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Article

Changing the surface properties of the backside of a silicon wafer to repel oil and prevent particle binding

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SUMMARY

Wafers are thin semiconductor materials essential in the production of microchips, which are present in almost every electronic device. A semiconductor wafer chuck, a plate that holds wafers, is used in multiple semiconductor processes, including cleaning, etching, polishing, lithography, and deposition. Even though wafers are produced as silicon, the backside of the wafer can be oxidized by air or water vapor to create silanol (Si-OH). Silanol can easily bind to silica particles to cause leveling issues on the chuck. In addition, though the backside of the wafer has many Si-OH bonds, oil can stick to the nonpolar area of the wafer, causing wafer slip that can result in shattering. To address these issues, we coated the wafer backside using [acetoxy(polyethyleneoxy)propyl]triethoxysilane (APTS), which prevents particle binding and repels oil effectively. We hypothesized that the siloxane bond from APTS would block incoming silica particles and that the polyethylene glycol (PEG) group would repel oil because PEG is a polar group that does not attract oil. We used a contact angle goniometer to confirm that the hydrophobic part of the wafer became hydrophilic (contact angle < 90°) and found that the wafer repelled oils. Additionally, we exposed the backside of the wafer to silica particles to examine whether APTS prevented particles from binding onto the wafer. SEM data confirmed that the APTS-coated wafer effectively blocked silica from binding to the backside of the wafer. This approach can improve semiconductor wafer reliability and benefit other research needing contamination-resistant surfaces.

INTRODUCTION

The world is undergoing a rapid transformation through the Fourth Industrial Revolution, a term for the current wave of technological improvements, with semiconductors at its core (1). Semiconductors have essential applications across numerous industries; in fact, semiconductor technology has been necessary for the computing revolution (1). The Fourth Industrial Revolution and AI technologies were made possible by a chip known as the integrated circuit (IC), which is made of semiconductors. ICs are approximately 2 mm x 2 mm and consist of electronic components. These must function together precisely to perform logic operations and store data (2). An IC is built on a base called a wafer, which is generally made of silicon (**Figure 1**).

While wafers can be made from various materials, including silicon and germanium, silicon-based wafers are popular for three reasons. Firstly, silicon is one of the most abundant elements on Earth, making it easy to use. Secondly, silicon wafers have a high heat resistance of about 150 °C. Lastly, silicon wafers have fewer free electrons at room temperature than other elements, such as germanium, which makes them better conductors (3).

Manufacturing the finest quality ICs requires six main steps: deposition, photoresist, etching, ionization, lithography, and packaging (4). Along with the six main steps, there are other sub-steps such as electroplating, wafer testing, and cleansing (5-7). A wafer must go through these sub-steps hundreds of times before becoming part of a device (5-7). At each step, an essential part known as the “chuck” holds the wafer in place in each tool (8). Potential problems with chucks are numerous and include uneven lithography due to improper alignment, wafer slip due to oil or nonpolar coating, helium pressure aborting due to particles causing registration issues, and the inability to proceed to the next downstream process due to bent wafers (9, 10). These problems mainly arise due to oil residues and particles that disrupt wafer uniformity during wafer chucking in each tool.

To address this issue, chemical compounds have previously been utilized to improve the surface properties of wafers. 3-Aminopropyltriethoxysilane (APTES) can be used to introduce amine groups, which can be further reacted with other molecules for various applications such as biosensors and adhesion promotion (11). Octadecyltrichlorosilane (OTS) is also used to create hydrophobic surfaces on silicon by forming a self-assembled monolayer (12). This is useful for applications requiring water-resistant coatings (12).

[Acetoxy(polyethyleneoxy)propyl]triethoxysilane (APTS) is a chemical compound with silanol functional groups that can react with other silicon-containing groups to form siloxane, which effectively prevents silica particle binding (**Figure 1**). APTS has unique properties: it possesses three Si-OEt bonds that can react with Si-OH groups to create stable siloxane linkages, while its hydrophilic polyethylene glycol (PEG) groups repel nonpolar oils, such as those commonly introduced by contact with skin.

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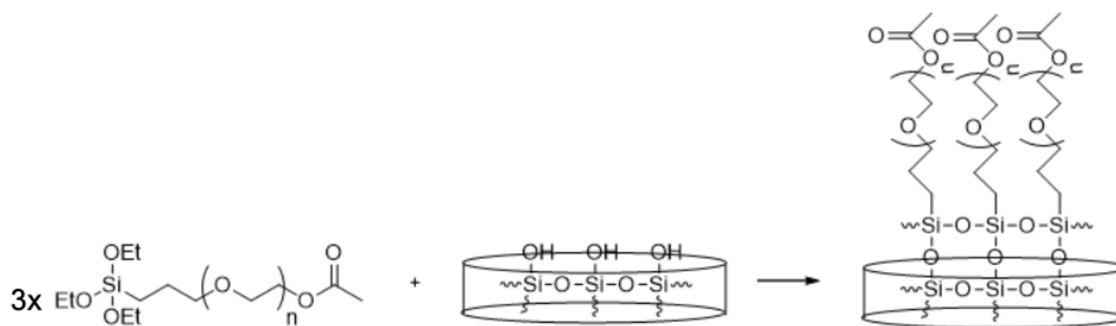

Figure 1: Reaction mechanism of three [acetoxy(polyethyleneoxy)propyl]triethoxysilane (APTS) on the surface of a silicon wafer. Three APTS molecules react with the surface of a silicon wafer to yield an APTS-coated wafer.

Semiconductor fabrication plants typically feature laminar airflow, which minimizes particle contamination on wafers by filtering outside air to remove microcontaminants (13). Despite efforts to control the number of particles, completely removing particles from the air in the plants is impractical. For this reason, semiconductor companies continue to face decreased efficiency in semiconductor production. In addition, depending on the height of the chuck, wafers that fall may shatter, releasing more small particles that can contaminate other clean wafers (15).

We hypothesized that coating the backside of the wafer with APTS would prevent both oil and particle issues. The backside of the wafer is made of polysilicon, which air and moisture convert to a small number of silanols (Si-OH) (16). These silanol functional groups can react with other silicon-containing groups to form siloxane, which effectively prevents silica particle binding. This dual functionality—resistance to both particles and oils—addresses critical challenges in wafer handling, improving leveling and slippage and thus enhancing the reliability and yield of semiconductor manufacturing processes.

Contact angle measurements, a widely used technique for assessing surface wettability, were conducted to verify the hydrophilic nature of the APTS coating. By placing a small droplet on the clean, coated wafer surface and capturing its profile with high-resolution imaging, we determined the contact angle — a metric indicating surface wettability. On a hydrophilic surface, contact angles fall below 90°, with values under 30° indicating very high wettability; hydrophobic surfaces exhibit angles above 90°, and superhydrophobic surfaces exceed 120°. These measurements are broadly important across material science, coatings, and industrial applications as they reveal how materials interact with liquids, helping to inform performance optimizations. Here, achieving a low contact angle confirmed that the APTS-coated wafer surface was sufficiently hydrophilic to repel oil.

To evaluate the effectiveness of APTS in preventing silica particle adherence, we exposed the wafer to 2 M hydrochloric acid, which can promote bonding between silica particles and any silanol groups present on the wafer. Scanning electron microscopy (SEM) analysis showed that the APTS-coated wafer was free from silica particles, whereas an uncoated wafer accumulated particle binding. This suggested that APTS effectively inhibited particle adhesion by forming siloxane bonds with the wafer's silanol groups and maintaining a hydrophilic surface due to the PEG groups. In summary, our experiments demonstrated that APTS coating prevented oil and particle contamination by forming siloxane bonds and maintaining surface hydrophilicity. This approach holds promise for enhancing semiconductor wafer reliability and may have broader applications in fields requiring contamination-resistant surfaces.

RESULTS

To study the effect of APTS on the backside of silicon wafers, we initially conducted a reference trial at 25 °C with 1.0 mL of water and we used these results from this trial as a reference for subsequent experiments (**Table 1**). This reference trial provided a baseline result that indicated effective coating. Then, we conducted tests with differing temperatures to determine the temperature at which APTS reacts most effectively with the silanols (**Table 1**). We observed higher contact angles at lower temperatures, indicating that APTS did not react, which led to our conclusion that the optimal temperature was 100 °C. Moreover, we also observed evaporation of a substance at temperatures exceeding 80 °C (data not shown). This was likely ethanol, as ethanol evaporates at 73 °C. Once we determined that the optimal temperature was 100 °C, we conducted four different reactions with varying time intervals to measure the rate of a reaction between APTS and silanols (**Table 2**). Ethanol evaporation commenced after 3 seconds and ceased after 7 seconds. After collecting data from time-variable tests, we tested different amounts of APTS, namely 0.05 mL, 0.1 mL, 0.2 mL, and 0.8 mL, to find the optimal amount of APTS (**Table 3**). 0.1 mL was found to be optimal. Lower amounts were insufficient in achieving the desired contact angle, likely

Trial	Time (s)	Amount (mL)	Temperature (°C)	Contact Angle (Left) (°)	Contact Angle (Right) (°)
reference	5	0.1	25	106	104
1	5	0.1	40	98	97
2	5	0.1	60	85	83
3	5	0.1	80	79	77
4	5	0.1	100	69	70
5	5	0.1	120	68	69
6	5	0.1	140	68	70

Table 1: Contact angle vs. temperature. The coated silicon wafers were placed on a contact angle goniometer, and a droplet of water was placed on the wafer using a 1 mL precision syringe. The image was analyzed using Ossila contact angle measurement software, and a contact angle measurement was determined.

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Trial	Time (s)	Amount mL	Temperature (°C)	Contact Angle (Left) (°)	Contact Angle (Right) (°)
1	1	0.1	100	92	91
2	3	0.1	100	79	80
3	5	0.1	100	68	68
4	10	0.1	100	67	68

Table 2: Reaction time variation tests. The coated silicon wafers were placed on a contact angle goniometer, and a droplet of water was placed on the wafer using a 1 mL precision syringe. The image was analyzed using Ossila contact angle measurement software, and a contact angle measurement was determined.

Trial	Time (s)	Amount (mL)	Temperature (°C)	Contact Angle (Left) (°)	Contact Angle (Right) (°)
1	5	0.05	100	88	87
2	5	0.1	100	70	69
3	5	0.2	100	70	70
4	5	0.8	100	79	79

Table 3: APTS variation tests. The coated silicon wafers were placed on a contact angle goniometer, and a droplet of water was placed on the wafer using a 1 mL precision syringe. The image was analyzed using Ossila contact angle measurement software, and a contact angle measurement was determined.

due to incomplete surface coverage. Higher amounts did not improve the contact angle further, suggesting that 0.1 mL is adequate for full surface reaction without excess reagent.

We compared the compiled data with the results of our reference trial, which was done at 25 °C with 1.0 mL of water, and proceeded to assess the anti-oil and anti-particle properties of the APTS coating (**Table 1**). For the anti-oil testing, we covered half of the wafer with aluminum foil to prevent coating and coated the other half with APTS (**Figure 2**). After applying the coating, we performed a finger oil test by using our fingers to deposit oils and observed any differences between the coated and uncoated areas of the wafer (**Figure 3**). Visually, the surface of the half of the wafer that was coated with APTS showed less visible oil residue, and our fingerprints were not as clear and distinguishable compared to that of the uncoated half of the wafer.

We then tested the efficacy of the APTS coating in preventing particle adhesion by submerging the wafer in a mixture of silica powder and hydrochloric acid. The wafer was then subjected to SEM analysis to quantify particle binding. SEM data revealed that the coated wafer was free of silica particles, while the uncoated wafer exhibited approximately 25 bonded silica particles per 50 μm^2 (**Figure 4**). As hypothesized, coating the silicon wafer with APTS minimized oil stains and prevented silica particles from bonding to the wafer.

DISCUSSION

In this study, we aimed to evaluate the effectiveness of APTS (3-aminopropyltriethoxysilane) as a coating to enhance the anti-oil and anti-particle properties of wafer surfaces.

Figure 2: APTS coating on a silicon wafer. The image shows the process of APTS coating on a hot plate. Half of the silicon wafer was covered using aluminum foil (left) while the other uncovered half of the wafer was coated with APTS (right). The half-wrapped silicon wafer was placed onto a hot plate at 100°C and the right side was coated with 25 g of 95% APTS for 5 minutes. The wafer was then rinsed with pure acetone and air-dried.

Our experimental design focused on testing various reaction temperatures and times to determine optimal conditions for APTS bonding on the wafer, and on assessing the performance of the coating in anti-oil and anti-particle tests. Our results indicated that the APTS coating enhanced the anti-oil and anti-particle properties of the wafer surface. This was likely due to strong siloxane bonds formed between the triethoxy groups of APTS and the silanol groups on the wafer surface, which prevented silica particle adhesion and contributed to oil repellency via the hydrophilic polyethylene glycol groups (**Figure 5**). While this bonding mechanism is a plausible explanation, further structural analysis of the coating would be necessary to confirm the exact chemical interactions responsible for these effects, as other possible mechanisms could also contribute. We believe that the triethoxy groups attached to the APTS were functionalized by OH groups present on the surface of the wafer, thus forming strong siloxane bonds (**Figure 1**). This dual functionality would address both particle-induced alignment issues and oil-induced slip, which are critical concerns in wafer processing. At low temperatures, APTS exhibited limited mobility, or the ability of the APTS molecules to move freely within a medium, due to high viscosity; however, temperatures exceeding 80 °C facilitated free movement of APTS due to low viscosity (**Table 1**).

To optimize APTS bonding, we investigated reaction temperatures and times. Initially, reaction tests at 40 °C and 60 °C with a 5-second reaction time showed contact angles indicating a hydrophilic surface, like those observed in our reference data, which was our first trial conducted as a baseline when comparing data points with differing temperature, time, or amount. This reference data came from initial trials without the APTS coating, providing a comparison for effective coating. These early results suggested that the reaction was not proceeding rapidly enough at these temperatures. Therefore, at 80 °C, we predicted that ethanol would be created as a side-product when silanol groups on the wafer surface reacted with APTS, as ethanol's boiling point is 73 °C, which is just below 80 °C. Although the reaction appeared to occur at temperatures as low as 80 °C, we selected 100 °C as the optimal temperature, as contact angles remained stable beyond this point, indicating a fully formed coating.

We then assessed optimal reaction times in the reaction between silanol groups on the wafer surface and silanes in APTS at this temperature, testing 1, 3, 5, and 10 seconds. Although hydrophilicity appeared at 1 second, we only observed appreciable changes at the 3-second mark. At 5 seconds, no further change was noted, implying that the

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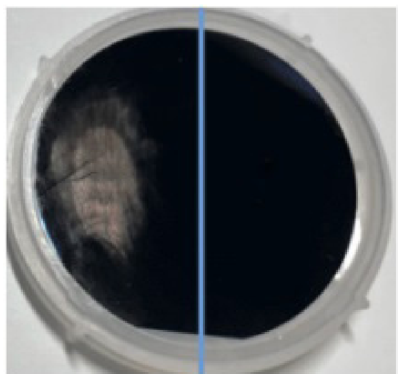

Figure 3: APTS-coated and non-coated sides of a silicon wafer tested with finger oil stains. The left side of the silicon wafer shows a distinct finger oil mark while the right side remains free of oil. Sebum was applied by rubbing one finger onto both the APTS-coated and uncoated sides of the silicon wafer for 20 seconds.

coating formation had reached completion by this point. This optimal reaction time of 5 seconds suggested that the reaction between the wafer surface and APTS was efficient, yielding consistent hydrophilic properties.

In sum, we found that a temperature of 100 °C and a reaction time of 5 seconds provided the best results in terms of contact angle measurements, indicating successful coating. The significance of these conditions lies in their ability to promote optimal reaction of APTS with the wafer surface, thereby maximizing the formation of siloxane bonds and enhancing the hydrophilic properties of the coating.

The anti-oil tests confirmed that the APTS-coated wafer resisted finger oil marks, supporting our hypothesis that polyethylene glycol groups effectively repel oil (**Figure 3**). This property is crucial for maintaining the cleanliness and performance of wafers during handling and processing. For anti-particle properties, the APTS-coated wafer showed almost no adherence of silica particles compared to the uncoated surface, as evidenced by SEM images. These findings demonstrated the efficacy of the coating in preventing particle-induced defects, which are a major cause of alignment issues and other processing problems.

A limitation of our investigation was the lack of our long-term durability testing for the APTS-coated wafers, particularly under conditions that mimic real-world handling and exposure, such as humidity and temperature changes. These factors could impact the coating's performance over time and should be addressed in future studies. In addition, while the anti-oil and anti-particle properties were studied, the compatibility of the APTS coating with other wafer materials and processing steps could be further evaluated. Addressing these challenges will require optimization of the coating process and testing under industrial conditions to ensure reliability and scalability.

The successful application of APTS coating on wafers suggests potential for broader applications, such as in the coating of glass surfaces. For instance, cell phone screens, which also contain silanol groups, could benefit from an APTS coating to repel finger oil and maintain clarity. However, further studies are needed to evaluate the longevity and durability of APTS coatings in practical use. Additionally, the safety and toxicity of APTS, especially with prolonged skin contact, need to be assessed to ensure safety.

In conclusion, the APTS coating provided an effective solution to enhance the anti-oil and anti-particle properties of wafer surfaces. The optimal coating conditions were identified as 100 °C for 5 seconds with 0.1 mL of APTS, ensuring complete surface reaction and maximum efficacy. Future studies could explore the application of similar coatings on other surfaces, such as glass, and investigate the potential of different hydrophilic groups to further improve anti-fouling properties. These advancements could lead to improvements in wafer processing and other fields where surface contamination is a concern.

MATERIALS AND METHODS

Method of Coating the Silicon Wafer with APTS

A hotplate (Four E's, #MI0102003) was covered with two sheets of aluminum foil (Great Value, #00078742349428). Half of a prime grade 2" x 0.4 mm silicon/silicon dioxide (Si/SiO2) wet thermal oxide P/Boron silicon wafer (MSE Supplies)

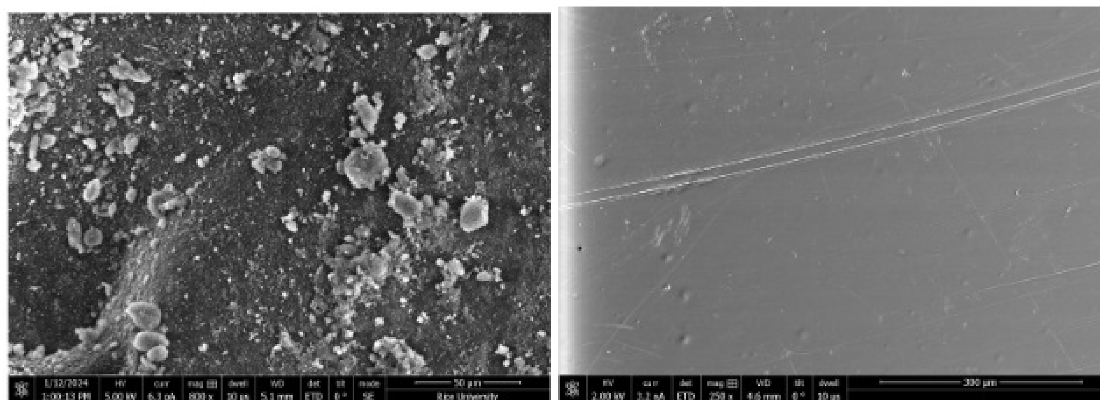

Figure 4: Scanning electron microscope (SEM) image of the surface of a non-coated wafer and a coated wafer. SEM image showing the microscopic surface of a silicon wafer before (left) and after (right) APTS coating magnified 800x and 250x, respectively. Pure fumed silica powder was dispersed into a 1.0 HCl M solution at a temperature of 60 ºC and onto the silicon wafer. The silicon wafer was then imaged using a scanning electron microscope. The SEM image showing the microscopic surface of a silicon wafer before APTS coating was magnified 800x to clearly show the bonded silica particles. The SEM image displaying the microscopic surface of a silicon wafer after APTS coating was magnified 250x after it was noted that the respective wafer showed a negligible difference between the two magnifications to show a more collective surface view.

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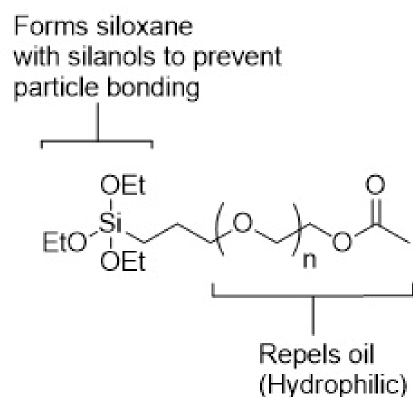

Figure 5: Segments of APTS, the silicon compound used in wafer coating. The left half of APTS includes a triethoxysilane group that undergoes hydrolysis and condensation to form stable siloxane bonds with silanol groups on surfaces, ensuring strong adhesion. The right half includes a polyethylene glycol (PEG) chain with a terminal carbonyl group, which is hydrophilic and repels oil, enhancing the molecule's hydrophobicity for certain surface applications.

was wrapped with aluminum foil. The wafer was then placed onto the hotplate (**Figure 2**). The temperature of the hotplate was initially tested at various temperatures, namely 25 °C, 40 °C, 60 °C, 80 °C, 100 °C, 120 °C, and 140 °C (**Table 1**). The optimal temperature of 100 °C was determined, and the hot plate was heated consistently to 100 °C for the other trials. Different volumes of 95% pure [Acetoxy(polyethyleneoxy) propyl]triethoxysilane (Gelest, #SIA0078.0), namely 0.05 mL, 0.1 mL, 0.2 mL, and 0.8 mL, were tested. As 0.1 mL of APTS exhibited desirable contact angles, it was used to coat the unwrapped half of the silicon wafer using a 1 mL precision syringe (Ibis Scientific, #008131). The silicon wafer remained on the hotplate for 5 minutes. After 5 minutes, the hotplate was turned off and the silicon wafer rested on the plate for 10 more minutes to cool before being removed from the hotplate. The wafer was then rinsed with 16 fl oz of 100% percent pure acetone (Eternal, #017502008410) and air dried.

Method of Testing Oil Resistance

After half of the wafer was coated and rinsed, oil resistance was tested using fingerprint oil, also known as sebum. Sebum was applied by rubbing one finger onto both the APTS-coated and uncoated sides of the silicon wafer for 20 seconds each to see if the finger oil stained the wafer. The silicon wafer was washed with acetone and water and reused for all 4 trials.

Method of Measuring the Contact Angle

A prime grade 2” x 0.4 mm silicon/silicon dioxide (Si/SiO2) wet thermal oxide P/Boron silicon wafer (MSE Supplies) was centered on the test surface of a contact angle goniometer (Ossila, #L2004A1). A droplet of water was deposited onto the coated half of the silicon wafer using a 1 mL precision syringe (Ibis Scientific, #008131). The water droplet was illuminated from behind through a light source, and an image was recorded by the camera. The image was analyzed using version 4.1.5 of the Ossila Contact Angle measurement software, and a contact angle measurement was determined. The contact angle measuring method was repeated with the APTS-coated and uncoated sides of the silicon wafer.

Method of Obtaining Scanning Electron Microscope (SEM) Images of the Surface of the Silicon Wafer

A 1.0 M hydrochloric acid (HCl) solution was created by diluting concentrated hydrochloric acid with deionized water. 1 qt of 100% pure fumed silica powder (TITGGI, #B09JSKQZL4) was then dispersed into the 1.0 M HCl solution at 60 ºC. This dispersion process involved slowly adding the powder to the stirred acid solution until there was uniform dispersion. For the subsequent testing, we added 10 g of silica powder to 100 mL of 2 M hydrochloric acid and fully submerged the wafer. The solution was stirred at 250 rpm for 10 minutes before removing the wafer and rinsing it with 200 mL of water. An even layer of silica dispersion on the surface of the wafer was achieved using a spin-coating technique, in which the wafer is rapidly spun to spread the solution evenly across its surface. The wafer was air-dried and sent to Rice University to obtain SEM data. Scanning electron microscopy (SEM) images were taken with FEI Helios NanoLab 660 DualBeam system using the following parameters: operating voltage (5.0 keV), current (6.3 pA), and dwell time (10 microseconds).

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