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ECR-Etching of Submicron and Nanometer Sized 3C-SiC(100) Mesa Structures

Lars Hiller[1,a], Thomas Stauden[1,b], Ricarda M. Kemper[2,c], Jörg K. N. Lindner[2,d], Donat J. As[2,e] and Jörg Pezoldt[1,f]

[1]FG Nanotechnologie, Institut für Mikro- und Nanotechnologien MacroNano®, Technische Universität Ilmenau, Postfach 100565, 98684 Ilmenau, Germany

[2]Department Physik, Universität Paderborn, Warburgerstraße 100, 33098 Paderborn, Germany

[a]lars.hiller@tu-ilmenau.de, [b]thomas.stauden@tu-ilmenau.de, [c]rkemper@mail.uni-paderborn.de, [d]lindner@physik.upb.de, [e]d.as@uni-paderborn.de, [f]joerg.pezoldt@tu-ilmenau.de

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Abstract Anisotropic etching processes for mesa structure formation using fluorinated plasma atmospheres in an electron cyclotron resonance (ECR) plasma etcher were studied on Novasic substrates with 10 µm thick 3C-SiC(100) grown on Si(100). To achieve reasonable etching rates, a special gas inlet system suitable for injecting SF_6 into the high density downstream Ar ECR plasma was designed. The influence of the etching mask material on the sidewall morphology was investigated. Masking materials with small grain sizes are preferable to achieve a desired shape. The evolution of the mesa form was investigated in dependence on the gas composition, the applied bias, the pressure and the composition of the gas atmosphere. The achieved sidewall slope was 84.5 deg. The aspect ratios of the fabricated structures in the developed residue free ECR plasma etching process were between 5 and 10. Mesa structures aligned to [100] and [110] directions were fabricated.

Introduction

Silicon carbide is a promising material for various applications in electronics, micro- and nanoelectromechanical systems, as well as for optoelectronical and optical applications. 3C-SiC (100) grown on silicon allows the combination of the outstanding properties of SiC with the large area abilities of silicon technology. Generally, the formation of controlled surface morphologies or lateral structures with dimensions down to sub 100 nm feature size is required. Dry etching techniques are the methods of choice in order to meet the requirements, not only because of the high intrinsic chemical stability of the SiC, but also due to the intended morphology and feature size of the mesa structures.

For several decades the effects of O_2 concentration, temperature [1], bias, pressure [2,3], gases and gas mixtures containing NF_3, SF_6, ICl, IBr, Cl_2, BCl_3, CH_4/H_2, Ar, NF_3/O_2, NF_3/Ar, CF_4 or SF_6/Ar [1,2,3,4] on silicon carbide etching have been investigated. Most of these investigations focused on micron structures and deep etching with high aspect ratios, primarily for applications in high power devices. For this reason inductively coupled plasma (ICP) technology has been used, and high etch rates of about 1.5 µm/min were achieved [5]. ECR etching was used for the preparation of MEMS structures based on epitaxially grown SiC on Si [6]. During recent years, efforts were focused on deep etching, especially using reactive ion etching (RIE) and deep reactive ion etching (DRIE) [7].

In this paper, the investigations are concentrated on submicron and nanometer mesa, as well as gratings. In previous studies electron cyclotron resonance (ECR) plasma etching was used to achieve isotropic etching profiles in SiC [8-10] for usage in MEMS. It will be demonstrated that nanosized structures can be achieved if electron beam lithography (EBL) and a two layer shadow mask technique in combination with lift-off technology are used. Feature sizes down to 50 nm with high aspect ratios, orthogonal slope and low edge roughness can be realized. The influence of the masking material, the SF_6, O_2 and Ar mixing ratio, RF bias and process pressure was investigated to enhance the structure properties in terms of sidewall steepness and line edge roughness.

Experimental

10 µm 3C-SiC(100) grown on 4" Si(100) wafers by Novasic® were used for the experiments. After cutting the wafer into squares of 10 mm x 10 mm, the samples were cleaned in acetone, isopropanol and DI water using an ultrasonic bath. For the first layer of the mask ALLRESIST® CoPMMA AR-P617.06 was spin coated and baked for 1 min at 180 °C. Subsequently, a second layer of PMMA AR-P679.02 was spin coated and prepared in the same way. The two layer system consists of PMMA layers of different thicknesses and sensitivities and for the creation of masks with an undercut and edges with low roughness.

The structures were defined with a Raith 150 EBL system at 10 kV acceleration voltage using a 30 µm aperture. The layout of the test mask contains an exposure dose variation for areas, lines, dots and different gratings. The exposed samples were developed in AR-600-56 developer for 30 s. After a cleaning step in isopropanol and DI water, the masking materials (Ni, Al) were deposited using LES 250 electron beam evaporation. The thickness of the deposited materials was 30 nm. The PMMA resist system was removed together with the surplus metal in a lift-off process, leaving the specimen in 80°C DMSO for 30 min. Cleaning with isopropanol and DI water was repeated.

The masked samples were etched in a dry etching process using an ECR plasma etching tool. The etching setup consists of a box coater type recipient equipped with a turbo molecular pumping system (Pfeiffer Vacuum). The ECR plasma source SQ160 made by Roth und Rau AG has a maximum power of 800 W and works at a frequency of 2.45 GHz. The plasma source is mounted on top of the recipient as a remote type plasma source. All working gases (Ar, O_2, SF_6) were fed from the mass flow controllers (MFC) into the plasma source. The water-cooled 4 inch substrate holder can be charged with DC bias up to -500 V or RF bias by a 13.6 MHz generator up to 100 W. In combination with the large distance of 250 mm between the plasma source and the chuck, these conditions have the potential to vary the etching profile from nearly isotropic profiles as demonstrated in [8-10] to anisotropic profiles [11, 12].

The samples were loaded into the chamber and pumped down to a pressure less than 5×10^{-6} mbar. To remove residues from the PMMA lift off mask an oxygen cleaning process at a pressure of 2.5×10^{-3} mbar, an ECR power of 640 W and a DC bias of -100 V was performed for 10 min. The cleaning process was followed by the SiC etching process. All processes were carried out at an ECR power of 640 W with an RF bias applied to the substrate in a SF_6/Ar mixture. The pressure was varied from 1.5×10^{-4} mbar to 1.5×10^{-2} mbar. The platen power ranged from 2 W to 100 W, resulting in bias voltages from -100 V to -400 V. The influence of mixing additional gases such as O_2, CF_4, CHF_3, C_2H_4 and H_2 was investigated. The nickel mask was removed in $(NH_4)S_2O_8$:$FeCl_3$:H_2O by wet chemical etching.

Results

Fig. 1 displays a typical non-optimized etch profile of a mesa structure prepared by an ECR etching process. The parameters are 40 sccm Ar, 20 sccm SF_6, working pressure 2.5×10^{-3} mbar, ECR power 640 W, platen power 20 W, bias -200 V. The profile shows a relatively anisotropic part on top of the structure which becomes slightly isotropic at the bottom. The increase of the mesa cross section from top to bottom is due to the tapering effect [13]. A strong trenching effect can also be observed. Fig. 2 shows a chevron grating structure with 50 nm line width and 100 nm spacing. The aspect ratio is approximately 5. The structures are well defined and the chevron structure exhibits sharp corners. The bottom of the sample is residue free.

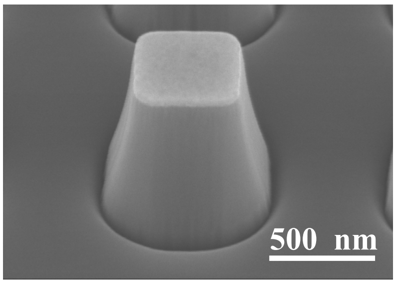

Fig. 1 Etching profile in 3C-SiC using a SF_6/Ar mixture at 640W ECR power, 20 W platen power, 2.5×10^{-3} mbar, nickel mask

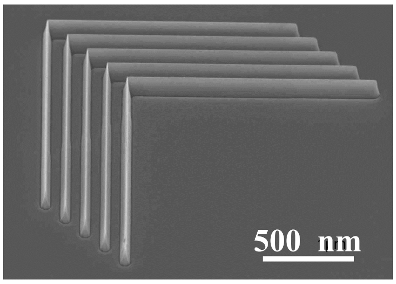

Fig. 2 Lattice structure with line width of 100 nm

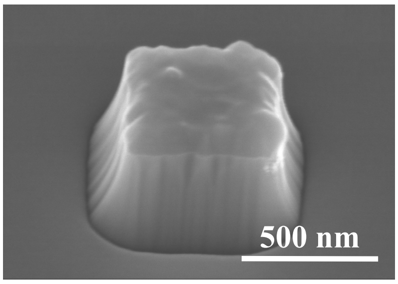

Fig. 3 Etching profile in 3C-SiC using SF_6/Ar mixture at 640W ECR power, 20 W platen power, 2.5×10^{-3} mbar, aluminum mask

The anisotropy is known to depend on a set of parameters including plasma power, platen power, working pressure, gas flows and composition of the gas atmosphere. Initially the masking material had to be optimized. Due to the large projected etching depth, conventional EBL resists such as PMMA or HSQ are not capable of withstanding the SiC dry etching process. Aluminum and nickel were tested as alternative materials. Only the behavior of the nickel mask satisfied the need for high selectivity to SiC as well as fine grains, and was removable by wet chemical etching. The use of aluminum masks led to strongly rippled edges of the mesa sidewalls (Fig. 3). The appearance of this morphological feature results from the larger grain size of the deposited Al layer. The coarser grain structure of the Al layer led to an increased line edge roughness of the mask. This line edge roughness is then transferred to the mesa side wall by the anisotropic etch process. Fig. 3 demonstrates the formation of ripples on the sides of the mesa caused by the columnar structure of the aluminum mask. Metal masks exhibiting smaller grain sizes, for example Ni, allow the fabrication of mesa structures with reduced side wall roughness (Fig. 1).

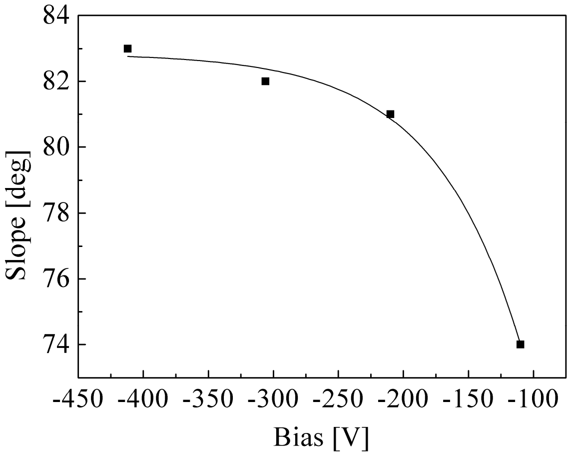

Fig. 4 Side wall slope vs. bias voltage (SF_6/Ar mixture at 640 W ECR power, 2.5×10^{-3} mbar, nickel mask)

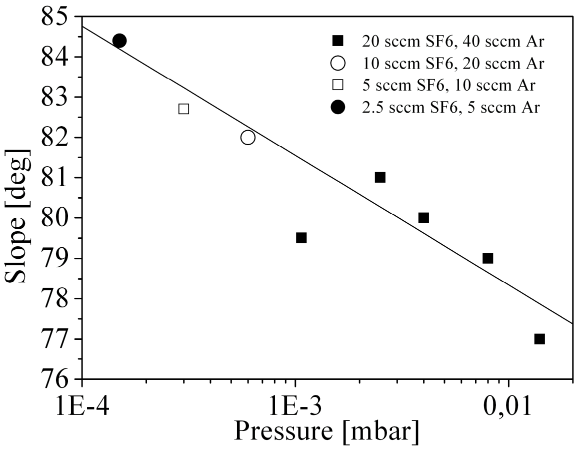

Fig. 5 Side wall slope vs. pressure (SF_6 /Ar mixture at 640 W ECR power, 20 W platen power, nickel mask)

To improve the slope of the mesas, several parameters of the process were investigated. As a first step the gas composition was varied. Gas additions such as oxygen, as well as other gases like CF_4, CHF_3 and C_2H_4, do not increase the sidewalls of the mesa structures substantially. As a second parameter the platen power was varied from 2 W to 100 W resulting in bias voltages ranging from -100 V to -400 V, respectively. The dependence of the side wall slope on the platen power is given in Fig. 4. The steepness of the mesa sidewalls does not increase considerably at bias values above -200 V. It can be seen that the slope will not exceed 83 deg, even with higher bias.

In addition to the platen power the influence of the working pressure was studied. The results are shown in Fig. 5. It is evident that a decrease in pressure led to an increase of the mesa profile’s abruptness. The steepness of the side wall reaches values higher than 84 deg at a process pressure of 1.5×10^{-4} mbar. In contrast to conventional RIE or ICP etching setups, stable plasma conditions can be realized at pressures below 1×10^{-3} mbar in ECR etching machines. In order to reach low working pressures the gas flows had to be reduced. These lower gas flows result in a slight loss in etching rate because of a lower concentration of radicals. Also, due to the low pressure, which causes a higher mean free path length of the species, the flow of the ions and radicals is parallelized and the amount of non-normal approaching species is reduced. This leads to the observed increase in the steepness of the edges of the mesa structures.

Summary

Mesa structures, lines and gratings down to the sub 100 nm range were fabricated by low pressure ECR etching in SF_6/Ar atmosphere. Residue free surfaces were achieved. The sidewall morphology strongly depends on the line edge roughness of the metal mask. It was found that nickel is a suitable material with small grain size and high selectivity to SiC. Smooth side walls were realized with slopes up to 84 deg at a working pressure of 1.5×10^{-4} mbar.

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