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Low-cost top-down zinc oxide nanowire sensors through a highly transferable ion beam etching for healthcare applications

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ABSTRACT

In this work, we demonstrate a wafer-level zinc oxide (ZnO) nanowire fabrication process using ion beam etching and a spacer etch technique. The proposed process can accurately define nanowires without an advanced photolithography and provide a high yield over a 6-inch wafer. The fabricated nanowires are 36 nm wide and 86 nm thick and present excellent transistor characteristics. The pH sensitivity using a liquid gate was found to be 46.5 mV/pH, while the pH sensitivity using a bottom gate showed a sensitivity of 366 mV/pH, which is attributed to the capacitance coupling between the top- and bottom-gates. The maximum process temperature used in the fabrication of the nanowire sensors is optimized to be 200 °C (after wet oxidation) which makes it applicable to low-cost substrates such as glass and plastic. The Ion Beam Etching (IBE) process in this work is shown to be highly transferable and can therefore be directly used to form nanowires of different materials, such as polysilicon and molybdenum disulfide, by only an adjustment of the etch time.

1. Introduction

Over the past decades, Ion Sensitive Field Effect Transistors (ISFETs) have been intensively researched for a wide range of sensing applications, such as ion [1], pH [2], DNA [3] and protein [4] sensing. Silicon-based nanowires have been of particular interest as their large surface-to-volume ratio offers a high sensitivity [4,5]. Nanowires fabricated in a top-down process are the preferred solution for commercialization of the technology by the semiconductor industry due to precise dimension and position control. However, when compared to conventional ISFETs, top-down silicon nanowires require costly e-beam or deep UV lithography and Silicon-on-Insulator (SOI) substrates [6,7]. To significantly reduce the fabrication cost, we have proposed a thin-film technology (TFT) alternative using polysilicon nanowires formed by reactive-ion etching (RIE) through a spacer etch technology [8]. However, the formation of polysilicon and gate dielectrics requires high temperature processes such as silicon recrystallization and thermal oxidation. The high temperature budget makes this process incompatible with low-cost substrates and increases the overall cost. To overcome the temperature constraints we propose the use of zinc oxide (ZnO) as the semiconducting layer and alumina as the gate dielectric.

ZnO has been a popular metal oxide semiconductor in electronics for its low cost, wide bandgap and self-doping characteristics [9,10]. Recently, top-down ZnO nanowires were reported to be fabricated using conventional lithography, atomic layer deposition (ALD) and RIE [11]. However, the transistors of [12] were formed at chip level and cannot be directly transferred to wafer level due to issues such as chemical loading effects and plasma condition changes. The plasma-based chemical etch processes normally require complicated optimizations and are not transferable to other materials and wafer-size changes.

In this work, we report a low temperature zinc oxide nanowire sensor process that uses Ion Beam Etching (IBE) and a spacer etch technique. The proposed IBE process is highly transferable to other materials and can form ZnO nanowires over a 6-inch wafer with a high yield. The maximum temperature used is 200 °C after underlying layer formation and is therefore fully compatible with low-cost substrates, which is of particular interest for point-of-care (PoC) healthcare applications. Our fabricated ZnO nanowire sensors show excellent transistor characteristics in air and high sensitivity to pH measurements. Top-gate sensing sensitivity was measured to be 46.5 mV/pH whereas bottom-gate sensing provided a sensitivity of 366 mV/pH. The results are comparable with those of reported CMOS nanowire sensors [13].

2. Experimental procedure

The ZnO nanowire fabrication process is schematically shown in Fig. 1a–d. The fabrication process started with a 100 nm silicon dioxide layer grown on a 6-inch n-type Si substrate by a wet oxidation and then a 280 nm silicon nitride (Si_3N_4) layer deposited by low pressure chemical vapor deposition (Fig. 1a). Using conventional photolithography

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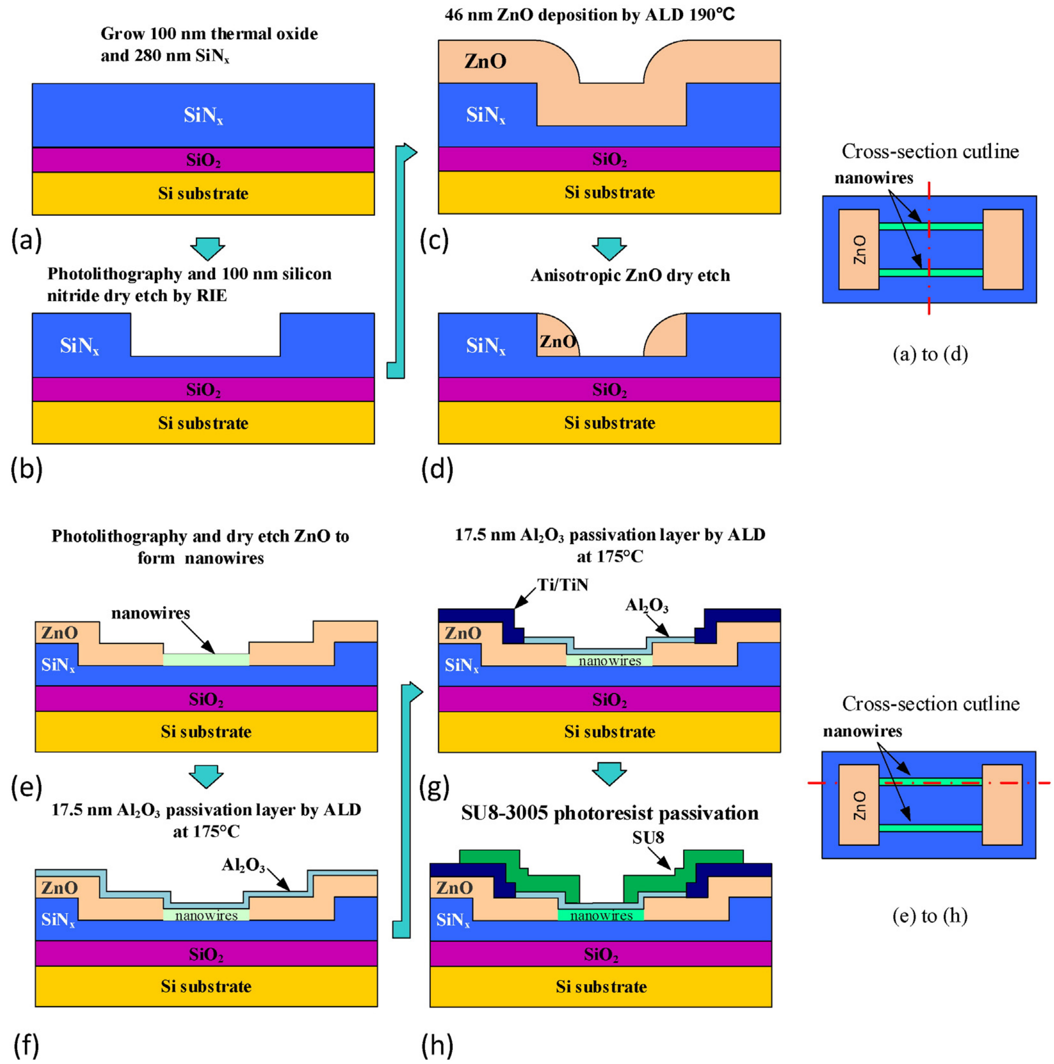

Fig. 1. Schematic of the nanowire sensor fabrication process. Spacer nanowire formation (cross-section in direction perpendicular to nanowires) through dielectric formation (a), step definition by RIE silicon nitride etch (b), ZnO deposition by ALD (c) and ZnO etch by IBE (d). Sensor fabrication (cross-section in direction parallel to nanowires) through ZnO formation (e), Al_2O_3 passivation formation by ALD (f), Al_2O_3 IBE patterning and Ti/TiN electrodes by sputtering and lift-off (g), and SU8 photoresist passivation (h).

and anisotropic RIE etching 100 nm deep steps were defined on the Si_3N_4 layer (Fig. 1b). A 46 nm ZnO layer was subsequently deposited at 190 °C (Fig. 1c) by an Oxford Instrument Plasma Technology (OIPT) FlexAl Atomic Layer Deposition (ALD) system using diethyl zinc (DEZ) precursor (dose time of 50 ms, pressure of 80 mTorr) and oxygen plasma (O_2 flow of 60 sccm, pressure of 15 mTorr and RF power of 100 W) [12]. Subsequently, a spacer etch was done to fully remove the ZnO layer leaving ZnO nanowires along the Si_3N_4 step sidewalls (Fig. 1d). The etching was done in an OIPT Ionfab 300 plus IBE system and the etching conditions were set as argon flows of 5 sccm for the beam and 12 sccm for the neutralizer, beam bias of 500 V and beam current 300 mA, beam acceleration voltage of 400 V, sample tilt angle of 20° and chuck rotation rate of 20 rpm. The IBE was first developed on 20 mm × 20 mm chips where the etch time was varied (1.5, 2 and 2.5 min) and the etching result was characterized by inspections with a Field Emission Scanning Electron Microscope (SEM). Unlike plasma etching processes, the ion beam etching process is a physical process based on ion bombardment and thus is not affected by the size of the area to be etched. This characteristic of IBE makes it possible to directly use a process developed at chip level to etch a full wafer. Therefore, after the optimization of the etching time using the small chips the IBE process was successfully applied to a 6-inch wafer to form the ZnO nanowires.

The fabricated ZnO nanowires can be subsequently used to form transistors/sensors capable of liquid measurements using the process described in Fig. 1e–h. During the IBE, the source and drain areas were protected by photoresist (Fig. 1e). After the etching, a 17.5 nm aluminum oxide (Al_2O_3) layer was deposited on the ZnO as a passivation layer by a thermal ALD process at 200 °C (Fig. 1f) and then partially removed by a time controlled IBE process to open metal/ZnO contact windows. To etch the Al_2O_3 the same IBE process as for the ZnO was used by adjusting the etching time. The etch rate for Al_2O_3 was found to be 5.1 nm/min which is expected for a hard material. The accurate and slow etch rate achieved by the IBE process allowed the full removal of Al_2O_3 over the ZnO layer. The ellipsometry measurements showed that the overetching of Al_2O_3 caused a ZnO etch of about 14 nm leaving enough ZnO to form good contacts with the metal layer. Ti/TiN contacts were then formed by sputtering and lift-off (Fig. 1g). Finally, an SU8 layer was formed using photolithography and then fully cross-linked to function as a passivation layer (Fig. 1h).

Fig. 2a shows a fabricated ZnO nanowire sensor including 100 parallel 40 μm long nanowires with 30 μm wide SU8 sensing windows. The fabricated sensors were first characterized as transistors in a dry environment and then their performance in detecting ionic changes was assessed by pH measurements. Fig. 2b shows the configuration for pH