented $\{0\,0\,0\,1\}$ p-type 6H–SiC wafers with a doping concentration of 2×10^{18} were purchased from Cree [16] (Fig. 1a). After the modified RCA clean process, the oxide layer is grown at 1150 °C for 5 h (Fig. 1b). Then, an 890 nm thick poly-Si layer is deposited at 590 °C by LPCVD (Fig. 1c). Using standard photolithography, the areas to be etched are opened on the C-face of the silicon carbide wafer (Fig. 1d). The poly-Si and oxide layer on the Si-face are removed by RIE and a HF dip, respectively Fig. 1d and e). Finally, a 1 μm thick Al/Si 99/01 is sputtered on the Si-face of 6H–SiC by magnetron sputtering(Fig. 1f). As-deposited, the contact between 6H–SiC and Al behaves like an open-circuit. During annealing at 800 °C for 6 min in an N_2 environment, an ohmic contact is formed and the current–voltage curve becomes linear.

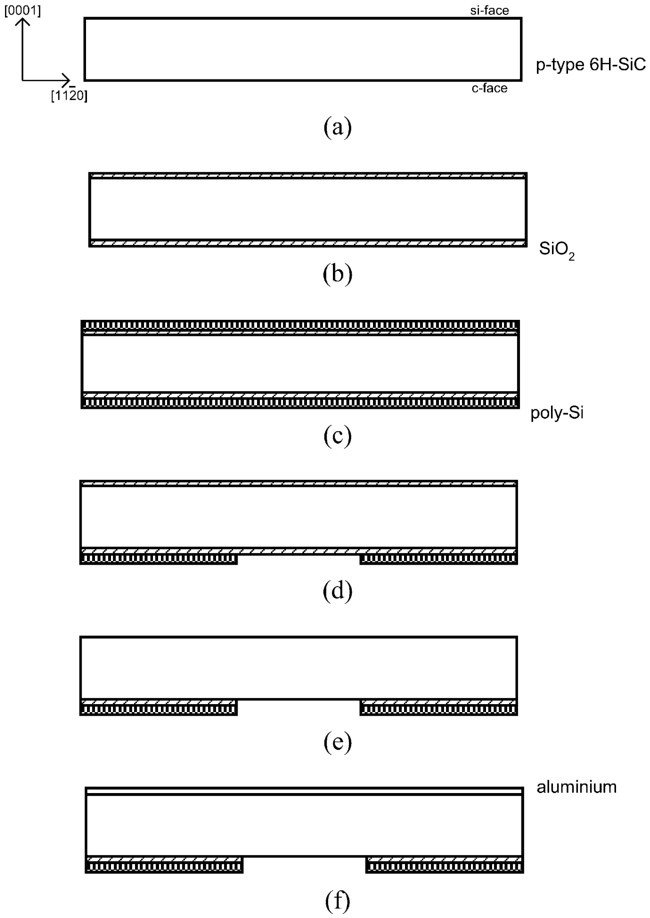

Fig. 1. Process flow for the preparation of p-type 6H–SiC samples: (a) p-type 6H–SiC substrate; (b) wet thermal oxidation; (c) deposition of LPCVD poly-Si; (d) patterning and removing poly-Si by RIE; (e) etching SiO_2 by HF; and (f) sputtering and annealing Al.

2.2. Etching environment

The setup for the electrochemical etching used in our experiments is illustrated in Fig. 2. The sample to be etched,

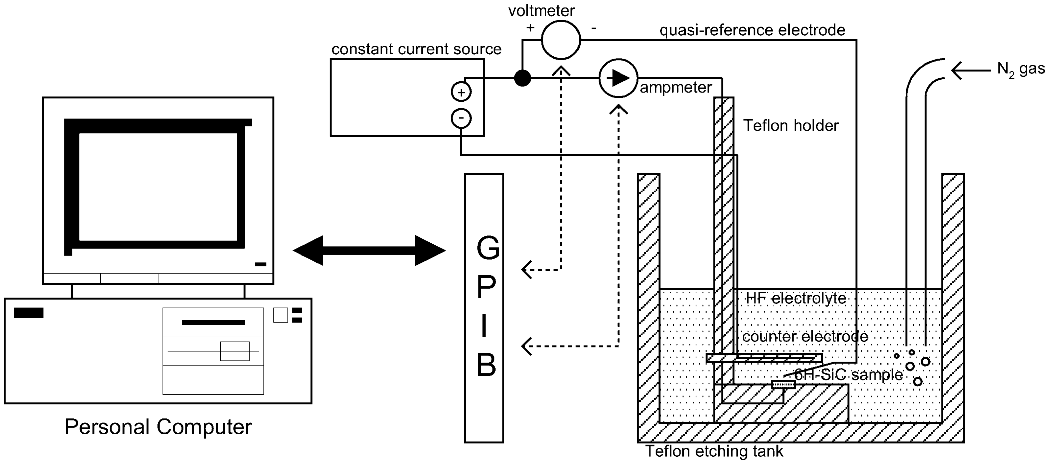

Fig. 2. Setup of the p-type 6H–SiC electrochemical etching.

which serves as the working electrode, is sealed by wax and attached to a Teflon holder. A platinum plate is used as counter electrode and attached to the Teflon holder. There is a platinum wire which serves as a quasi-reference electrode near the working electrode. The working and counter electrodes are connected to a current or voltage source depending on the experiments. A voltmeter and an ammeter (Keithley 192) are connected to the etching cell and a personal computer to monitor the change of the voltage and current during the etching. N_2 is bubbled through the electrolyte during the electrochemical etching. The electrolyte used is HF (50%) diluted by DI water. The concentration of the electrolyte and the current density are varied to study the anodization process of p-type 6H–SiC.

3. Results and discussion

In this section, first, the electrical behavior of the system (SiC sample, electrolyte, and electrode) shown in Fig. 2 is studied. Then, the phenomena observed during the electrochemical etching at a constant current density are described. Finally, etch depths and profiles achieved with different current densities and electrolyte concentrations are measured in order to optimise the process.

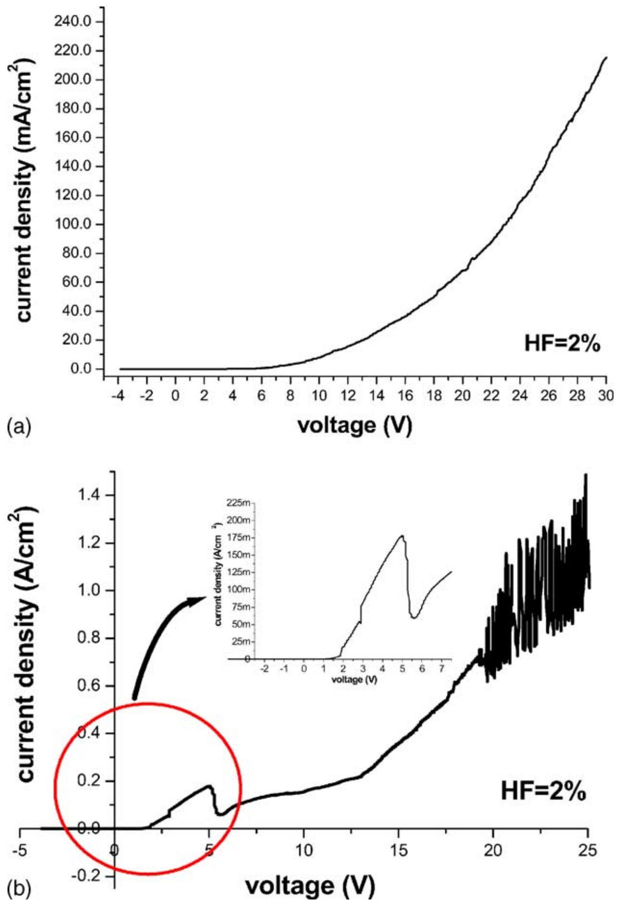

Fig. 3. *J–V* curve of the p-type 6H–SiC: (a) without stirring and (b) with stirring.

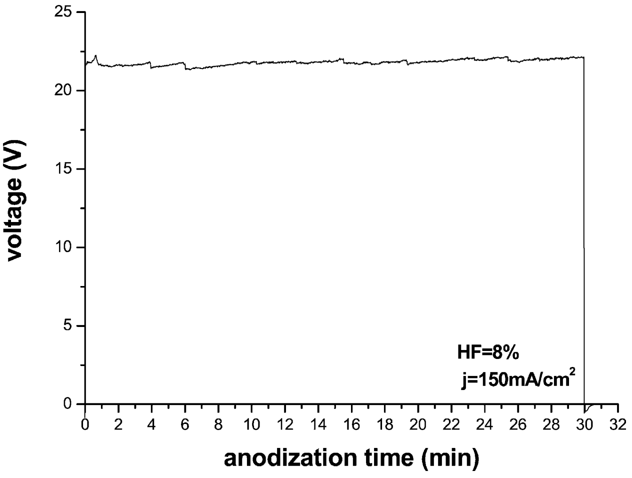

Fig. 4. *V–t* curve of an electrochemical etching of p-type 6H–SiC at a current density of 150 mA/cm^2 in 8% HF. At 30 min, the current source turns off, so the corresponding voltage is zero.

3.1. Linear voltage sweep diagram

The current density depending on the applied voltage, the *J–V* curve, is first measured to evaluate the anodization mechanism. As illustrated in Fig. 3a, the *J–V* curve of a

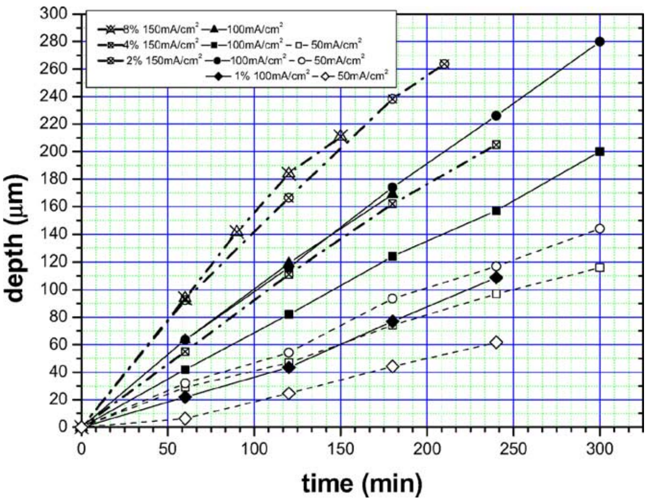

Fig. 5. Etching depth vs. etching time with different current density and electrolyte concentration.

Table 1
p-Type 6H–SiC anodic etching rate (unit: μm/min) with respect to different HF concentration and current density

HF current density (mA/cm^2)	1%	2%	4%	8%
50	0.24	0.40	0.49	
100	0.43	0.67	0.95	0.96
150		0.88	1.31	1.48

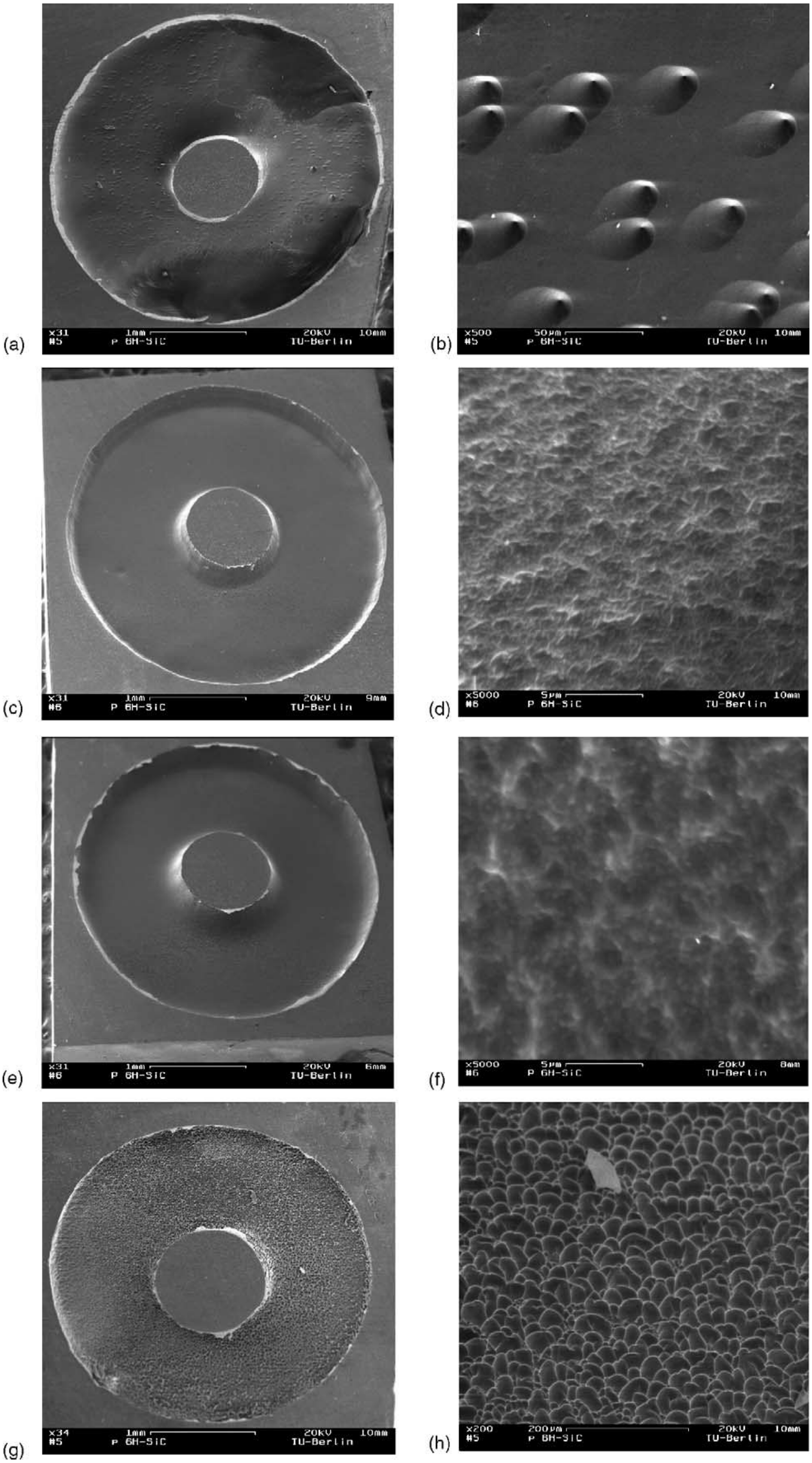

Fig. 6. Anodic etched profiles with different conditions: (a) 8% HF, 100 mA/cm^2; (c) 4% HF, 100 mA/cm^2; (e) 2% HF, 100 mA/cm^2 and (g) 1% HF, 100 mA/cm^2 are the etched annular structures; (b), (d), (f) and (h) are the enlarged pictures from the etched bottom, respectively.

p-type 6H–SiC in a 2% HF electrolyte behaves like a diode because of the junction between p-type 6H–SiC and HF electrolyte. When the electrolyte is stirred using the same sample and concentration of electrolyte, the current density increases compared with the value without stirring, as illustrated in Fig. 3b. This may suggest that the reaction is controlled by the transport of ions as the concentration of HF is low. Fig. 3b shows the flat band voltage, at which the current begins to flow, is about 1.5 V. When the current density exceeds about 750 mA/cm^2, fluctuations in the *J*–*V* curve are observed which means the anodization process is not stable. A situation like this has also been reported in [11] which was explained by extreme gas formation at the surface. Since the anodization process just starts when the applied voltage is larger than the flat-band voltage, the current density at that voltage is very small. In order to increase the reaction rate, the current density must be increased. Therefore, as one can see from the curve in Fig. 3a, the voltage ought to be higher than 25 V, when no stirring is applied.

3.2. *Constant current electrochemical etching*

As Faraday’s law states [17], the mass of an element discharged at an electrode is directly proportional to the amount of electrical charges passed through the electrode. So if the p-type 6H–SiC sample is electrochemically etched at a constant current over a certain time, the amount of the etched mass should be proportional to the etching time. If this hypothesis is true, the etching depth would be proportional to the etching time. So the etching depth could be obtained by time-controlled etching. In order to obtain a linear relationship between the etching time and the etching depth, the samples are anodised at a constant current density. A typical diagram of the anodic voltage changing with time,

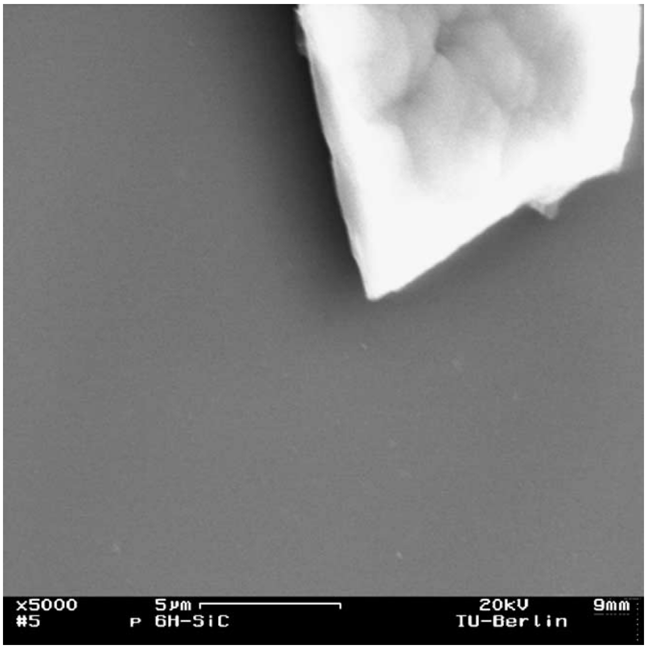

Fig. 7. Surface of the etched bottom at current density 100 mA/cm^2, 8% HF. The white part in the picture is a particle, which is used to adjust the focus.

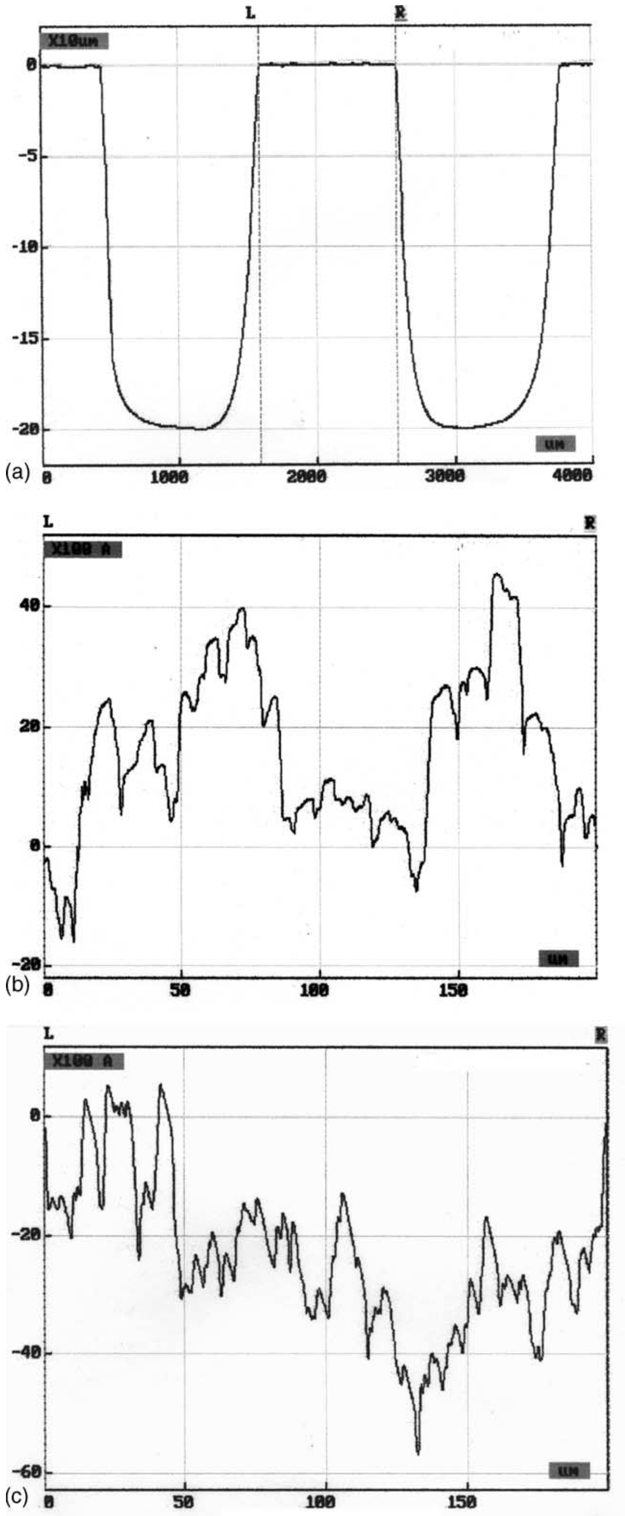

Fig. 8. Profiles measured by a profile meter: (a) an anodic etched annular structure with a 200 µm deep annular cavity; (b) the roughness of the structure before anodic etching and (c) after anodic etching.

the so-called *V–t* curve, in 8% HF is illustrated in Fig. 4. The average working voltage is about 22 V. This voltage remains constant and does not show any evident climb or drop during the anodization process. This means the anodization process will continue as long as electrical charges are constantly supplied.

When an electrolyte with a lower concentration of HF is used, the working voltage increases at the same current density due to the low conductivity of the electrolyte. In our experiments, when a 1% HF was used as electrolyte, the voltage was over the voltage-limit protection of the current source at a current density of $100\,\mathrm{mA/cm^2}$. When the corresponding voltage is higher than this limit, the supplied current will be reduced in order to protect the equipment. In this situation, the total transferred charged per time unit is not uniform, so the etching rate will also not be uniform. This situation must be avoided if a constant etching rate is desired.

3.3. Etching profile

During the anodization, the p-type 6H–SiC begins to dissolve. The anodic etching process of SiC is believed to consists of two steps [18]: first, electrochemical oxidation of silicon carbide occurs at the interface between the SiC sample and electrolyte. The as-formed oxidation layer is then removed by HF in the second step. The observed etching depths for different etching times, current densities, and electrolyte concentrations are illustrated in Fig. 5. The etching rates, which are shown in Table 1, are evaluated by a linear-curve fitting of the results from Fig. 5. There are some interesting points that we can find in Fig. 5 and (Table 1). Firstly, the relation between etching depth and etching time at the same current density and electrolyte concentration is linear. With this linear dependence, we can obtain the desired etching depth by controlling the etching time carefully. Secondly, the etching rate is proportional to the current density for the same HF concentration. This means that we can control the etching rate by the current density. However, a higher current density will result in a violent reaction, which will introduce a large number of bubbles making the etched surface not uniform but rough. Thirdly, at the same current density, the etching rates are similar for HF concentrations of 4 and 8%. But it decreases for HF concentrations lower than 4%. This situation may indicate that a different number of charges are consumed or that other reactions are taking place, such as electrolysis of water, and consume the applied charges as a different concentration of electrolyte is used.

The etched profiles resulted from different etching conditions are observed by scanning electron microscopy (SEM) and illustrated in Fig. 6. When the electrolyte concentration is 8%, the etched surface looks rough as be seen in Fig. 6a. Moreover, the etched surface contains conical pits (Fig. 6b), which have also been reported by other researches [11,13].

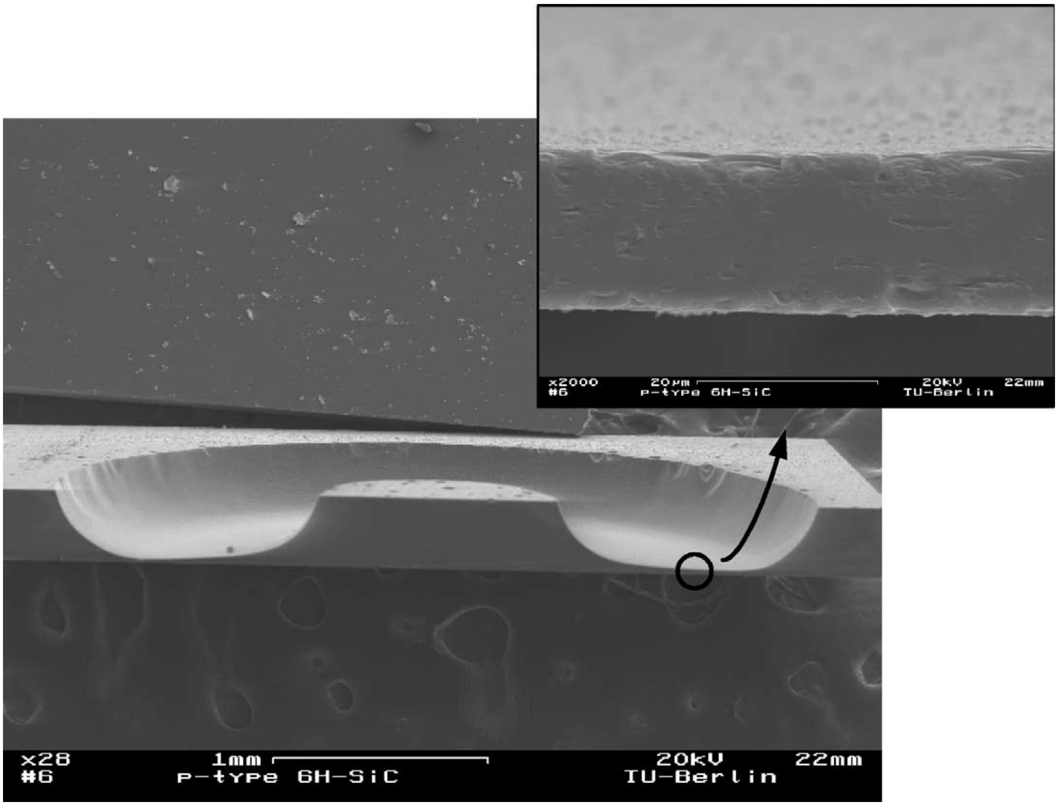

Fig. 9. An electrochemical etched p-type 6H–SiC annular structure. In the right-up window, the membrane about 20 µm thick is magnified from the annular structure.

Although with the conical pits and non-uniform etched profile, the etched surface is very smooth as illustrated in Fig. 7 due to a so-called electropolishing effect. When a 1% HF solution is used, the etched surface, shown inFig. 6g and h), is covered with pores having diameters of about 10 μm. In addition, the surface is very rough. Only when the electrolyte concentrations are 4 or 8%, more uniform and smooth etched surfaces are obtained as illustrated in Fig. 6c–f. With 4% HF, the etched wall is steeper than with 2% HF. However, the etched profile and surface at 4% HF is more non-uniform and rougher.

An etch profile obtained with 2% HF electrolyte and a 50 mA/cm^2 current density measured with a Tencor α-step profile meter is illustrated in Fig. 8. In Fig. 8a, an etch depth of 200 μm is obtained by time-controlled etching. The average roughness of the surface of the p-type 6H–SiC sample before the etching, which is shown in Fig. 8b, is 116 nm, while the roughness of the etched surface is only 83 nm after the etching (Fig. 8c). Fig. 9 demonstrates an etched p-type 6H–SiC substrate with an circular structure, or so-called center-boss structure, which could have applications to pressure sensors.

4. Conclusion

Electrochemical etching at a constant current density is used to fabricate three-dimensional structures in 6H–SiC. With a poly-Si/SiO$_2$/6H–SiC passivating scheme, 200 μm deep structures with smooth surfaces are achieved. Different current densities and HF concentrations are used to obtain a high etching rate with uniform etch depth and smooth surface. The results show, with an 8% HF solution, electropolishing is achieved. It makes defects of the crystal to reveal and results in a non-uniform etch depth. As a 1% HF is used, the lower conductivity of the electrolyte causes a higher voltage, which is not suitable for the electrochemical etching. Moreover, the etching rate is low and causes a rough surface filled with larger diameter pores. With a higher current density, 150 mA/cm^2, a violet reaction and therefore, larger bubbles will be created that make the etched profile non-uniform. A current density between 50 and 100 mA/cm^2 results in more uniform profile but lower etching rate.

The suggested method for the anodic etching of p-type 6H–SiC is to use an HF concentration between 2 and 4% and a current density between 50 and 100 mA/cm^2. With these parameters, a uniform etch depth, a smooth surface and an acceptable etching rate can be obtained.

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