

# Using Acid/Base with AHAPTAMOS to Control the Hydrophilic/Hydrophobic Character of Silicon Wafers

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## ABSTRACT

In the semiconductor industry, manufacturing processes require varying degrees of hydrophobicity/hydrophilicity in silicon wafers. To optimize transitions between these processes, the effectiveness of using acid/base to control the hydrophobicity/hydrophilicity of a silicon wafer following the addition of N-(6-aminohexyl)aminopropyltrimethoxysilane (AHAPTAMOS) was examined. After coating with AHAPTAMOS, the untreated wafer will become more relatively hydrophobic, but when acid is applied, the surface amino groups are protonated, theoretically increasing hydrophilicity; applying a base would neutralize the surface and reverse this process. A total of four trials were conducted, each trial consisting of four successive tests for a single wafer: *No Coating*, the control group, with the uncoated/untreated wafer; *Coated But Not Treated*, with AHAPTAMOS applied to the same wafer; *Acid Treated*, with weak acid applied to the coated wafer; *Base Treated*, with a weak base applied to the acid-treated wafer. For each test, the contact angle was measured with a goniometer and the Ossila computer software. Results indicated that the hydrophobicity of the wafer was successfully manipulated: upon application of AHAPTAMOS, the contact angle experienced a 46.18% increase from the control, and with the subsequent addition of acid, the contact angle yielded a 13.60% decrease. Then, the addition of a base resulted in a 23.14% increase in contact angle. These results have strong implications for future semiconductor research and manufacturing, as AHAPTAMOS can be explored as a more efficient coupling agent to control the hydrophilicity/hydrophobicity of silicon wafers.

**Keywords:** N-(6-aminohexyl)aminopropyltrimethoxysilane; AHAPTAMOS; semiconductors; semiconductor manufacturing; hydrophobicity, hydrophilicity; silicon wafer

## INTRODUCTION

Digital technology has become fundamental to our modern society and is continuously growing to be more prevalent in our everyday lives. The key to this technological advancement lies in the computer chip that powers every cell phone, computer, or headset ever manufactured: the semiconductor. As a result, the semiconductor industry that manufactures these

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computer chips is a highly relevant and impactful industry that must be maintained at maximum efficiency.

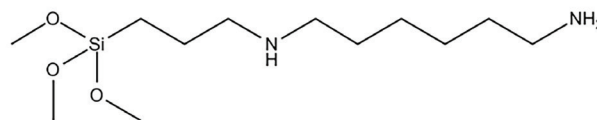
The silicon wafer (also called an integrated circuit or semiconductor device) is the foundation for creating a computer chip. These thin disks are made of purified crystalline silicon, a semiconductor material typically grown using the Czochralski Method to create its uniform circular shape (1). From this pure wafer to an integrated circuit, manufacturers often have thousands of manufacturing steps that either add to or remove material from the wafer surface (2). Important fabrication steps include deposition, which involves layering materials onto the surface of the wafer for additional properties; photoresist application, which applies a light-sensitive material on the surface that creates patterns when exposed to light; and etching, which cuts the surface of the wafer in specific patterns after the photoresisting step. In the deposition and photoresist application steps, hydrophobicity generally improves the adhesion of the material onto the surface of the wafer. However, hydrophilicity is also important for deposition, as it depends on the specific material whether a hydrophobic or hydrophilic surface is preferred for bonding/attaching to the wafer surface (3). As there are so many deposition steps throughout the entire process, manufacturers must constantly change between hydrophilic and hydrophobic states on the surface of the wafer. This is a potential inefficiency that can cost companies both time and money over extended periods.

Silicon wafers are naturally hydrophilic when untreated due to a native oxide layer of SiO<sub>2</sub>, SiH, and SiOH bonds (4). Manufacturers today use coupling agents that undergo substitution reactions by bonding to the surface silanols, changing the hydrophilic native oxide layer to hydrophobic. Hexamethyldisilazane (HMDS) is one of the most common coupling agents used in the industry to create a more hydrophobic surface relative to the control, due to its demonstrated reliability in places such as photoresist application (5). However, several problems come with HMDS: one of which is the problem of efficiency. As mentioned, manufacturers must frequently change the state of the silicon wafer surface between hydrophobic and hydrophilic. However, using HMDS means that HMDS must be constantly added to and removed from the wafer. Adding HMDS requires either heating the wafer, spin-coating HMDS on the wafer and baking it, or bubbling HMDS onto the wafer, and removing HMDS requires hydrofluoric acid. Even after removal, the wafer must be cleaned to prevent contamination. The reiteration of these numerous steps

just to change the hydrophobicity/hydrophilicity requires much time and effort. A second problem is the safety hazards posed by HMDS and hydrofluoric acid (HF) to the workers and the environment. HMDS is toxic through inhalation, and HF is a corrosive and toxic chemical. To solve these issues, a new chemical is proposed to replace HMDS, and experimentation was designed to answer the question of its effectiveness.

Aminopropylalkoxysilanes are a type of silane coupling agents used to modify oxide surfaces because of their ability to form covalent siloxane bonds while keeping amine functional groups. These amine groups can go through protonation and deprotonation, allowing interactions with water, resulting in control over surface wettability. Aminopropylalkoxysilanes have the ability to reverse surface properties through chemical treatments, differing from HMDS, which makes surfaces hydrophobic until its removal. AHAPTMOSS was selected from this group of silanes because of its extended alkyl chain and its many amine functions, which enhances surface stability and adds increased sensitivity to acid base interactions. Other than aminopropylalkoxysilanes, triethoxysilane (APTES) also has the ability to bond to oxide surfaces. However, when compared to HMDS, amino-functional silanes have changeable surface properties.

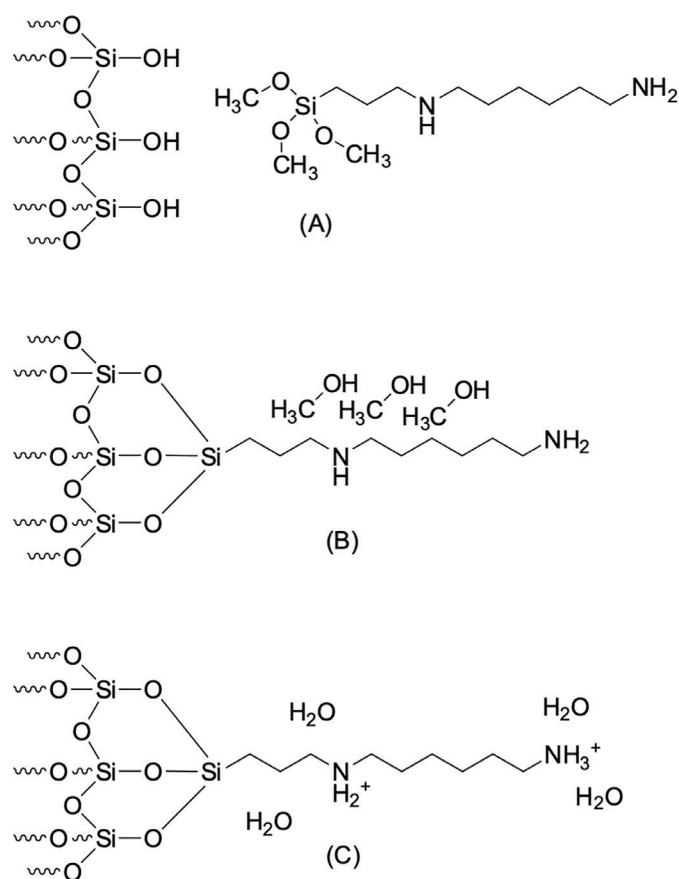
Aminopropylalkoxysilanes are common coupling agents as the amine groups help to catalyze the bonding reaction between the molecule and the surface silanols (6). For the present experiment, N-(6-aminohexyl) aminopropyltrimethoxysilane (AHAPTMOSS) was selected as the coupling agent (Figure 1). Previous studies have not deeply considered the application of AHAPTMOSS in the semiconductor manufacturing industry. Theoretically, AHAPTMOSS would bond to the surface of the silicon wafer, similarly to HMDS, when exposed to enough heat through a substitution



**Figure 1.** Structure of N-(6-aminohexyl)aminopropyltrimethoxysilane (AHAPTMOSS). This figure shows the skeletal structure of N-(6-aminohexyl)aminopropyltrimethoxysilane. This molecule is composed of a trimethoxysilane group attached to a propyl linker connected to a secondary amine and a six-carbon chain.

reaction by attaching its three methoxy ends to three corresponding silanol sites (Figure 2A). Though some methoxy ends may form weaker hydrogen bonds instead of being firmly attached through covalent bonds, this was deemed negligible as AHAPTAMOS would retain most of its surface functionality.

When AHAPTAMOS is applied, it would initially make the surface more hydrophobic like any other coupling agent (Figure 2B). However, exposing this surface molecule to a weak acid would change its hydrophobic property to more hydrophilic as the amino groups get protonated and attract water molecules (Figure 2C). A weak base can deprotonate the surface of AHAPTAMOS and return the wafer to its original, more hydrophobic state. As such, AHAPTAMOS has



**Figure 2.** (A) Silicon wafer surface with native oxide and surface silanol (left) and AHAPTAMOS (right). (B) AHAPTAMOS bonded to the surface with three moles of methanol as a byproduct. (C) Amino groups in AHAPTAMOS are protonated, making the surface hydrophilic.

the unique property of being able to change its degree of hydrophobicity or hydrophilicity by using acid or base, which has not been discussed. This property could make industrial manufacturing processes more efficient because manufacturers can simply expose the silicon wafer surface to an acid or a base to make it more hydrophobic or hydrophilic, rather than having to entirely remove the chemical. HF would be used only for removing AHAPTAMOS, which is only required once throughout the process. Our experiment therefore addressed whether acid/base could be effectively used to control the hydrophobic/hydrophilic character of silicon wafers.

Hydrophobicity and hydrophilicity can be measured with the contact angle of a water droplet on the surface of the material – the angle created by the tangent line of the water droplet and the surface. A greater contact angle would indicate a greater hydrophobicity because the material repels the water droplet more noticeably, while a smaller angle would indicate a greater hydrophilicity because the water droplet is more adhered to the surface. Conventionally, hydrophobicity is defined as a contact angle greater than 90° while hydrophilicity is classified to be less than 90°. For this research, however, wettability is defined comparatively, with relative increases in contact angle indicating greater hydrophobic behavior.

## METHODS AND MATERIALS

Four 100 mm diameter single-side polished silicon wafers from Fuleda Technology were purchased for the experiment. The four separate silicon wafers represented the four different trials in the experiment. For each of the silicon wafers, four tests measured its hydrophobicity/hydrophilicity at different stages. One droplet was measured per condition per wafer. Contact angle was used to measure hydrophobicity/hydrophilicity, and an Ossila contact angle goniometer connected to a computer was used to determine the contact angle. After using a microsyringe to drop a very small microliter scaled distilled water droplet (~2 μL) onto the surface of the wafer, a camera in the goniometer would send images to the computer software, which would then determine the left and right contact angles and the average of the two contact angles.

99% pure AHAPTAMOS, a moderately viscous liquid at room temperature, was synthesized and received from Seoul National University. First, the average contact angle of the pure, untreated wafer was measured and recorded under *No Coating* (using the microsyringe and

the Ossila contact angle goniometer and software). Then the same silicon wafer was coated with AHAPTAMOS by heating the wafer to 120°C on a hot plate and then spreading AHAPTAMOS over the surface. Methanol was observed to be evaporating off the wafer, evidencing the chemical reaction of AHAPTAMOS bonding to the surface.

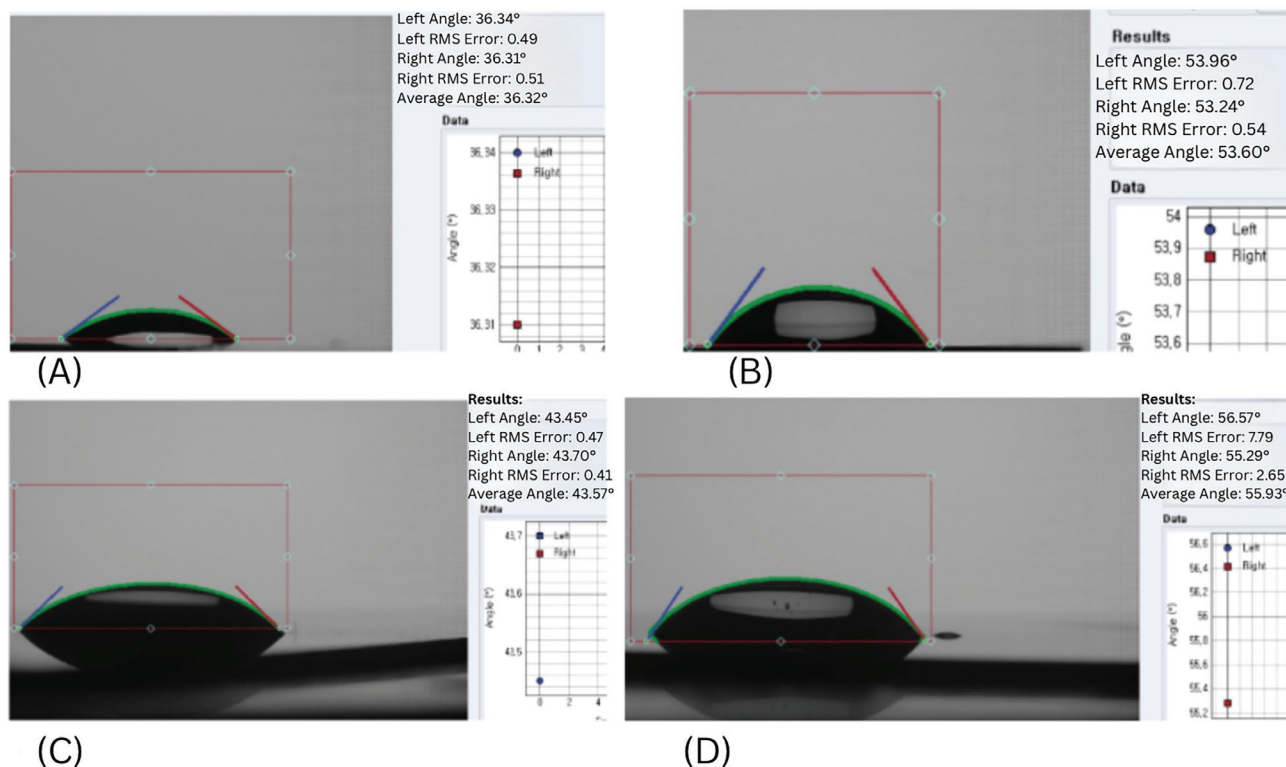
Once AHAPTAMOS was applied, the wafer was cooled down and the procedure of recording the contact angle was repeated (*Coated But Not Treated*). Then the coated wafer was immersed in a 5% acetic acid solution (vinegar) for five minutes for the complete protonation of amino groups. After being taken out and dried, the contact angle of the wafer was again recorded under *Acid Treated*.

Finally, the coated wafer was immersed in a saturated sodium bicarbonate solution (baking soda) for five minutes to deprotonate the amino groups on the surface AHAPTAMOS molecules. The contact angle was recorded once more under *Base Treated*. As a safety precaution to avoid inhalation of methanol, the experiment was conducted in a well-ventilated open garage.

## RESULTS

Contact angle measurements are shown in Figure 3 and compiled into Table 1. The data was fairly consistent throughout all four trials for each of the tests, indicating precise data from the goniometer; the standard deviation was calculated to be 0.12° for *No Coating*, 0.31° for *Coated But Not Treated*, 1.49° for *Acid Treated*, and 0.67° for *Base Treated*, all relatively small values. The only outlier was the first trial of the *Acid Treated* test, which is attributed to errors from the goniometer.

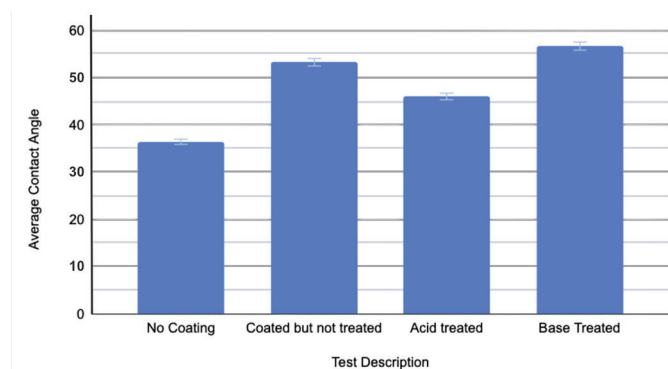
The contact angles for each test were averaged and graphed (Figure 4), and a clear pattern can be observed. As hypothesized, the uncoated wafer surface exhibited the lowest contact angle, with the addition of AHAPTAMOS yielding an increase of 46.18%. Treating the surface with acid then decreased the contact angle by 13.60%, before base treatment increased the contact angle back up by 23.14%. Therefore, the *Acid Treated* results exhibited lower contact angles relative to the *Coated But Not Treated* testing, signifying greater hydrophobic conditions compared to acid treated conditions.



**Figure 3.** Raw data taken from Ossila contact angle goniometer software. (A) Trial 1: No Coating. Average angle of 36.32° (B) Trial 1: Coated But Not Treated. Average angle of 53.60° (C) Trial 1: Acid Treated. Average angle of 43.57° (D) Trial 1: Base Treated. Average angle of 55.93°.

**Table 1.** Contact angle measurements for silicon wafers under four surface conditions. This table presents the water contact angle measurements obtained from four independent trials evaluating the wettability of silicon wafers under four surface conditions: No Coating, Coated But Not Treated (AHAPTAMOS applied), Acid treated, and Base Treated. Contact angle measurements were averaged from the left and right angles of a  $\sim 2$   $\mu\text{L}$  deionized water droplet using a contact angle goniometer. Higher contact angles indicate greater hydrophobicity, while lower values indicate greater hydrophilicity.

Trial	No Coating	Coated But Not Treated	Acid Treated	Base Treated
1	36.32	53.6	43.57	55.93
2	36.5	52.85	46.76	57.7
3	36.62	53.43	47.38	57.09
4	36.59	53.62	46.73	56.39
avg	36.51	53.37	46.11	56.78



**Figure 4.** Average contact angle across four trials of each: No Coating, Coated But Not Treated, Acid Treated, Base Treated. Angle measurements taken from Table 1, "Avg."

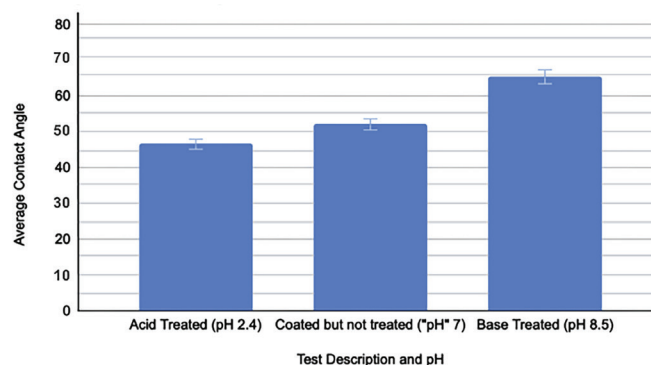
## DISCUSSION

The purpose of the experiment was to analyze if the unique properties of AHAPTAMOS could be demonstrated and to see its effectiveness in demonstrating this hydrophobic/hydrophilic property with acid and base treatment. While this property is depicted in Figure 4, all of the contact angles are below  $90^\circ$ , indicating hydrophilic behavior by conventional standards. Thus, it is clear that AHAPTAMOS is not as effective as HMDS with its hydrophobicity; for reference,

the largest contact angle measured in the experiment was  $57.70^\circ$  in the second trial of the *Base Treated* test (Table 1) while HMDS application will result in a contact angle of  $65^\circ$ - $80^\circ$  (8). Moreover, the difference in contact angle between the hydrophilic *Acid Treated* state and the more hydrophobic *Base Treated* state of AHAPTAMOS is only  $10.68^\circ$ , representing a modest difference in surface properties. Because there was a limited sample size, small differences in contact angle were noted as trends rather than conclusive differences. In practice, the deposition of a material that favors hydrophobicity could have similar results when testing either state, which could potentially limit the applicability of the usage of this chemical in processes requiring a stronger relative hydrophobic difference.

The pH was not directly measured during the experiment but was calculated for both the acetic acid and saturated sodium bicarbonate solutions as 2.4 and 8.5, respectively. While only two pH values were used in the experiment, the *Coated But Not Treated* may be considered a neutral pH of 7, although this value remains speculative as pH was not directly measured. As a result, an apparent trend between pH and contact angles is therefore seen. Since *Coated But Not Treated* pH value was not definitively measured, this cannot be considered sufficient evidence for the relationship between pH and contact angle; however, it provides valuable insight into future experimentation (Figure 5).

An unexpected observation was that the *Base Treated* contact angle was larger on average than the *Coated But Not Treated* contact angle. At first, this was again attributed to error like the first trial from the *Acid Treated* group, but the consistency of the error across all four trials



**Figure 5.** Average contact angle as a function of pH. 2.4 (Acid Treated), 7 (Coated But Not Treated), and 8.5 (Base Treated). Angle measurements taken from Table 1, "Avg."

indicated that the pKa of AHAPTMOS itself was never considered nor measured. The pKa of amine functional groups likely exceeds 7 due to the presence of the amino groups, meaning that it may be partially protonated, most likely by the atmospheric moisture (6). Additionally, environmental conditions such as temperature and humidity were not measured or controlled during this experiment. Since surface protonation may be influenced by atmospheric moisture, therefore changes in the environment could have led to the differences in contact angle. This suggests that even before the experiment was conducted, AHAPTMOS was likely to be partially protonated, reducing the contact angle of the *Coated But Not Treated* test below what it should have been without this unexpected process. The *Base Treated* test is likely a better representation of the contact angle with pure AHAPTMOS, as the sodium bicarbonate solution would have deprotonated the molecule.

Despite these potential limitations and inaccuracies, the acid/base dependent properties of AHAPTMOS that were observed from the experiment demonstrate significant contributions to the current body of research, as using acid/base treatment to control hydrophobic and hydrophilic behavior has not been researched extensively in this specific context. Furthermore, this suggests a greater potential for AHAPTMOS, as future developments could create an improved molecule that is more sensitive to changes in pH and can create larger differences in its hydrophobic and hydrophilic states.

## CONCLUSION

This study investigated whether using N-(6-aminohexyl)aminopropyltrimethoxysilane (AHAPTMOS) on silicon wafers could enable wettability changes dependent on pH conditions. Contact angle measurements demonstrated consistent and repeatable hydrophobic and hydrophilic behavior changes following AHAPTMOS, acid, and base chemical treatment processes. AHAPTMOS coating initially increased contact angle relative to the uncoated wafers, and acid treatment reduced contact angle while base treatment restored it, confirming the reversible protonation and deprotonation effects associated with this amine group.

Although all measured contact angles were under 90° and were thus conventionally classified as hydrophilic, and the observed angle shifts were modest compared to commonly-used industrial coupling agents such as HMDS, the demonstrated wettability changes serve as a crucial proof of concept, paving the first steps towards

the use of chemical tuning to influence hydrophobic and hydrophilic behavior without repeated coating removal. Future work may focus on investigating a more direct pH-wettability relationship across a broader range, optimizing coating parameters to improve the observed results, or regulating external conditions to limit the influence of environmental circumstances on outcomes. Ultimately, this work demonstrates foundational evidence for a novel idea of chemical coating and treatment in semiconductor processing. Adopting AHAPTMOS could enable wettability change; however, more testing on durability, repeatability, and practicality is still required before it can be formally considered for industrial applications.

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## CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest related to this work.

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