YES HMDS VAPOR PRIME PROCESS APPLICATION NOTE

ABSTRACT

Since the early 70's spin dispensed HMDS priming has been used for improving photo resist adhesion to substrates. At present, HMDS vapor priming has become a well-established and widely used technique for photo resist coating. Vapor priming is safer and less expensive due to reduced chemical consumption and the treated surfaces are chemically stable for several weeks. In this overview paper we discuss the various aspects of vapor prime procedures; Comparison results between spin coated, track dispensed HMDS procedures with the YES - HMDS Vapor Prime Oven system. We also present an example of statistically designed experiments (DOE's) for process parameter optimization for specific applications and summarize the results from the statistically designed experiments (DOE's) conducted at the University of Texas at Dallas.

INTRODUCTION

HMDS (hexamethyldisilizane) was first described in U.S. Patent 3,549,368 by R. H. Collins and F. T. Devers of IBM (1970) as a photoresist adhesion promoter for semiconductor applications. Since then HMDS Vapor priming has become a well-understood and preferred technique for photoresist coating applications. It allows for reduced chemical consumption which can be chemically stable for several weeks (Ut, UCB). In addition to proper adhesion, surface moisture is also a major factor that also could degrade resist adhesion and could result in resist pattern peeling off or unwanted lateral etching through the cracks under the resist. YES Vacuum Vapor prime offers the advantage of combined dehydration and priming in the same process chamber.

"Yield Engineering Systems has combined the most effective application methods of vacuum baking and vapor priming. By utilizing the same chamber, the system creates a heated vacuum environment for dehydration and vapor priming, greatly reducing the risks of rehydration or contamination of the wafers." ***- Princeton University, Micro/Nano Fabrication Laboratory***
http://www.prism.princeton.edu/PRISM_cleanroom/equip/image%20reversal%20oven/YES.html

I. HMDS (Hexamethyldisilazane) PROCESS:

HMDS Reacts with the oxide surface forming a strong bond as shown in **Figure 1**, but at the same time leaving free bonds to react with the photoresist and to improve adhesion.

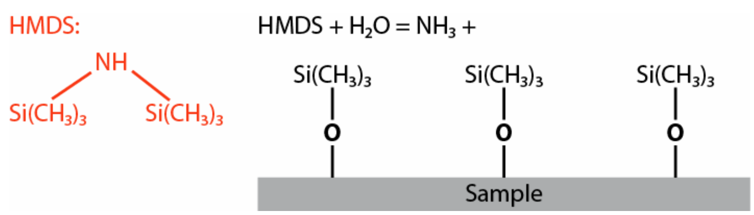


*Figure 1: The behavior of silicon oxide surface treated with HMDS.

Vapor priming of HMDS with a Dehydration bake (YES process): HMDS will tie up the molecular water of the hydrated wafer surface and increase liquid contact angle as the wafer surface turns more hydrophobic. An initial high temperature bake and Dehydration process is also needed for a uniform and stable vapor priming of the substrates. The need for such a complete Dehydration process prior to HMDS applications to produce stable surfaces is presented in **Figure 2**.

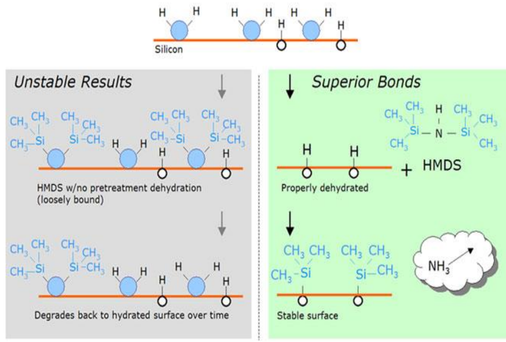


Figure 2: The behavior of HMDS coated surfaces with and without dehydration prior to HMDS processes.

II. HMDS PRIME PERFORMANCE COMPARISON STUDIES

The ever diminishing critical feature size reduction in the extreme sub-micron regime is requiring superior photolithography procedures during their fabrication. The adhesion of photoresist is always of great concerns as it directly affects the control of critical dimensions of etched images. Consequently, the HMDS process has also been implemented in various coater tracks with varying degrees of success.

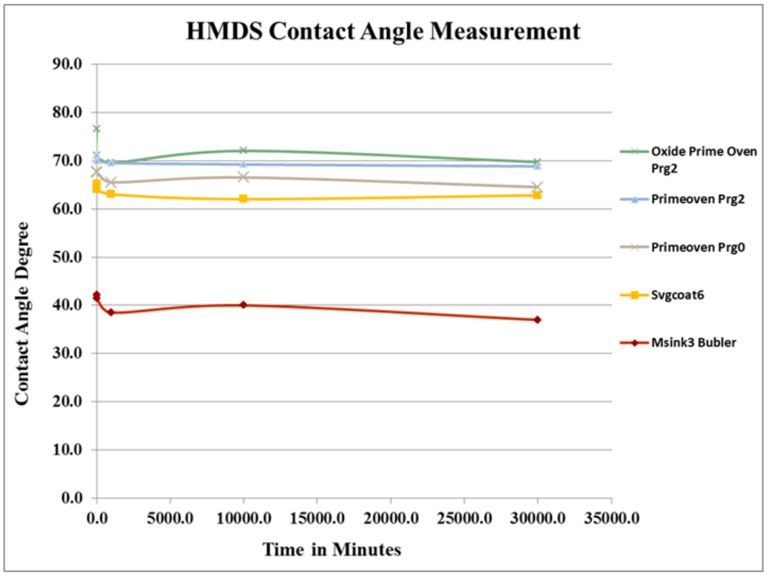
II.A. SVG Coater Track versus YES HMDS Vapor prime process

HMDS priming effect of the various commonly used HMDS application methods were studied at UC Berkeley's Marvell Nano Lab by Kate O'Brien. During her studies the effect of the different HMDS techniques were monitored by comparing water droplet contact angle behaviors on the treated samples using various techniques. The contact angle images and the contact angle data collected from three different YES HMDS Vapor prime procedures with an SVG coater track and a bubbler tank immediately after the treatments are presented in **Figure 2**. All three YES HMDS procedures demonstrated superior contact angle behaviors compared the wet tank with bubbler and the popular SVG coater tracks.

HMDS Application Method	Contact Angle Measurement *Taken Immediately*	Kruss Contact Angle Image
Msink3 Bubbler for 1 minute	42.2	
Svgcoat6 for 1 minute	65.2	
Primeoven Program 0 for 1 minute	67.7	
Primeoven Program 2 for 2 minutes	71.1	
Primeoven Program 2 *on an Oxide Wafer* for 2 minutes	76.5	

***Figure 3: T**he comparison of contact angles with the associated contact angle images obtained from various HMDS application methods collected immediately after each treatment

During this study the contact angles were also monitored for up to three weeks and the results are presented in Figure 3. All samples demonstrated good long term surface stability with the consistently superior behaviors from all three samples primed using YES HMDS vapor prime oven.

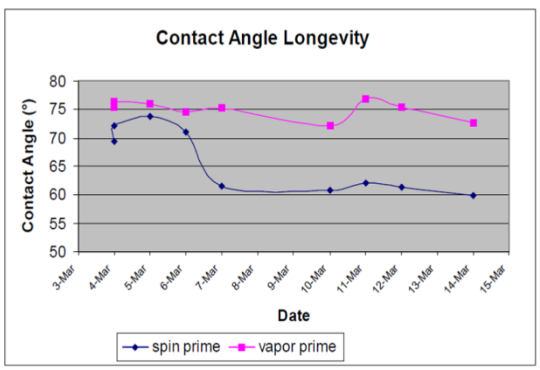


*** Figure 4: T**he long term contact angle behavior from the samples treated with various HMDS Techniques.

****Figures 1, 3 and 4*** are Re-printed with permission from the UC Berkeley's Marvell Nano Lab, from a report by Kate O'Brien on HMDS application methods.

II.B. Spin Coating versus YES HMDS Vapor prime process

A similar longevity comparison studies between the YES vapor prime and the conventional spin prime procedures were conducted at the UTD and the experimental behaviors of the two methods are presented in Figure 4. The plots in **Figure 4** shows stable contact angles on the vapor primed wafer for at least two weeks while the spin-on primed wafer's contact angles degrade below the recommended contact angle after three days.



** F**igure 5:** The contact angle stability and longevity behavior of the vapor primed wafer showing good stability at least two weeks, while the spin-on primed wafer's contact angles degrade below the recommended contact angle after three days.

III: HMDS PROCESS CHARACTERIZATION IN A DOE

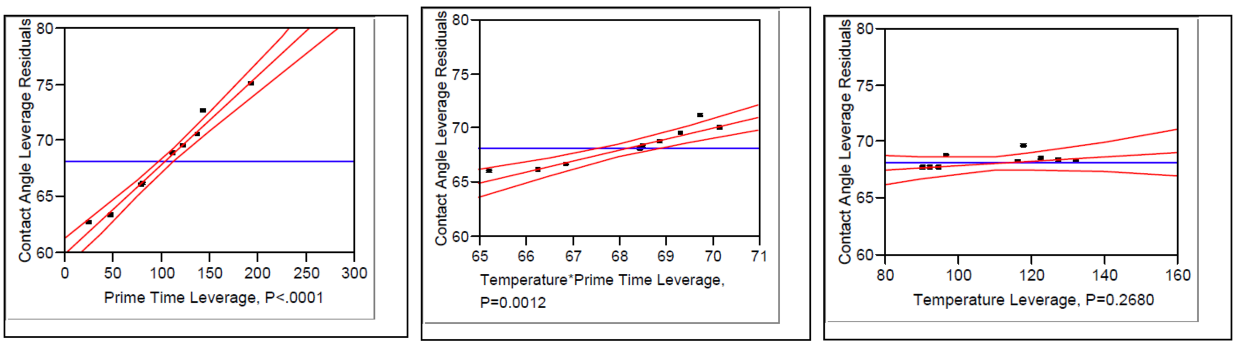
Statistically designed experiments (DOE's) are a popular technique in semiconductor process characterizations. The reported DOE's for the HMDS process all show good agreement with each other on the effects of dehydration bake, dispense time, dispense flow rate, and chemical reaction time**.** In a similar fashion, Statistical experiments were designed to study the Effects of Oven Temperature (°C) and the Duration of the Prime (sec.) and the interaction of the Temperature * Time.

The Designed experiments were conducted UT Dallas clean room and the contact angle data was collected for the effects and interaction analysis. The response of the DOE is the post process Contact Angle (°) collected during the experiments. SAS Institute JMP 4.0 software was used to model the process results collected. Please refer to the document "HMDS Process Setup" # SP2003-LI-001 Copyright © 2003 The University of Texas at Dallas, for experimental details.

** **Table 2.** Effect Tests Summary

Source	**Nparm**	**DF**	**Sum of Squares**	**F Ratio**	**Prob > F**
Temperature	1	1	0.79757	1.5522	0.2680
Prime Time	1	1	**135.37852**	**263.4612**	**<.0001**
Temperature*Prime Time	1	1	1 22.63740	263.4612	0.0012

The summary of Test results on the the various effects and interactions are presented in Table 2. Table 2 numerically shows that Vapor Prime Time is the largest effect on contact angle followed by the interaction of Temperature*Prime Time, and lastly Temperature. These effects are graphically depicted in **Figures 6** (a), (b), and (c) respectively.



(a) Prime Time (b) Temperature* Prime time (c) Temperature

****Figure 6:** Vapor Prime Time effects are graphically depicted in figures a, b, and c respectively.

***** DOE Experiments results in Table 2, Figures 5 and 6*** are Re- printed with permission from The Erik Jonsson School of Engineering and Computer Science, at University of Texas at Dallas, Document "HMDS Process Setup" # SP2003-LI-001 Copyright © 2003 The University of Texas at Dallas

IV: YES HMDS PROCEDURES:

Chamber pump purge cycles*:* The deposition process begins with the pump and purges cycles of the150C pre heated vacuum chamber after an initial bake of up to 10 min to properly dehydrate the substrates. The chamber is evacuated to low pressure and refilled with pure nitrogen several times to completely remove water vapor. The initial programmable preheat dehydration step provides enough heating time to pre heat the wafer to 150°C process temperature. A typical YES-58TA HMDS Vapor Prime Process Cycles are presented in **Figure 9**.

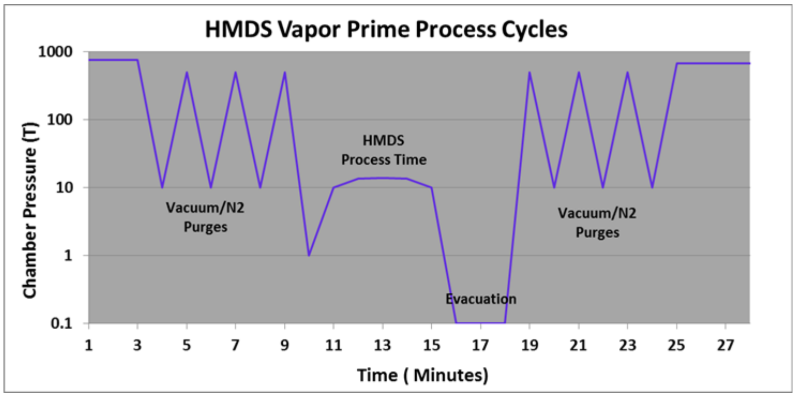


Figure 3: Typical YES-58TA HMDS Vapor Prime Process Cycles

***Vapor Prime**:* Once cycle purges are finished, the process chamber is taken to lower 1 T pressure, a higher vacuum than the flask holding the HMDS chemical before opening the vapor valve. When the vapor valve is open, the chamber draws the chemical vapor over into the chamber. The HMDS vapor then react with the wafers. A 5 min vaporization time is often a wafer fab standard for priming bare Si wafers. The vaporization time is adjustable and can be increased as needed to ensure priming of various types of surfaces.

Process Flow Chart: The overall dehydration and the prime process can be grouped into four distinctive process steps as presented in **Figure 2**.

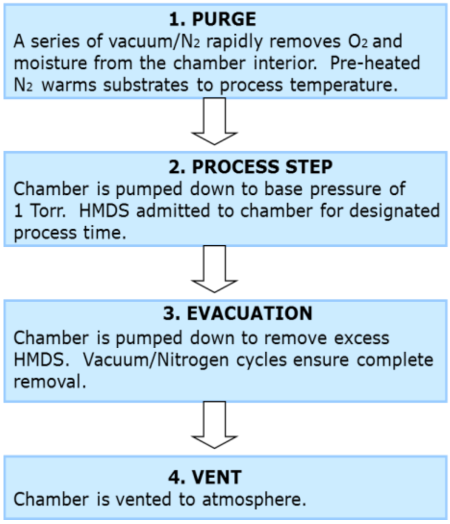


Figure 2: YES-58TA HMDS Vapor Prime Process Flow Chart

HMDS Process Verification: The process results are usually verified by including monitor wafers in the process chamber and monitoring the pre and the post process contact angles using a goniometer.

V. CONCLUSIONS

Water droplet contact angle measurement is a practical way to indicate adhesion. The DOE results clearly indicate prime time to be the most significant in vapor prime process. It is clear from the DOE work that water contact angle can be improved and optimized with vapor priming process optimization.

Contact angle behavior comparison studies conducted at UC Berkeley and UT at Dallas clearly and consistently indicate the superiority of YES HMDS Vapor priming capabilities with pre-process dehydration capabilities compared to SVG coater track and spin coating dispensing.

HMDS is a carcinogen and exposure should be avoided. In the YES HMDS oven, the user is not exposed to the HMDS vapors, and less than 1ml is used to prime up to 200 wafers. Vapor prime is therefore safer, less expensive and as shown in experimentation achieves superior contact angles with superior long term stability compared to coater track or spin dispense procedures.

Photolithography is an integral part of the semiconductor manufacturing process. It is used to delineate patterns representing particular device or circuit structures on the surface of a silicon wafer. These patterns are made with photoresist masks, which protect the substrate underneath it from subsequent processing. The physical or electrical characteristics of the unprotected surfaces are altered by various types of process steps such as etch, deposition, ion implantation, sputtering, etc. This cycle is repeated many times until the entire device is completed. Photolithography is a time consuming and a costly process. Inadequate adhesion usually results in PR lifting, pattern distortion, and the process needs to be repeated again after the resist has been stripped off. If the YES-HMDS oven vapor priming process can eliminate one lithography rework process in two weeks, due to their superior adhesion properties compared to HMDS priming using a coater track, it adds up to a significant cost and time savings to any fab.

Yield Engineering Systems would like to thank the UC Berkeley and UT at Dallas for their permission to re-print their YES Vapor prime tool related experimental results.