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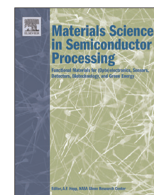

Chlorine based focused electron beam induced etching: A novel way to pattern germanium

M.M. Shawrav, Z.G. Gökdeniz, H.D. Wanzenboeck*, P. Taus, J.K. Mika, S. Waid, E. Bertagnolli

Institute of Solid State Electronics, Vienna University of Technology, Floragasse 7/1, E 362, 1040 Vienna, Austria

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ABSTRACT

Focused electron beam induced etching (FEBIE) with chlorine as etching agent has been used to geometrically shape and to electrically modify semiconductor nanodevices. Selected sections of monocrystalline nanowires were modified directly without the requirement for a photomask or a resist layer. FEBIE as a subtractive nanofabrication technology allows to locally etch active semiconductor devices made of Si or Ge. In this work, chlorine is used as the etchant gas to thin germanium channel structures fabricated by standard photolithography. For effective material removal a sufficiently high electron influence is essential to avoid the pitfalls of this method. Topography and conductivity of FEBIE-modified structures prior and after the etching process was studied by AFM and by electrical *I–V* characteristics. The presented work demonstrates the potential of Cl-based FEBIE for device prototyping and electrical trimming of future Ge-based nanodevices.

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1. Introduction

Due to its high mobility of charge carriers and its CMOS process compatibility, Germanium (Ge) has recently experienced a renaissance as semiconductor material. Moreover, due to its transparency in the mid-infrared range and its suitability for high-frequency applications, Germanium (Ge) based sensors have received increasing attention including bio-chemical applications [1–3]. For the wide range of surface-sensitive sensor devices, it is essential to locally modify the surface of Ge. Conventional fabrication techniques such as wet etching [3], electron beam lithography [4] and nanoimprint lithography [5] have been employed to modify Ge surfaces of nanodevices. However, these conventional techniques have two essential shortcomings that make them ineffective for local trimming and modification: first, standard lithography requires to process the entire substrate and second, these are multi-step processes that require a thin resist layer to be applied on the flat sample surface. Direct modification with a focused beam can solve these two issues- the widely-used direct-writing by focused ion beam (FIB) modification suffers from another vital drawback for semiconductor device: Ga^{+} implantation can severely affect the semiconductor surface [6]. In this work we present Focused Electron Beam Induced Etching (FEBIE) as a gentle direct write method which can modify Ge surface without any of the above mentioned disadvantages.

FEBIE is a direct-write technique where materials can be removed in situ in a single process step with the help of an externally added etch gas [7–10]. This mask-free, resist-free technique gained much attention as subtractive lithography due to its material selectiveness, nanometer precision and direct alignment on the substrate [9,11–13]. FEBIE has already been used to etch Titanium [14], GaAs [15], Silicon [16] and Alumina [17]. However, compared to the additive sibling of this technique-Focused Electron Beam Induced Deposition (FEBID)-very few work has been reported on FEBIE. This is mainly due to the complex pre-requirements which is extremely vital for this process. To perform successful FEBIE, there are certain process conditions which need to be fulfilled: (i) the hydrocarbon contamination level of the microscope chamber must be low [11] (ii) the gas distribution [8] must be controlled to ensure sufficient etch gas supply. Moreover, choice of parameter sets (for example high accuracy vs fast writing) can lead to undesired deposition of carbon instead of the desired etching.

In this work, we will show how a smart parameter choice can avoid the undesired deposition and can lead to a successful etching that will be demonstrated by thinning a Ge channel via chlorine based FEBIE. The developed process can be used to prepare Ge-based single electron transistors or Ge-nanowires in future nanoelectronic devices.

2. Materials and methods

Germanium on Insulator (GeOI) is used as a substrate. The Ge layer was lithographically pre-structured to obtain a 4-point

* Corresponding author.

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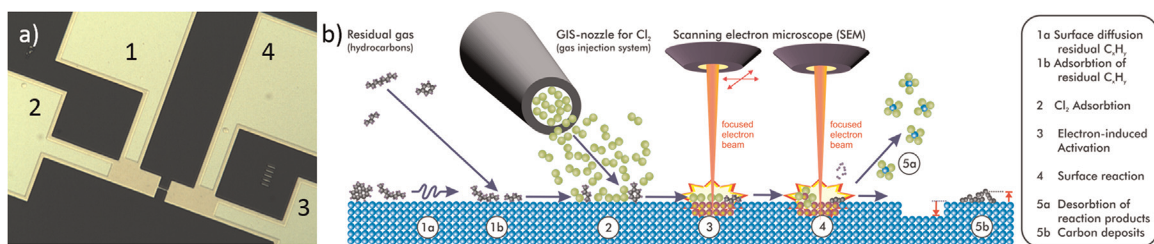

Fig. 1. : (a) Ge channel and four-Ni Contacts made by standard lithography. (b) FEBIE process – (1) adsorption. (2) Diffusion. (3) activation by e-beam. (4) formation of volatile product. (5) Desorption.

Kelvin probe structure. The bulk material was electrically contacted and could be used as back-gate contact. The Kelvin probe structure featured a 4 μm × 20 μm channel (Optical lithography with AZ5214 photoresist, etching with hydrogen peroxide). On the contact pads nickel metal contacts with a thickness of 40 nm was processed to generate highly conductive, ohmic contacts for the tungsten measurement probes used in the electrical measurement. The optical image of the structure is shown in Fig. 1a.

For FEBIE a focused electron beam of a LEO 1530 VP scanning electron microscope (SEM) is used to locally activate the adsorbed chlorine gas molecules. The vacuum system has a base pressure of $\sim 2.8 \times 10^{-6}$ mbar. Chlorine is injected into the chamber via a home built gas injection system (GIS) with a constant chlorine flow of 0.13 sccm resulting in a process pressure of 4.5e-5 mbar in the chamber The electron beam scans over the desired area from where material shall be modified (see Fig. 1b). Etching was performed with 5 kV acceleration voltage with high current mode.

A Dimension 3100 atomic force microscopy (AFM) system from Veeco was used to obtain the height profile of the structures. *I–V* characteristics were obtained using a Karl Suss probe station which is connected to a HP 4156B semiconductor parameter analyzer controlled by labview software.

3. Results

Modification of the monocrystalline germanium of the channel region is intended by chlorination yielding the stable and volatile products germanium dichloride (b.p. 450 °C) and germanium tetrachloride (b.p. 86.5 °C). However, on the surface two electron induced reactions are competing: (i) the chlorination of the germanium and (ii) the decomposition of hydrocarbons adsorbed from residual gas contaminations. While hydrocarbon contamination in former times originated from oil diffusion pumps, the source of residual hydrocarbons in oil-free pumping systems is widely unclear [18] and can only be avoided by oxidative cleaning procedures [11], in an oxygen atmosphere [19] or in UHV systems [20]. With hydrocarbon levels being below the threshold limit of surface analysis techniques [21–22], only the solid deposits could be analyzed and composition varied around 90 at.% C and 10 at.% O [18].

In an experimental approach we investigated the undesired carbon deposition and established an approach to avoid this. The goal was to locally modify the geometry of a Ge-channel. The concept for narrowing a Ge-channel over a 20 μm length in between the contacts is presented in Fig. 1. How carbon contamination can lead to failure in etching and how appropriate choice of parameters results in controlled etching is summarized in Table 1.

As displayed in Fig. 1 the carbon deposition and germanium etching are competing processes. In the first experiment, we demonstrated that inappropriate parameters lead to carbon deposition instead of Germanium removal (Fig. 2). We used a beam current of 1 nA and scanned at a 51 μs pixel dwell time with a 34.6 nm pixel spacing. On each scan area of 20 μm × 2.2 μm, the electron exposure was delivered for a duration of 11 min. As an example a SEM image of such an unsuccessful etching of a channel is shown in Fig. 2a. The 20 μm × 2.2 μm reduced area marked by 2 rectangular areas designated by red dotted lines corresponds to the targeted area where electron beam induced etching was performed. These scan areas have been selected for etching with the electron beam in order to thin the channel region of the Ge device.

Table 1
Parameter selection for promoting either carbon deposition or etching. The used combination of wrong and proper values is given as indicative example. Values depend on the level of residual gas.

	Inappropriate parameters → Result: residual gas **deposition**		**Appropriate parameters →** Result: **etching** of Si or Ge	
Beam current	**Low**	1 nA	**High**	5 nA
Pixel dwell time	**Short**	51 μs	**Long**	204.8 μs
Pixel spacing	**Wide**	34.6 nm	**Narrow**	6.9 nm
Frame refresh time	**Long**	95.6 s	**Short**	3.2 s

The SEM image Fig. 2b shows the Ge channel still intact over the full width but with 2 grey areas in the formerly scanned region. Hence, the channel was not etched, rather deposition of carbon occurred. This can be verified by the significant increase of the height of the structures after the FEBIE process as obtained by the AFM (Fig. 2c). In addition, *I–V* measurement also shows that the conductivity of the Ge-channel changed before and after etching. These results indicated that etching on each side part of the whole channel resulted in an un-desired carbon deposition instead of etching.

If an insufficient electron flux is used, the carbon deposition exceeds the etching removal. A thin carbon layer (seen as darker colored area in the SEM image Fig. 2b) is deposited instead of etching. For the experiment displayed in Fig. 2, the electron dose per scan cycle was so low that assumably the hydrocarbon residuals were never completely consumed so that chlorine etching could outpace the hydrocarbon replenishment – consequently, carbon deposition was the dominant process.

On the other hand, the choice of proper experimental parameters can lead to the successful FEBIE etching of germanium. In the second experiment, we used a beam current of 5.05 nA and scanned at a 204.8 μs pixel dwell time with a 6.9 nm pixel spacing.

An example for successful thinning down of the Ge channel is shown in Fig. 3a. In this case, a smaller reduced area of 1 μm × 0.75 μm applied to the targeted channel is used (Fig. 3a), so that the scan time per area is shorter and hydrocarbons have less time to replenish on the surface. The total etching period was 20 min. The SEM image presented in Fig. 3b shows that etching occurred in the channel. The channel width is reduced from 2 sides as indicated by 2 arrows. In this case the electron flux was sufficient for etching to occur. Chlorine required for etching is

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