

## Wettability Analysis on Platinum Deposited Wafer After Reactive Ion Etching Using SF<sub>6</sub>+Argon/CF<sub>4</sub>+Argon Gaseous

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ARTICLE INFO	ABSTRACT
<p><b>Article history:</b> Received 11 September 2013 Received in revised form 21 November 2013 Accepted 25 November 2013 Available online 5 December 2013</p> <p><b>Key words:</b> Wetability; Contact angle; Platinum; RIE; DOE</p>	<p>Wettability in microfluidic has direct influence to its fluid flow channels. This paper investigates the variable parameters that affect the wetability in terms of contact angle on a Platinum deposited wafer after reactive ion etching (RIE). A total of four controllable process variables, with 16 sets of experiments were scrutinized using a designed design of experiment (DOE). The four variables in the investigation are ICP power, Bias power, working pressure, and type of gaseous used. The result suggests that the type of gaseous is the most significant effect contributing to the contact angle values where SF<sub>6</sub>+Ar gives higher values of contact angle compared to CF<sub>4</sub>+Ar gas. All the experiments produced the contact angle greater than 90° and are included in hydrophilic category.</p>

### INTRODUCTION

Rapid growth within the microfluidic discipline has been seen in recent years. Microfluidic includes a vast field of applications including life sciences, industry, agriculture, and pharmaceuticals [1]. Unlike conventional equipment, the advancement of miniaturized devices to process and control diminutive volume of fluids has many advantages such as rapid and repeatable analysis [2], reaction time is shortened, more effective, and accurate by the increased surface-to-volume ratio, as well as reducing error rates by being more consistent during channels measurement [3]. In microfluidic, wettability has direct influence to its fluid flow channels. Generally, the most basic measure of wettability for a particular liquid/solid combination is the contact angle. As the contact angle changes, the device could be either in a hydrophobicity or hydrophilicity state [4,5]. There were many researches done on the wettability characterization in microfluidic field. Fu and his co-workers grafted PNIPAAm onto porous anodic aluminum oxide. The results from the nanostructured surfaces displayed changes in wettability and surface roughness at the same time by alternating the temperature [6]. Adam R. Abate, Julian Thiele, Marie Weinhart, and David A. Weiz used flow confinement as an approach to spatially pattern microfluidic devices surface properties. Surface patterning with micron-scale resolution can be achieved using the method introduced. Furthermore, W/O/W and O/W/O double emulsions were generated using pattern wettability to reflect its efficiency [7]. To the best of the author's knowledge, there are minimal studies been done towards the relationships between the process parameters and the wettability effect, hence this paper studies the relationship between controllable process parameters and wetability of the etched Platinum metallization layer after Reactive Ion Etching (RIE). The reason that Platinum metallization is chosen is because of its high thermal coefficient resistance and inert characteristic to oxygen. Thus, it will help to overcome the corrosion problem that occurs in a device and withstand high operating temperature [8].

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**Methodology:**

A design matrix was designed using full factorial design of experiment (DOE) with four factors and one response. The set of experimental combinations systematically study the variables that influence the quality of the surface roughness. DOE enables quick perception of the process or system performance over a specified region of interest [9]. Table 1 shows the list of variables in the experiment and the values for the low and high levels.

**Table 1:** Process Variables

Process variables	(-1)	(+1)
Gaseous	CF <sub>4</sub> +Ar/ Sccm	SF <sub>6</sub> +Ar/ Sccm
ICP Power /W	400	800
Bias Power/W	140	160
Working Pressure/ mTorr	0.5	1.5

A total of 16 experiments were carried out in this study. Different types of gaseous, varied values of ICP power, bias power and also working pressure were chosen as the experiment parameters while the response will be the contact angle values. Next, water droplet test were performed on the samples to calculate the contact angles. The test samples used in this investigation were prepared from blank wafers. The silicon wafer was first diced into smaller samples for experimental purpose. Next, cleaning process using Piranha solution. Piranha was chosen because it is a strong oxidizer and has the ability to remove most organic matter especially oxide. Approximately 200 Angstrom thickness of Platinum was deposited onto the active side of the wafer by sputtering. Next, the samples were ready for etching process using ICP-RIE. Reactive Ion Etching (RIE) is a technique for removing material from the wafer surface with both a reactive chemical process and a physical process using ion bombardment. For the purpose of removing Platinum, two types of gaseous are used which are CF<sub>4</sub>+Argon and SF<sub>6</sub>+Argon. Pressure can affect plasma uniformity and cause the etch rate or deposition rate across a wafer to change. Moreover, ion bombardment is a very important property of plasma.

**RESULT AND DISCUSSION**

Wettability is a study of how a liquid spreads out when a liquid is droplets on it. When a drop is placed on the sample and it spreads completely, the sample surface is called hydrophilic surface and this phenomena is called total wetting [9]. When a drop is place on the sample and it remain stuck in its place, the sample surface is called hydrophobic and this phenomena is called partial wetting. The wettability of a sample surface can be determined by calculate the contact angle of a liquid that droplet resting on a solid surface.

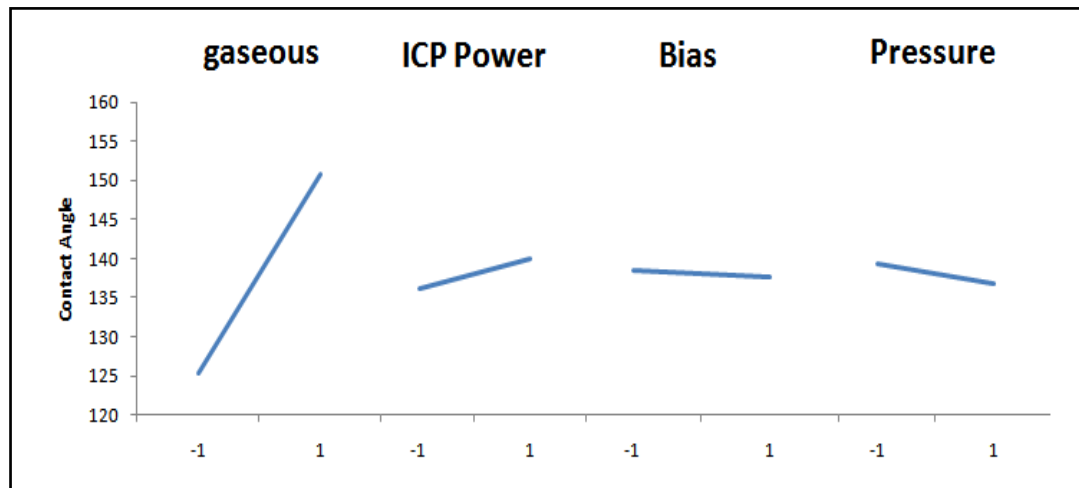
It was necessary to construct the coded design matrix as shown in Table 2 in order to compute the main effects. Main effect is calculated using the following equation [10]:

Main effect of a factors = average surface roughness at high level- average surface roughness at low level (1)

**Table 2:** The Design Of Experiment Of Rie And The Surface Roughness Result

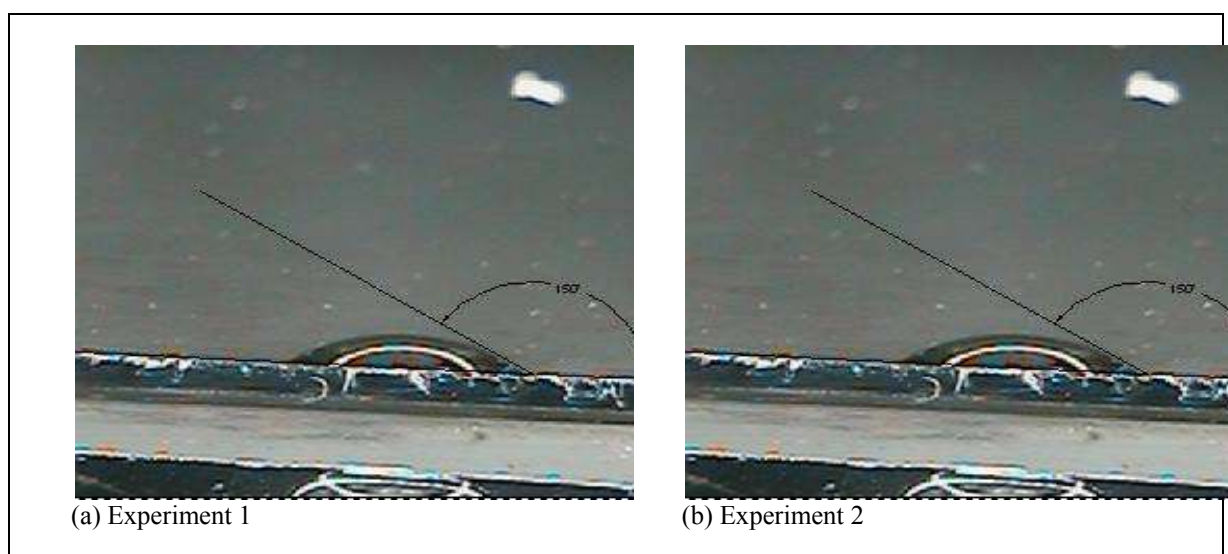
Trial No.	Gaseous	ICP Power	Bias	Pressure	Contact angle (°)
1	-1	-1	-1	-1	150
2	-1	-1	-1	+1	118
3	-1	-1	+1	-1	111
4	-1	-1	+1	+1	145
5	-1	+1	-1	-1	118
6	-1	+1	-1	+1	114
7	-1	+1	+1	-1	112
8	-1	+1	+1	+1	135
9	+1	-1	-1	-1	134
10	+1	-1	-1	+1	141
11	+1	-1	+1	-1	158
12	+1	-1	+1	+1	133
13	+1	+1	-1	-1	170
14	+1	+1	-1	+1	163
15	+1	+1	+1	-1	162
16	+1	+1	+1	+1	145

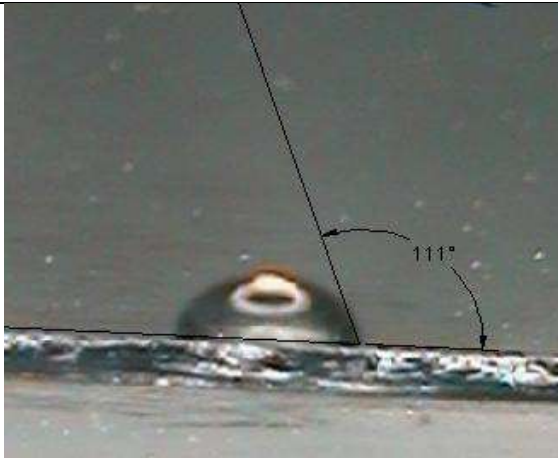
The estimate of the effect calculated for gaseous, ICP Power, Bias, and Pressure are 25.375, 3.625, -0.875, and -2.625 respectively. An effect is negative when the average contact angle is high at a low level of the factor. The significance of an effect depends on the gradient of the graph. Steeper slope suggests that the factor is more influential.



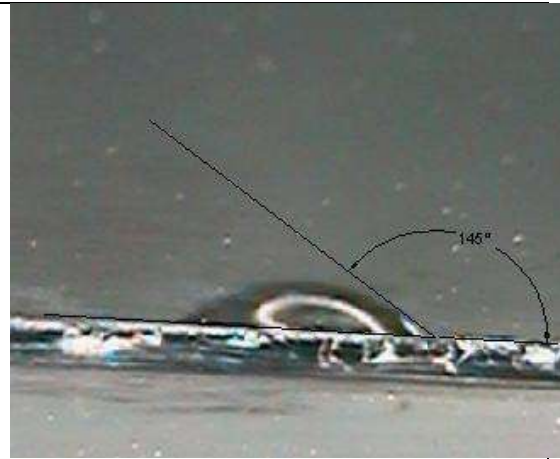
**Fig. 1:** The main effects plot for contact angle

From the graphs in Fig. 1, the type of gaseous is the most significant factor in relationship to the contact angle because the gradient of the graph connecting the low and high level of gaseous has the highest gradient.  $\text{SF}_6+\text{Ar}$  results in larger values of contact angle compared to  $\text{CF}_4+\text{Ar}$  gas. In RIE process, etching mode contains ion-bombardment and chemical etching. Ar can be added to increase the effect of ion-bombardment. Etching rate is limited by the ratio of F radical and  $\text{SiF}_x(\text{gas})$ , and the mean free path of F radical. When the  $\text{SF}_6$  flow rate is getting larger, etching rate is increased due to the higher concentration of F radical. However, too much F radical may dilute the  $\text{SiF}_x(\text{gas})$  concentration and decrease the mean free path of F radical, so that the diffusion control will dominate the etching process to reduce the etching rate. For ICP Power, it has a positive effect whereby contact angle is high when the ICP is high but the effect is not significant compared to gaseous. On the other hand, bias power and working pressure are both inversely proportional to the contact angle values. Fig. 2 displayed the contact angle measurements for all 16 experiments. Fig. 2 (a) represents the contact angle for the first experiment while Fig 2 (p) show the wettability from the droplet test for experiment 16. All measurements were taken using the AutoCAD software. A contact angle of  $90^\circ$  or greater generally characterizes a surface as wettable, and one less than  $90^\circ$  as non-wettable. In the context of water, a wettable surface may also be termed as hydrophilic and a non-wettable surface as hydrophobic. Wetting is also important in the bonding or adherence of two materials. Wetting and the surface forces that control wetting are also responsible for other related effects, including so-called capillary effects. All the experiments produced the contact angle larger than  $90^\circ$  and are included in the hydrophilic category.





(c) Experiment 3



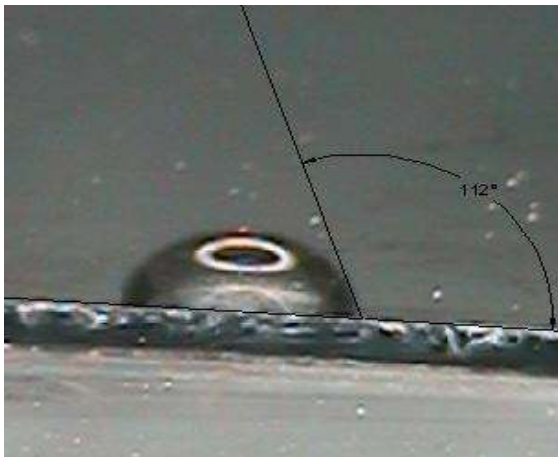
(d) Experiment 4



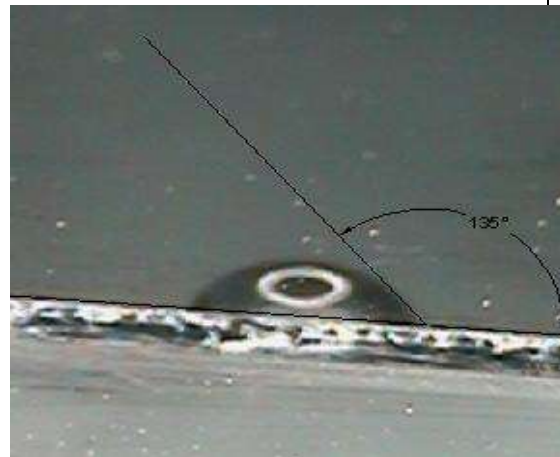
(e) Experiment 5



(f) Experiment 6



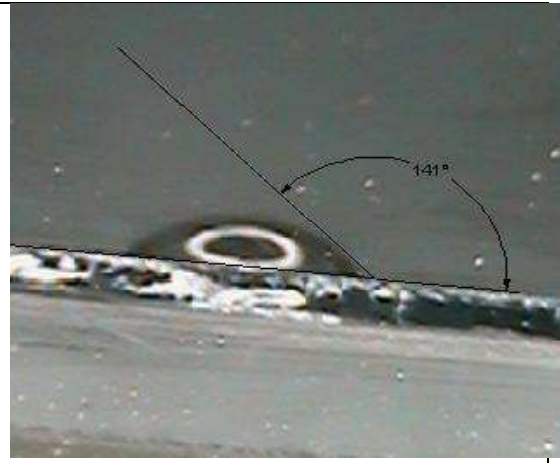
(g) Experiment 7



(h) Experiment 8



(i) Experiment 9



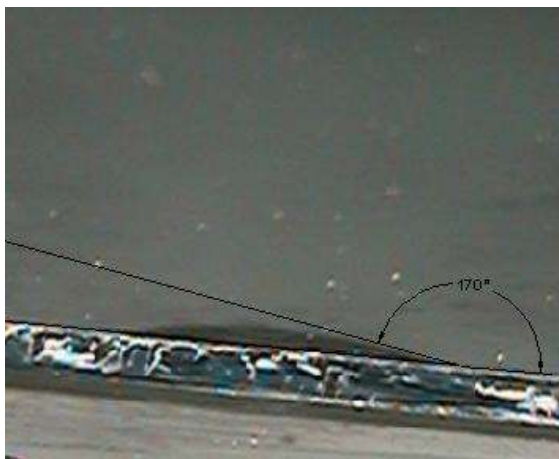
(j) Experiment 10



(k) Experiment 11



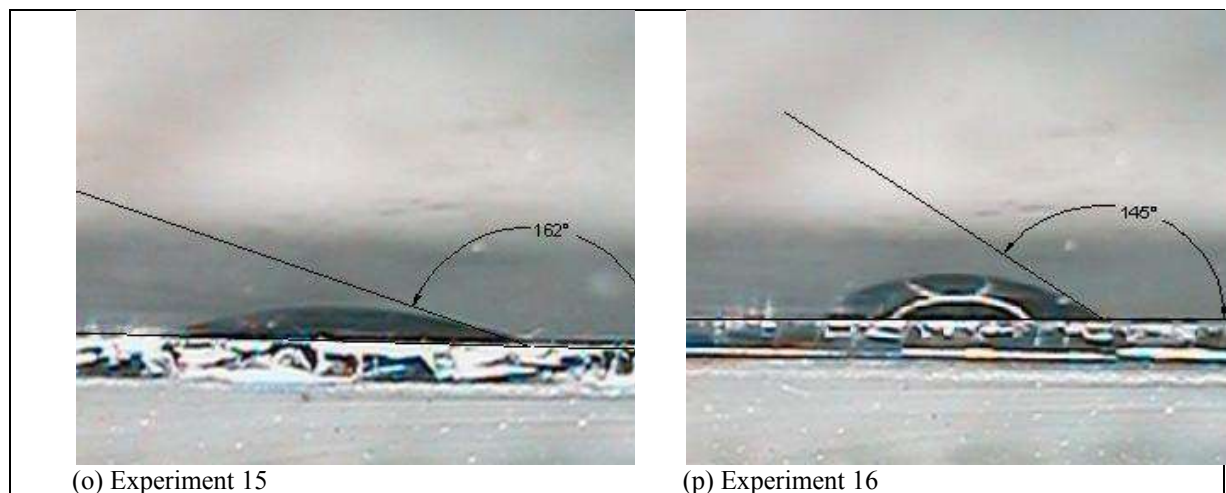
(l) Experiment 12



(m) Experiment 13



(n) Experiment 14



**Fig. 2:** Contact angles for experiment 1 till experiment 16 pictured in (a) till (p)

**Conclusion:**

This investigation evaluates the effects of different controllable process parameters on the wettability quality after Reactive Ion Etching (RIE) calculated in the form of contact angle. The type of gaseous is the most significant factor in relationship to the contact angle where SF<sub>6</sub>+Ar gives higher values of contact angle compared to CF<sub>4</sub>+Ar gaseous. Bias power and working pressure are both inversely proportional to the contact angle values but not significant effects. For ICP Power, it has a positive effect whereby contact angle is high when the ICP is high but the effect is also not significant compared to gaseous. All the experiments produced a contact angle larger than 90° and fall in hydrophilic category.

**ACKNOWLEDGEMENT**

The author would like to thank School of Microelectronic Engineering, Universiti Malaysia Perlis for their support and facilities. The authors would also acknowledge the financial support and aid from the Ministry of Higher Education of Malaysia (MOHE).

**REFERENCES**

- [1] Neethirajan, S. *et al.*, 2011. "Microfluidics for food, agriculture and biosystems industries," *Lab on a Chip*, 11: 1574-1586.
- [2] Sia, S.K. and L.J. Kricka, 2008. "Microfluidics and point-of-care testing," *Lab on a Chip*, 8: 1982-1983.
- [3] Dittrich, P.S. and A. Manz, 2006. "Lab-on-a-chip: microfluidics in drug discovery," *Nature Reviews Drug Discovery*, 5: 210-218.
- [4] Anastasiadis, S.H., 2013. "Development of Functional Polymer Surfaces with Controlled Wettability," *Langmuir*, 2013.
- [5] Dickinson, T., A.F. Povey and P.M.A. Sherwood, 1975. *J. Chem. Soc. Faraday Trans.* 71: 298.
- [6] Ye, W., *et al.*, 2010. "Surfactant-free and controllable synthesis of hierarchical platinum nanostructures and their comparative studies in electrocatalysis, surface-enhanced Raman scattering and surface wettability," *Electrochimica Acta*, 55: 8649-8654.
- [7] Argentiére, S., *et al.*, 2014. "Smart Microfluidics: The Role of Stimuli-Responsive Polymers in Microfluidic Devices,"
- [8] Abate, *et al.*, A.R., 2010. "Patterning microfluidic device wettability using flow confinement," *Lab on a Chip*, 10: 1774-1776.
- [9] Dickinson, T., A.F. Povey and P.M.A. Sherwood, 1975. *J. Chem. Soc. Faraday Trans.*, 71: 298.
- [10] Dutschk, V., 2013. "Surface Wetting: From a Phenomenon to an Important Analytical Tool," in *Without Bounds: A Scientific Canvas of Nonlinearity and Complex Dynamics*, ed: Springer, pp: 227-257.
- [11] Antony, J., 1999. "Improving the wire bonding process quality using statistically designed experiments," *Microelectronics Journal*, 30: 161-168.