FLUOROCARBON FILMS DEPOSITED BY DEEP REACTIVE ION ETCHING FOR STICION MINIMIZATION OF MEMS STRUCTURES

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ABSTRACT
Fluorocarbon films, which can be used to minimize stiction of silicon microstructures, have been deposited by passivation process in deep reactive ion etching tool. The wettability, surface energy, nano-scale adhesive force, and thermal stability have been investigated by contact angle measuring system, atomic force microscopy (AFM) and ellipsometry. The fluorocarbon films are good for anti-stiction applications due to their high water contact angle (110°), low surface energy (14.5mJ/m²), low nano-scale adhesive force (33 nN) and high thermal stability up to 300°C.

Key words: Fluorocarbon film, wettability, adhesive force, anti-stiction coating, thermal stability, MEMS.

INTRODUCTION
Stiction is a serious problem in Microelectromechanical systems (MEMS) due to large surface area-to-volume ratio, which affects reliability, long-term stability, and yields of MEMS devices [1]. Although polymers have been increasingly used in bio-MEMS, micro fluidic devices, and various sensors, silicon and silicon-related materials are still widely used in MEMS devices due to their mature fabrication techniques and unique properties. However, silicon and silicon-related materials have low water contact angles, and thereby higher surface energies. They have quite serious stiction problems during fabrication (release stiction) and/or in applications (in-use stiction). Special drying processes such as CO₂ drying can eliminate release stiction in some situations, but in many processes and applications, this is not feasible. In-use stiction related failure is a major problem and will be increasingly important with miniaturization towards nano-scale structures. One efficient way to minimize stiction is to coat the surfaces with low surface energy materials. Fluorocarbon film coatings are good candidates for anti-stiction applications. Here, the fluorocarbon films are deposited by passivation process in deep reactive ion etcher (DRIE) developed by Surface Technology Systems (STS) and studied with regards to contact angle, wettability, surface energy, adhesive force, and thermal stability.

EXPERIMENTAL TECHNIQUES
Fluorocarbon films were deposited on silicon substrates using passivation process in DRIE where C₃F₈ is used as a feed gas. Before deposition, the silicon substrates were given a buffered hydrofluoric acid (BHF) dip followed by 5 min water rinse and spin dry afterwards. The deposition parameters, which were varied in DRIE, are C₃F₈ gas flow, coil power, and process time. In this investigation, the deposition temperature is 20°C, the Bias power is 20 W and base pressure is 0.1 mTorr. Thicknesses of the fluorocarbon films were measured by variable angle spectroscopic ellipsometry. A three layer model, i.e. (substrate)/(isotropic Cauchy film)/(air), is used to fit the data to get the thickness and refractive index. Contact angle measurements were carried out using contact angle meter DSA10 from Krüss GmbH equipped with automatic dispensing system and Frame grabber. The contact angles were determined by drop shape analysis software. Test liquids are deionised (DI) water, dioxomethane, ethylene glycol and n-Hexadecane. Both static and dynamic contact angle were measured. The detailed description of measurement and calculation of surface energy can be found in Ref. 2. The nano-scale adhesive force tests were performed with a commercial AFM system (Dimension 3100, Digital Instrument) operating under ambient conditions of 22°C and 45-50% RH. Square pyramidal Si₃N₄ with a nominal 20-60 nm radius mounted on gold-coated Si₃N₄ cantilevers with a nominal spring constant of 0.32N/m (Digital Instrument) were used in this study. The adhesive force measurements were carried out in force calibration mode [3]. Three samples with various initial thicknesses of 3.6, 15.3 and 195.2 nm were annealed at a given temperature in an oven in air for 10 minutes, taken out from the oven and cooled down to room temperature. Then static contact angles of four test liquids and thickness of films are measured at room temperature to evaluate the thermal stability of the fluorocarbon films. The accuracy of the annealing temperatures is ±5 °C.

RESULTS AND DISCUSSIONS
The fluorocarbon films are deposited on single silicon substrates by passivation process in DRIE tool. The results
show that the deposition rate reaches maximum at C\textsubscript{3}F\textsubscript{8} gas flow rate of 120 sccm when the coil power is 300 W, and deposition rate linearly increases with the coil power in the range of 100-600 W at C\textsubscript{3}F\textsubscript{8} gas flow of 120 sccm. The contact angle measurements and surface energy calculation display similar results for all the fluorocarbon films deposited on single silicon substrates, and those on poly-silicon substrates and low stress silicon nitride substrates. The films have the static water contact angle of 110±2 degrees, surface energy of 14.5±0.5 mJ/m\(^2\), and near zero polar component of surface energy. Fig.1 compares the static water contact angle and surface energy of the fluorocarbon films with other commonly used MEMS materials. It can be seen that the fluorocarbon films have highest water contact angle and lowest surface energy. The nano-scale adhesive force between Si\textsubscript{3}N\textsubscript{4} AFM tip and the fluorocarbon film is 33 nN, while the adhesive force of uncoated silicon wafer with native oxide is 148 nN, which are 4.5 times larger than that of the fluorocarbon films. It can be concluded that the static and adhesion of silicon microstructures can be greatly reduced by depositing fluorocarbon films on them due to decreased surface energy.

Fig. 2 shows effect of the annealing temperatures on the static water contact angles and surface energies of some commonly used MEMS materials and the fluorocarbon films. Similar trends have been found for the other two fluorocarbon films with initial thickness of 3.6 and 195.2 nm, respectively.

Fig. 3 displays the static water contact angles (right axis) and the thickness (left axis) at various annealing temperatures for the three selected films. The degradation temperature, from which the water static contact angle decrease, depends on the initial thickness, i.e. 300°C for the thinnest film, 330°C for the film with 15.4 nm thickness, and 350°C for the film with initial thickness of 195.2 nm. However, the thicknesses of the annealed films decrease even at 100°C without decreasing static water contact angle. When annealing temperature is higher than 300°C, the thickness dramatically decreases, and the static water contact angle decreases quickly with annealing temperature, meaning that the decomposition mechanism at higher temperature might be different from that at low temperature (less than 300°C). It can also be found from Fig.3 that there is critical thickness for the fluorocarbon films, beyond which the fluorocarbon film has higher water contact angle and good anti-stiction ability. For H-terminated substrates produced by BHF dip, the critical thickness is around 3 nm.

CONCLUSIONS

Fluorocarbon films have been deposited onto silicon substrates using passivation process in deep reactive ion etcher. It has been shown that the fluorocarbon films have high water contact angle of 110 degrees, low surface energy of 14.5 mJ/m\(^2\) and 4.5 times smaller adhesive force than uncoated silicon wafer with native oxide. The fluorocarbon films are quite stable for anti-stiction application up to 300°C. There is critical thickness for the fluorocarbon films, beyond which the fluorocarbon film has higher water contact angle and good anti-stiction ability.

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