Conformal Coating Process of Anti-sticking Thin Film Using C_4F_8 and Ar Plasma without Additional Equipment

Changho Cho, Woonbae Kim, and Hyungjae Shin
Samsung Electronics Co., Ltd.
416 Mantan-3 Dong, Paldal-Gu, Suwon, Kyungki-Do, Korea
jancho@samsung.co.kr

Summary

The relatively conformal fluorocarbon deposition process on aluminum surface was developed to eliminate the sticking problem in MEMS applications such as micromirror. The process was carried out in a capacitively-coupled plasma reactor with octafluorocyclobutane (C_4F_8) gas. The process parameters were optimized for maximum conformal quality. Dynamic contact angle measurement and AFM/LFM analysis on deposited film showed low surface free energy and homogeneous coverage resulting in high hydrophobic characteristics. Ellipsometry analysis and reflectance measurement revealed high enough transparency to be used in optical applications.

Keywords: fluorocarbon, conformal, transparent

Introduction

Fluorocarbon films have been preferred as an effective methods to prevent stiction phenomena and/or to reduce friction, because of its excellent hydrophobic property as well as high mechanical and electric strength [1~6]. Several coating processes of fluorocarbon films have been studied, and the simplest way is to deposit thermally using evaporated solid precursor [1~4]. Although this method provides hydrophobic film with desirable properties, it requires extremely clean substrate and long process time. Coating processes by plasma polymerization have been explored by many researchers [5~6] because of its fast deposition rate and relative insensitivity on substrate condition. These works, however, were mainly focused on the hydrophobic coatings for silicon or silicon dioxide substrates, not for metallic surfaces.

In MEMS applications, contact usually occurs between the suspended member and underneath substrate. Current plasma assisted deposition studies, however, are mostly limited to flat open situations. In a case of achieving conformal coating process [5], the deposition rate was as low as 50*nm/hour*, and it was possible by forming the field free zone with special setup like Faraday cage.

In this paper, relatively conformal fluorocarbon coating process for aluminum surface is presented, which is carried out in a plain capacitively-coupled plasma reactor only by optimizing process parameters. Deposited film is then characterized to show its hydrophobic and optical properties to be used in optical MEMS applications.

Experiments

Apparatus shown in Fig. 1 was used in this experiment. The temperature of substrate, reactor walls, and gas lines could be controlled independently. C_4F_8 gas was used for supplying reactants and argon gas was added for the stabilization of plasma and activating surfaces.

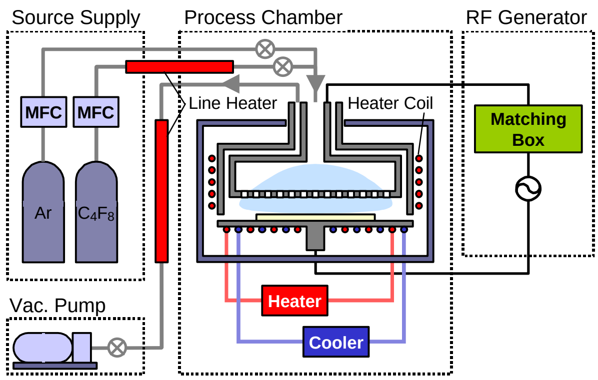

***Fig. 1**: Diagram of experimental apparatus.*

All samples were cleaned in O_2 plasma before the process, and annealed at 125^o*C* in air for two minutes after coating. Three types of samples were used to test the coating process. Aluminum and silicon were selected to determine the process could be applied on various surfaces. Suspended aluminum cantilever beams shown in Fig. 2 were utilized to examine the conformal quality (CQ). The gap between beam and electrode was 3.5*mm* and the spacing between the beams 5*mm*. The widths of the beam were two kinds as 5*mm* and 10*mm*. The thickness ratio (T_{under}/T_{over}) of the film under the beam to over the beam was used to define CQ. T_{over} was measured by an ellipsometer and an optical profiler

(Vision 32, WYKO) was used to calculate T_{under}. Before measuring thickness profiles, the cantilever beams were removed and 30*nm* of aluminum was then sputtered to avoid measurement errors of the optical profiler.

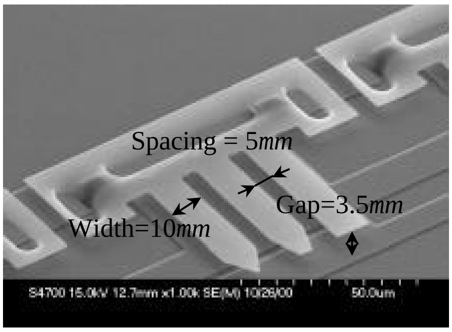

Fig. 2: *SEM image of cantilever beams.*

The process parameters, such as process pressure, RF power, partial pressure of C_4F_8, and substrate temperature, were varied to attain the conformal process. Fig. 3 shows the main effects of the process parameters. RF Power was one of the major effects. As the power decreases, reactants reach under the beam easily because the self-bias of the plasma decreases. The increase of CQ as the pressure increases could be explained in the similar way. The increase of C_4F_8 partial pressure weakens the effect of argon plasma that activates the surface and has the directionality. However, when the process pressure or C_4F_8 partial pressure was too high, the plasma could not be ignited or CQ decreased.

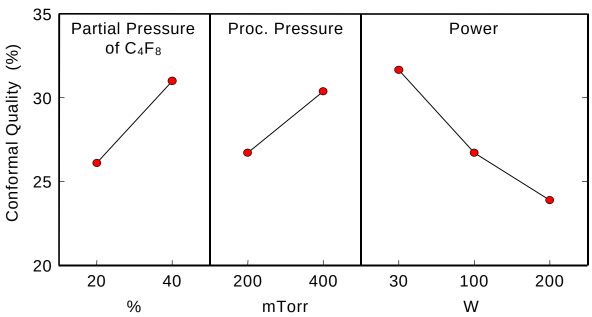

Fig. 3: *Main effects of process parameters.*

Fig. 4 illustrates the influence of the substrate temperature to CQ. The deposition rate the process is inversely proportional to the substrate temperature and proportional to surface activation. Thus the deposition rate under the cantilever beams increases faster than over the beams as the temperature decreases.

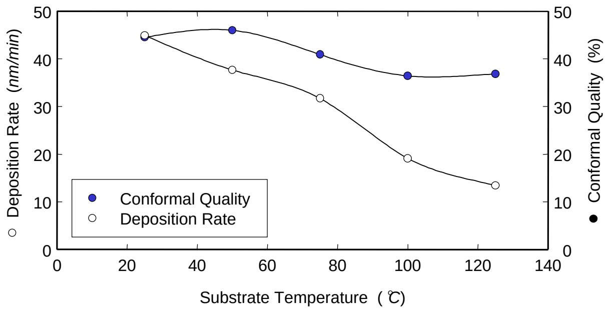

Fig. 4: *Variation of conformal quality and deposition rate with respect to substrate temperature.*

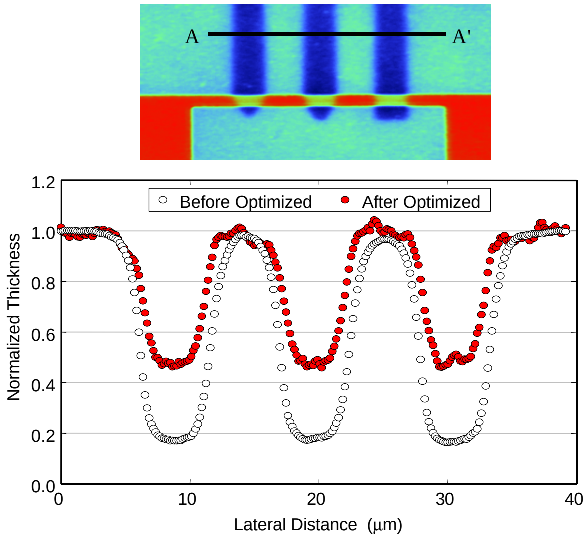

Fig. 5: *Optically profiled image and comparison of line scanned data upon optimization.*

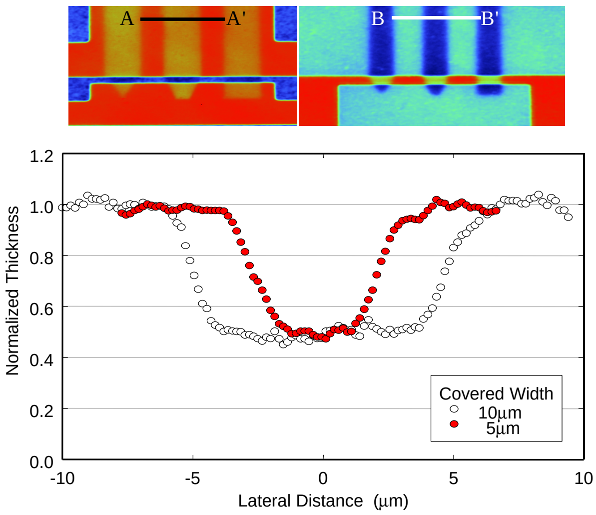

Fig. 6: *Optically profiled image and comparison of line scanned data between different beam widths.*

After the optimization experiments, CQ was improved from 0.19 to above 0.45 as shown in Fig. 5. CQ in beam width of 5*mm* was 0.47 and that of 10*mm* was 0.46, Fig. 6. This implies that the developed process could be used in other applications such as a wider micromirror or a plate. The deposition rate of the optimized process was about 30 *nm/min*, which is about 36 times higher than that of the process with a Faraday cage [5].

Characterization and Analysis

The deposited film with optimized process was analyzed for its hydrophobicity, homogeneity, and optical characteristics.

Hydrophobicity

The static contact angles of the developed films on aluminum and silicon were 119.7^o and 116.2^o, respectively. These values is higher than previous works [2~6], and revealed that it is highly hydrophobic and the process is applicable to the various surfaces.

Surface free energies of the films were calculated based on the Lifshitz-van der Waals and Lewis acid-base theories, and were 6.06*dynes/cm* and 6.50*dynes/cm* on aluminum and silicon, respectively, as shown in Table 1. These values were lower than that of Teflon, 18*dynes/cm*, and comparable to the critical surface tension of CF_3 functional group, 6.0*dynes/cm* [7]. These results demonstrated that the films might be terminated with CF_3 group. The composition of the deposited film was analyzed by X-ray photoelectron spectroscopy (XPS) and the result showed the CF_2 and CF_3 were two dominant functional groups, Fig. 7.

Table 1: *Contact angles and surface energy.*

Surface	Contact Angle(^{o})			Surface Energy (dynes/cm)
	Water	Formamide	Diiodo-methane	
Al	119.7	109.6	108.0	6.06
Si	116.2	111.3	106.7	6.50

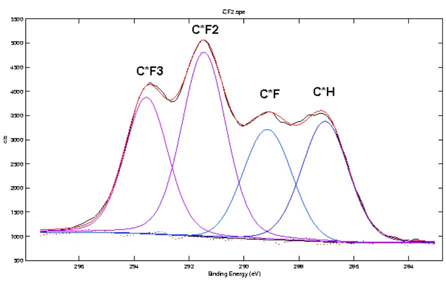

Fig. 7: *XPS C1s spectra.*

Homogeneity

The static contact angle is not sufficient to understand the surface characteristics of thin films. The dynamic contact angles and the hysteresis of contact angle provide not only the hydrophobicity but also other properties such as homogeneity, surface polarity, surface roughness, etc [8]. Small hysteresis indicates that the film is homogeneous and its roughness is low. The developed film exhibited much lower hysteresis than PFDA of previous work [4] and solid Teflon (PTFE) as shown in Table 2 and Fig. 8.

Table 2: *Contact angle hystereses (DH) of fluorocarbon film deposited on aluminum and silicon.*

Property		On Al	On Si
Dynamic Contact Angle	Advancing (q_a)	122.2^o	122.1^o
	Receding (q_r)	107.5^o	109.0^o
Hysteresis (*DH*)		14.7^o	13.1^o

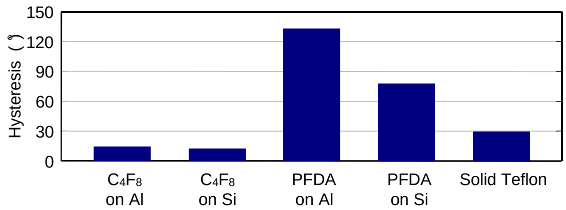

Fig. 8: *Contact angle hystereses of various surfaces.*

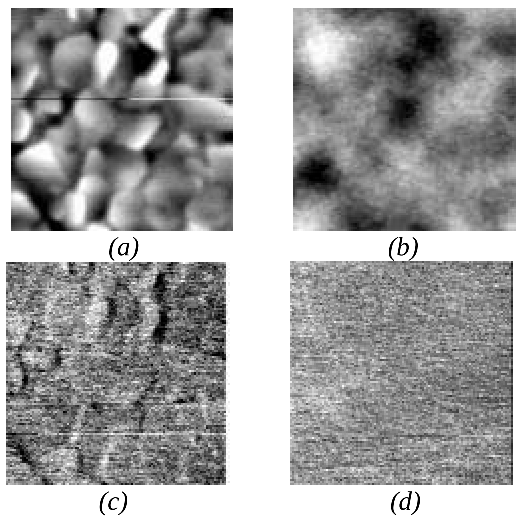

Fig. 9: *The AFM/LFM images (a) AFM image before coating, (b) AFM image after coating, (c) LFM image before coating, and (d) LFM image after coating.*

The homogeneity of the film was confirmed by the AFM/LFM. The RMS roughness was reduced from 2.67*nm* to 1.16*nm* after the coating process, the relative frictional force of the deposited surface is 57.9% of that of pure aluminum surface, and any island of sphere shape reported in [4] could not be found as shown in Fig. 9.

Optical Characteristics

From the ellipsometer measurement, the refractive index (n) of the film was 1.29 at 550*nm* and extinction index (k) was zero from 200*nm* to 900*nm*. Essential Macleod+ simulation with these values for 30*nm* thick film resulted in 1.76% loss of reflection at 550nm. This simulation result was confirmed by spectrometer measurement as 1.67%. Actual measurement results over the visible wavelength are shown in Fig. 10.

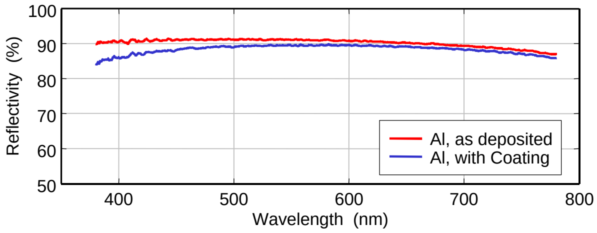

***Fig. 10:** Reflectance reduction after coating process*

The characteristics of the film coated in the optimal process are summarized in Table 3.

***Table 3:** Characteristics of fluorocarbon film coated in the developed process*

Property	**Value**
Surface energy	6.06*dynes/cm*
Static contact angle of water	119.7^o
Contact angle hysteresis	14.7^o
Reduction in reflectance (at 30*nm*)	1.67 %
Deposition rate	30*nm/min*
Conformal quality	> 0.45

Conclusion

Fluorocarbon deposition process on aluminum surface is developed for optical MEMS application. C_4F_8 was employed as a precursor and a plain plasma reactor was utilized for plasma polymerization. Process parameters were optimized to maximize the conformal quality. As a result, the conformal quality is improved by more than two-fold, even though more work on generalization of the term "CQ" still needs to be carried out.

With the optimized process parameters, the deposition rate is within reasonable range. Resulting film exhibits high hydrophobic properties with excellent coverage, and is relatively insensitive to substrate materials. At applicable thickness of 30nm, the film is transparent enough to be used in optical application.

Acknowledgments

The authors would like to gratefully acknowledge Professor Jin-Goo Park at Hanyang University for his extensive and endless advice, and colleagues at Opto-mechatronics Lab. for their help on preparing cantilever array specimen.

References

[1] S.A. Henck, "Lubrication of digital micromirror devices^{TM}", *Tribology letters*, Vol. **3**, pp. 239~247, 1997.

[2] S.H. Lee et al, "Preparation and characterization of perfluoro-organic films on aluminum surfaces", *Surface and Coating Technology*, Vol. **112**, pp. 48~51, 1999.

[3] K.K. Lee et al, "Optical and tribological characterization of perfluoropolymer films as an anti-stiction layer in micromirror arrays." *Thin solid films*, Vol. **377~378**, pp. 727~732, 2000.

[4] J.S. Kim et al, "Characterization of fluorocarbon thin films by contact angle measurements and AFM/LFM", Journal of Microelectronics & Packaging Society, Vol. **7**, pp.35~40, 2000

[5] P.F. Man et al, "Elimination of post-release adhesion in microstructures using conformal fluorocarbon coatings", *Journal of MicroElectroMechanical Systems*, Vol. **6**, pp. 25~34, 1997

[6] Y. Matsumoto and M. Ishida, "The property of plasma-polymerized fluorocarbon film in relation to CH_4/C_4F_8 ratio and substrate temperature", *Sensors and Actuators*, **83**, pp.179~185, 2000.

[7] R.E. Johnson, R.H. Dettre, in J.C. Berg (ed.) *Wettability*, Marcel Decker Inc., New York, p.2-p.73, 1993

[8] J. –H. Wang et al, "Dynamic contact angles and contact angle hysteresis of plasma polymers", *Langmuir*, Vol. **10**, pp.3387~3897, 1994