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Short communication

Effects on micropyramid and nanoneedle structures for superhydrophobicity on Si surface

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ABSTRACT

This study proposes a method for increasing hydrophobicity using a reactive ion etching (RIE) system. The aim was to study and optimize a surface with hierarchical structures. The effects of micropyramid and nanoneedle structures were investigated for improving superhydrophobicity on the Si surface. To investigate the effects of nanoneedle structures for hydrophobicity, RIE parameters such as the gas ratio, gas pressure, and process time were changed. To compare the contact angles with varying parameters, polytetrafluoroethylene (PTFE) was coated to eliminate oxidation effects on the Si surface. The optimized process conditions for the superhydrophobic surface were saw damage removal for 40 min and RIE for 20 min with O_2/SF_6 = 33%/67%. In the optimized process, the contact angles for the micropyramid, PTFE coating on the micropyramid, micropyramid and nanoneedle, and PTFE coating on the micropyramid and nanoneedle samples were 102.8°, 132.5°, 142.8°, and 171.7°, respectively.

1. Introduction

In recent years, many studies have conducted wettability and hydrophobicity experiments. Related technologies have been used in various applications such as sensors, solar cells, auto glasses, building walls, and biomedicine [1–4]. The hydrophobic effect is the observed tendency of nonpolar substances to aggregate in an aqueous solution and exclude water molecules. Many researchers have mimicked hydrophobic surfaces using artificially formed micro-, nano-, and hierarchical structures that reduce the surface energy [1–9]. Among them, Wenzel [5] and Cassie and Baxter [6] demonstrated the theory of the wettability of surfaces. Hierarchical structures can be formed by processes such as lithography, plasma treatment, and chemical vapor deposition (CVD)-based treatment [3,4]. Lithography requires complex equipment, such as nanoimprint lithography, electron beam lithography, and X-ray lithography machines. The use of this process is currently limited to producing nanoscale structures. However, plasma- and CVD-based treatments can easily be used to produce micro- and nanoscale structures and can make a surface superhydrophobic [10–13]. Reactive ion etching (RIE) has also been used for creating a hydrophobic surface [9,14–16]. RIE can make a surface regularly textured with micro-/nanopillars [17–20] or irregularly textured with micro-/nanopyramids (i.e., black Si) with or without an etching mask [9,14–16]. However, because texturing with micro-/nanopillars requires additional processes, in the present study, an irregular texturing process without an etching mask was used.

We developed a double-step surface texturing process with RIE to form needle structures (second step) on micropyramid structures (first step). This process affords advantages such as low cost and simple production procedure. Similar research on a surface texturing process to increase hydrophobicity exists. For example, Kwon et al. [17] demonstrated surface hierarchy with pillars using deep RIE and XeF_2 etching with a maximum contact angle of ~177.2°. Lee et al. [1] showed a similar RIE process with a maximum contact angle of ~161°. These processes can control the sizes of nanostructures using photolithography and RIE etching. However, they require many extra procedures compared to the irregular texturing process (for example, black Si). Nevertheless, as reported previously [21–24], we used a customized RIE system without photolithography masks. This technique can reduce the processing

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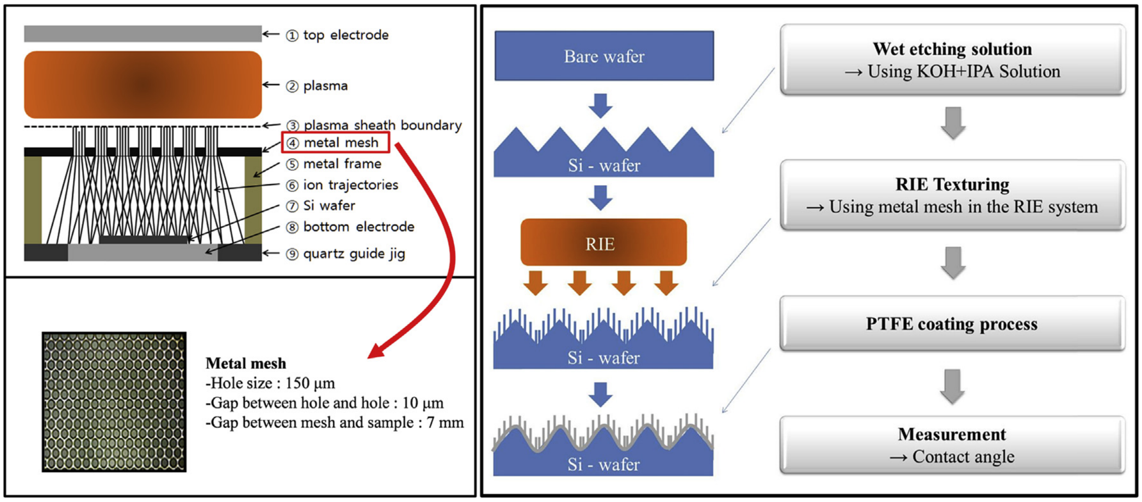

Fig. 1. Concept of customized RIE system and process flow of micropyramid and nanoneedle texturing surface.

Table 1
Process conditions of RIE texturing.

Process variables	Process conditions
O_2 fraction in O_2/SF_6 (%)	0, 25, 33, 40, 46
Pressure (mTorr)	90, 120, 150, 180, 210
Time (min)	5, 10, 20, 30, 40, 50
RF power (W)	100
Temperature (°C)	10

time and fabrication cost. In this study, we aim to investigate and optimize a surface with hierarchical structures. Our previous work [22] focused on the effects on the nanostructures and polytetrafluoroethylene (PTFE) thickness as parameters influencing hydrophobicity. However, in this study, we focused on the size of micropyramid and nanoneedle structures as parameters influencing hydrophobicity. We investigated the relation between the size of micro- and nanostructures and the contact angle with changing RIE pressure, time, and O_2 gas fraction in O_2/SF_6.

To improve hydrophobicity, our study focused on the size of nanoneedle structures on micropyramid structures. The micropyramid structure was fabricated using potassium hydroxide (KOH) and isopropyl alcohol (IPA). The needle textured structure was fabricated using RIE. After fabrication, PTFE was coated on the structures. A scanning electron microscope (SEM) was used to verify the difference in the pyramid-needle textured structure, and the contact angle was measured to check the hydrophobicity after the process. The process was performed with the wet etching of bare Si, RIE texturing, and PTFE coating, as shown in Fig. 1.

2. Experimental

2.1. Micropyramid structures

To form a micropyramid on the Si surface, we used a bare wafer for the solar cell in the fabrication of the pyramid structure. The structure comprised p-type (100) single-crystal Si wafers with an area of 156 × 156 mm^2, thickness of 200 µm, and resistivity of 0.5–3 Ω cm [21–24]. Thus, we could easily fabricate micropyramid structures on the surface by the wet etching process. The side of the bare Si substrate was cut by an initial saw cut. A saw damage removal (SDR) process was conducted to remove the damage incurred during the saw cut process on the surface. The SDR process, which formed an anisotropic etching profile on a silicon wafer, used a KOH (45%) + IPA + D.I. mixture. The process was performed for 10, 20, 30, and 40 min, respectively, to observe the size change in the micropyramid and contact angle transition depending on changes in the pyramid size.

2.2. Nanoneedle structures

To investigate the effect of the size of the nanoneedle structures, we varied the O_2/SF_6 gas ratio from 0% to 50% and the process pressure from 90 to 210 mTorr. Table 1 shows the process time.

As shown in Fig. 1, a customized RIE texturing system has a metal mesh, which is isolated to the bottom electrode (ground electrode) with a quartz jig [21–24]. RIE is a simple and economical approach for general plasma etching. The substrate is usually placed on a quartz or graphite coverplate to avoid sputtering/redepositioning of electrode material. Gas is injected into a process chamber via a showerhead gas inlet in the top electrode [21]. Negative self-bias forms on the lower electrode, and a single RF plasma source determines both ion density and energy [21,24]. The use of the metal mesh results in the formation of a remote plasma sheath boundary and causes ion trajectory bending. As a result, a nanoneedle structure is formed on the silicon surface [21]. A metal mesh is placed inside the RIE system to function as a shadow mask and for ion trajectory bending [21,24]. The ions are accelerated and scattered through the holes on the metal mesh placed between the sample and the plasma. The plasma ions collide with the sample. The RIE system is designed in this manner to enhance the scattering of the plasma ions [21,24]. The important parameters that enable the formation of nanoneedles are RF power, process pressure, and SF_6/O_2 ratio. In this study, the Si wafer surface was textured using only the SF_6/O_2 gas in the RIE chamber.

2.3. PTFE coating

In terms of the Si surface, the physically treated surface rapidly oxidizes at room temperature. It can therefore easily lower the hydrophobicity. Accordingly, the PTFE coating treatment is needed to further increase the surface hydrophobicity as soon as RIE etching is performed. The PTFE thin film is evaporated through high-frequency magnetron sputtering, stirred for 2 h using Solvay's F7-type PTFE powder ball mill, and then sintered for 2 h in air at 300 °C. This sintered powder is compressed into a disk-form pellet