PHOTOMASK

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PM15 1st Place Best Paper

Fabrication of a full size EUV pellicle based on silicon nitride

Dario L. Goldfarb, IBM T.J. Watson Research Center, 1101 Kitchawan Rd, Yorktown Heights, NY 10598

ABSTRACT

In this paper, the fabrication and initial characterization of an unsupported membrane composed of a single ultrathin silicon nitride (SiNx) layer with potential application as a EUV pellicle is described in detail. A full size free-standing pellicle with inner film area equal to 113x145mm and champion EUV transparency equal to 89.5% (single pass) is demonstrated utilizing the methodology presented in this study. The exemplary EUV transparency of the reported pellicle was achieved by limiting the membrane thickness to 16nm, while the intrinsic mechanical stability for the silicon nitride film was realized by adjusting the Si:N ratio to provide a non-stoichiometric layer featuring low tensile stress.

The pellicle thickness, elemental composition and mass density were used to calculate the expected EUV transparency, which was found to be in good agreement with experimental EUV transmission measurements. Additionally, careful consideration was given to process-induced mechanical instabilities exerted on the ultrathin pellicle during the wet etch, rinsing and drying fabrication steps, and a unique yet simple set of ancillary hardware, materials and processing techniques was introduced to minimize such disturbances and yield large-area pellicles that are free of visible defects and wrinkles. In the absence of commercially available actinic inspection tools, a distinctive advantage of the SiNx membrane versus a Silicon-based EUV pellicle solution is the demonstrated ArF transmission, making it attractive for through-pellicle mask defect inspection and advanced metrology work utilizing available 193nm excimer laser and detection systems. A preliminary heat load test indicates that the SiNx-based EUV pellicle would be marginally compatible with an equivalent 80W EUV source.

1. Introduction

1.1 Ultrathin EUV Pellicle - Motivation

Since a pellicle layer was first proposed in 1978^[1] as a means to control reticle defectivity during lithographic patterning, the semiconductor industry has adopted the use of such protective films for every optical wavelength utilized for chip manufacturing. With the advent of EUV lithography, several elemental pellicle material candidates have been proposed, including silicon (polycrystal-line^[2,3], monocrystalline^[4], porous^[5,6]), carbon (graphite^[7], grapheme^[8,9], nanotubes^[10]), and Si-Mo-Nb multilayers^[2]. The basic requirements for any EUV pellicle material are singlepass EUV transmission of at least 90%, thermo-mechanical stability under dynamic exposure conditions, and chemical in-

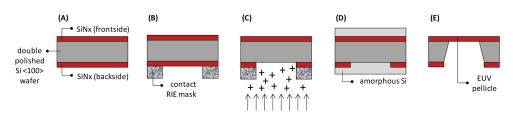


Figure 1. EUV pellicle fabrication process. (A) Silicon nitride deposition; (B) Dry etch mask placement; (C) Reactive ion etch; (D) Protective a-Si layer for wafer dicing; (E) Silicon wafer wet etch.



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EDITORIAL

The interesting life of 193

Paul Ackmann, GLOBALFOUNDRIES, Inc.

2015 is coming to a close. There have been many interesting activities already covered in our editorials this year. In considering what to include in this editorial, I decided to avoid any previous topic. Looking at past editorial headlines: technical talent loss to the community, industry consolidation, a new roadmap update for the mask business, and the continued progress of EUV toward insertion, I decided to focus on the 21st century's biggest lithography event: the 193nm mask business to date.

The spectacular extension of 193nm lithography into the shrink path has been the technology driver for the mask industry. 193nm Lithography supplanted the 248nm scanner workhorse as the critical patterning tool in the early 2000's. The 193nm tool suppliers have successfully extended this wavelength by the water immersion. There were many technology options to augment, improve, or replace simple mask with more features such as pixelization, attenuated shifter, hard shifters and other mask techniques to drive lithography process windows in the wafer fab. Approaches, which would allow less OPC and simpler masks depended upon the introduction of 157nm and EUV lithography. Many worried that 193nm lithography was only a flashin-the-pan and, at most, a two or three node solution. The lithography community expected 157nm and then EUV to become High Volume Manufacturing (HVM) solutions with 193nm relegated to "block" layers or removed completely. As we all know, immersion landed at 1.35NA; 157nm lithography went away; and EUV continues to progress towards insertion.

It is likely that 193nm lithography will extend to a 7th or 8th major logic node and will continue even when EUV moves to HVM. However, while the extension of 193nm lithography has been great, the complexity of the reticles due to the increased masks per node requirements drives high cost.

Advanced illumination optics has extended the capability on new scanners which created higher data density on each mask and therefore shot count ("pixel") increased. More shots meant longer write times. New writers dealt with the higher "pixel" count with better tool performance for each new technology node. The 193nm wavelength has created some unintended consequences for old Chrome on glass (COG) mask material (Chrome Migration) that was solved with the more advanced MoSi based offerings. The extension of 193nm has added other complexities to mask makers. Looking forward, some worry that Manhattan layouts have limited design and mask making and free form (curvilinear) designs may be needed which will add to even longer write times. Multi-Beam writers are the next evolution to address many aspects of sub-10nm 193nm and EUV reticle manufacturing.

We must remember the hard work done to continue the roadmap with 193nm immersion lithography. We, in the mask industry, spend much of our time wondering why there are fewer mask houses; poor cost recovery model for commercial suppliers; or any other myriad of complaints. Many are real issues and their impact depends upon the observer's perspective in the industry. Regardless of position, looking back at mask tool and Fab enablement, presents an interesting and successful story. Mask enablement to support the ITRS roadmap will continue. Some day we will have EUV or another enabling technology. Until then, 193nm optical reticles rule.

Thanks again for supporting the mask community through BACUS in 2015 and now moving into 2016.



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P.O. Box 10, Bellingham, WA 98227-0010 USA Tel: +1 360 676 3290 Fax: +1 360 647 1445 www.SPIE.org help@spie.org

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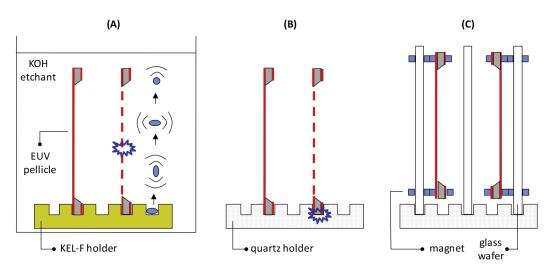


Figure 2. EUV pellicle failure mechanisms and solution during wet etching and handling. (A) Coalescence and dislodging of bubbles from hydrophobic surfaces creating pressure waves on pellicle surface; (B) Mechanical friction between pellicle frame and substrate holder; (C) Processing hardware and mounting configuration designed to overcome the issues indicated in (A) and (B).

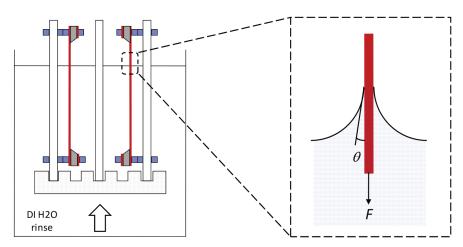


Figure 3. EUV pellicle assembly being withdrawn from the DI H₂O rinse tank. Inset: downward pulling force F acting on either side of the membrane along the liquid contact line as a result of the capillary effect.

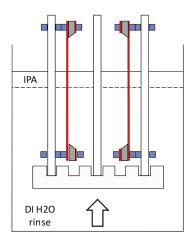
ertness towards ionizing radiation in the presence of a low-pressure hydrogen atmosphere. Due to the reactive environment inside the EUV scanner, a capping layer (3-5nm thick) such as silicon nitride or ruthenium is usually added to protect the core material, which needs to be budgeted in the overall pellicle performance. A capped free standing EUV pellicle based on polycrystalline silicon (44-65nm thick) has been extensively characterized^[2,3], showing EUV transmission larger than 85% and acceptable mechanical properties and imaging performance, but requiring improvement in relation to its heat load resistance at ultimate incident EUV peak power (5.6 W/cm²). Since silicon is the pellicle material candidate with the lowest EUV photoabsorption cross-section, any alternative material combination would require the use of much thinner films to achieve the desired transparency, thus making the fabrication of a full size (113x145mm) pellicle a challenging task.

In this work, the fabrication of a full size EUV pellicle based on ultrathin silicon nitride is undertaken. Historically, small silicon nitride membranes have been used in MEMS technology^[11] and X-ray lithography^[12], and a large body of information is available regarding its compositional, physical, thermal, mechanical and electrical properties. Being used as an EUV capping layer already, silicon nitride constitutes a suitable learning vehicle to understand the limitations to the manufacturing of unsupported films featuring ultralarge area-to-thickness ratios, and to develop the hardware and processing steps that would enable the successful production of such ultrathin membranes for application as EUV pellicles.

2. EUV pellicle fabrication process

The fabrication process of ultrathin silicon nitride pellicles is





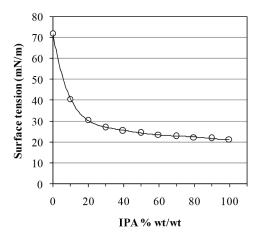
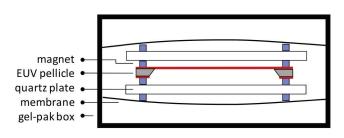


Figure 4. EUV pellicle assembly being dried under low surface tension conditions enabled by an isopropanol (IPA) layer floating on top of the DI H2O rinse liquid (left). Surface tension for IPA-water mixtures showing low $y_{_{1V}}$ conditions (20-30mN/m) across a large dilution range^[30].



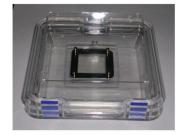


Figure 5. Cross-sectional sketch of EUV pellicle packaging used for storage and transport (left) and actual assembly (right).

schematically summarized in Figure 1. Steps 1A and 1D were carried out in a cleanroom environment. The remaining steps took place in a research lab without environmental control. A detailed description of the different processing steps, including ancillary hardware, materials and processing techniques is provided in the remaining of Section 2.

2.1 Substrate Preparation

Substrates consisted on 200mm double-polished (100) silicon wafers (p-type, 730µm thick) that were pretreated with a modified RCA clean. A first cleaning chemistry removes organic contaminants and passivates the silicon surface by forming a thin layer of hydrous oxide by using a 40:1:1 mixture of water, hydrogen peroxide, and ammonium hydroxide at 35°C. A second cleaning chemistry removes ionic contamination with a 100:1 mixture of water, and hydrochloric acid, followed by a water rinse and spin dry.

2.2 Silicon Nitride - Film Deposition

Silicon nitride films were simultaneously deposited on both substrate faces (Figure 1A) utilizing a standard low pressure chemical vapor deposition (LPCVD) process comprising the reaction of ammonia (NH₃) and dichlorosilane (SiH₂Cl₂) at 760°C in a dedicated horizontal tube furnace, yielding films with acceptable thickness uniformity for the purposes of this work (4% 3σ). Improved film thickness uniformity has been reported by replacing SiH₂Cl₂ with silane (SiH₃)^[13]. The obtained films display excellent chemical re-

sistance towards alkaline etchants and act as a wet etch masking material for both wafer faces in the subsequent fabrication step. At the same time, the films constitute the EUV pellicle to be fabricated (wafer frontside). Typically, films with 20-25nm nominal film thickness were deposited. Film thickness and optical constant determination for the as-deposited films was performed in the UV-visible spectrum range (153-803nm) with a variable angle spectroscopic ellipsometer (VASE) by applying a Cauchy model to the experimental data.

2.3 Silicon Nitride - Composition and Mechanical Properties

The application of a thin suspended membrane as a protective pellicle requires the precise control of its mechanical properties during fabrication and utilization. EUV pellicles are required to maintain a tensile stress in order to avoid the formation of wrinkles which may impact imaging quality. It has been noted that such wrinkles would result in an apodization effect^[2], with an estimated maximum local angle allowed at the pellicle plane no larger than 200mrad to ensure a critical dimension uniformity (CDU) impact below 0.1nm. On the other hand, an upper limit to the tensile stress is given by the material fracture strength. For the case of silicon nitride, tensile stress values in excess of 1GPa are attained for a stoichiometric composition (Si_3N_a), while fracture strength values up to 14 GPa have been reported^[14]. This extremely high failure stress is contradicted by experience with etched membranes where failure stresses are found below 1GPa. The discrepancy is attributed



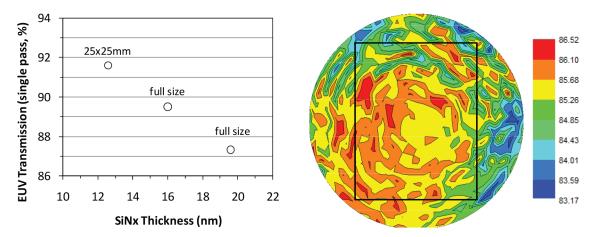


Figure 6. EUV pellicle transmission data calculated based on measured pellicle thickness, atomic composition and density, along with the accomplished pellicle size (left). EUV pellicle transmission uniformity (%, single pass) over inscribed full size pellicle area (113x145mm) inferred from film thickness measurements for a LPCVD SiNx film deposited on a 200mm silicon wafer and immersed in aqueous KOH for the duration of the wet etch process (right).

to stress concentration at the sharp corners in anisotropically etched specimens $^{[15]}$, and therefore rectangular membranes are more difficult to stabilize mechanically with respect to a circular geometry of comparable area. During the course of this work, a $\mathrm{Si_3N_4}$ composition with tensile stress larger than 1GPa has been shown to be incompatible with the fabrication of thin membranes (< 25nm) larger than 50x50mm.

The mechanical stress of LPCVD-deposited silicon nitride has been shown to be easily modulated by its elemental composition $^{[16]}$, with an approximately linear tensile stress variation from $+1.0 \, \text{GPa}$ to a relaxed layer (zero stress) for Nitrogen:Silicon (N:Si) ratios ranging from 1.33 (Si $_3 \, \text{N}_4$) to 1.0 (SiN). In this work, a siliconrich silicon nitride film (SiNx, x=1.1) with calculated tensile stress equal to $+300 \, \text{MPa}$ was selected, which allowed for the fabrication of stretched out membranes that were wrinkle-free, without compromising their intrinsic mechanical stability. The tensile stress for the as-deposited SiNx films was experimentally verified by measuring the wafer curvature change pre- and post-deposition of the film. Since both sides of the wafer received a coating, a selective removal of the SiNx film from one of the wafer faces was introduced between measurements.

2.4 EUV Pellicle Area Sizing

The fabrication of SiNx membranes of arbitrary sizes was achieved by designing contact etch masks made out of diced (100) silicon wafers. The silicon wafers were cleaved to cut out open areas that equaled the intended size of the EUV pellicle. The remaining silicon pieces were bonded together using kapton tape to form the masking material, thus providing a simple yet durable and reusable template for the dry etch removal of the backside SiNx area where the pellicle is intended to be located. The dry etch mask was placed in direct contact with the wafer backside (Figure 1B) and the wafer notches were made to coincide to make sure that the crystallographic planes of the pellicle substrate were aligned with the diced edges of the etch mask. The wafers were affixed with a Teflon guard wrapped around the edges of the contacted samples, which also served the purpose of raising the pellicle wafer surface in the reactive ion etch (RIE) chamber to avoid scratching the frontside SiNx film against the bottom plate electrode.

The wafer assembly was manually loaded into a Unaxis 790 RIE chamber and the backside SiNx was removed using $\mathrm{CF_4/O_2}$ etch chemistry (pressure=100mtorr, $\mathrm{CF_4}$ flow=25sccm, $\mathrm{O_2}$ flow=3sccm, power=100W) without the need of endpoint detection (Figure 1C). Typical etch rates for the SiNx layer were in the order of 20nm/min.

2.5 EUV Pellicle Frame Sizing

The outer dimensions of the pellicle frame were realized by dicing the etched wafers by mechanical sawing utilizing an automated dicing saw. In order to protect the sensitive frontside and backside areas during the sawing process, a sacrificial dummy layer composed of amorphous silicon (a-Si) was deposited on top of the SiNx films before cutting the wafers (see Figure 1D). The a-Si layer (500nm) was deposited in a separate LPCVD furnace from SiH₄ gas at 525°C. In this work, pellicle frame width was limited to 5mm in order to facilitate pellicle handling, however it should be possible to produce a smaller frame width within production specifications (2-3mm) using the described methodology.

2.6 Substrate Wet Etch - Materials and Processing

The final step in the fabrication of the EUV pellicle based on ultrathin SiNx is accomplished by selectively wet etching the a-Si protective layer and the silicon substrate containing the pellicle material layer to create the desired freestanding membrane (Figure 1E). The remaining backside SiNx acts as masking material for the underlying silicon which makes up the pellicle frame material. The wet etch process utilized a 25% w/w potassium hydroxide (KOH) aqueous solution^[17] instead of the alternative tetramethylammonium hydroxide (TMAH) or ethylenediamine-pyrocatechol (EDP) formulations typically used for CMOS applications^[18] due to cost, toxicity, safety, and waste disposal considerations.

The processing temperature of the wet etch process was selected to minimize pellicle failure. A high wet etch processing temperature (60-80°C) during the fabrication of full size pellicles increased the chance of disruption of the SiNx layer. Failure seemed to originate at the Si/SiNx interface and was evidenced by the appearance of cracks propagating in a discontinuous trajectory across the wafer surface. The fundamental cause of such phenomenon is still under investigation. These detrimental



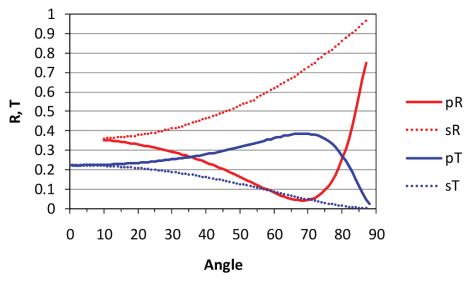


Figure 7. Transmittance (T) and reflectance (R) measured on a 20nm SiNx membrane using s- and p- polarized light at λ =193nm at variable angle of incidence.

events were minimized by operating at a temperature below 50° C. Typically, wafers were etched at 46° C in an agitated tank with a calculated etch rate of 10μ m/hr.

The anisotropic etching mechanism of silicon wafers in alkaline solutions is well documented [19] and several features have direct impact on the reliability of the EUV pellicle fabrication process. During silicon dissolution, the (100) plane is preferentially removed over the (111) plane via oxidation by hydroxyl ions (OH)⁻ to form a silicate, accompanied by the reduction of water to liberate H2 bubbles. It is noted that the active etching species is H2O and that the (OH)- ion plays a role as a catalyst [20] and is therefore not consumed during the reaction:

Si + 4 H2O \rightarrow 2 H2 + Si(OH)4 \rightarrow soluble silicates

Implicit in the overall reaction is the formation of transient Si-H surface groups and their hydrolysis to form silanol (Si-OH) moieties [21]. The presence of Si-H groups imparts a hydrophobic nature to the etching surface, which leads to the attachment of hydrogen bubbles and blocking of the reacting area, manifesting in the formation of protrusive microscopic pyramidal hillocks and uneven surface removal. The creation of topographic features on the silicon surface during dissolution amplifies the described phenomenon by promoting further bubble attachment. This effect is mitigated by the competing hydrolysis which favors the formation of a hydrophilic surface and bubble detachment [22]. The significance of bubble formation and adhesion in relation to EUV pellicle fabrication is paramount. During the last stages of silicon removal, the suspended membrane consisting of SiNx and the residual silicon becomes very thin and is subjected to the intrinsic buoyant force imparted by the attached gas bubbles. The compounded force of multiple attached bubbles can easily dislodge the pellicle from the frame edge in the case of free-standing films with ultrahigh area-to-thickness ratios.

Different methods have been applied to prevent hillock formation during silicon wet etch, such as the addition of oxidants (oxygen^[23], peroxides^[24]) or surface wetting agents (surfactants^[25], isopropanol^[26]). In this work, provisions to reduce the rate of hydro-

gen evolution and avoid the formation of a hydrophobic surface and surface roughness during substrate wet etch were made for the fabrication of large membranes. Firstly, the KOH etchant was allowed to cool down to 20-25°C as the silicon etch process approached completion, in order to reduce the rate of hydrogen production, and therefore the size and quantity of the gas bubbles. Additionally, the KOH etchant was replaced periodically between wet etch runs to avoid excessive build up of soluble silicates that might affect the wetting properties of the silicon surface. Last, oxygen gas was frequently bubbled through the KOH etchant in order to compensate for the degassing effect of the evolving hydrogen bubbles.

Caution! - Reports of a pungent odor emanating from the KOH etchant bath during silicon dissolution have been communicated^[27], indicating that besides H₂, which is odorless, some other gaseous reaction product is formed. Chemical analysis indicates the likely presence of SiH₄ gas in ppm concentrations^[28]. It is recommended that the process discussed above be carried in an exhausted space.

2.7 Substrate Wet Etch - Hardware

The subtractive process described in the previous section requires that the ultrathin unsupported membrane be able to withstand the forces exerted on its surface during etch, rinse and drying steps without breaking. Such forces include mechanical vibrations induced during handling of the pellicle, pressure differentials on either side of the membrane created by convective flow during etching, and capillary forces due to meniscus formation at the liquid-air interface during drying. Managing the mechanical stability of the ultrathin membrane in the presence of such forces is particularly challenging given the large area of the EUV pellicle.

Among the extrinsic forces exerted on the EUV pellicle surface, pressure waves generated by buoying bubbles originating from dissolved gases (H_2 , N_2 or O_2) present in the etchant solution and dislodged from hydrophobic surfaces can be particularly problematic. This issue is solved by utilizing processing hardware with a hydrophilic surface where gas bubbles would not easily coalesce. A quartz substrate holder was consequently designed to hold multiple pellicle substrates, while a quartz tank was built



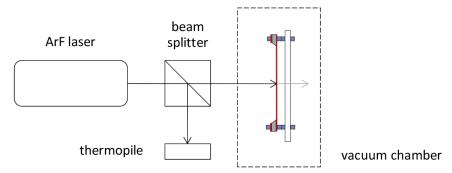


Figure 8. Schematics of the experimental setup used to measure the thermal stability of EUV pellicles.

to contain the etchant solution. This combination proved to be very effective since quartz has relatively good corrosion resistance towards hot concentrated KOH and is perfectly wetted by the aqueous etchant. The need for quartz materials resides also in its low triboelectric charging properties, as opposed to other corrosion-resistant materials that are hydrophobic and have a tendency to become electrostatically charged (e.g. PTFE, PFA, KEL-F). Statically charged surfaces have proven to be disruptive if placed in close proximity to a large area pellicle.

Yet another source of failure during pellicle fabrication was due to mechanical vibrations induced during pellicle handling. Friction between the pellicle frame edge and pellicle-holding articles such as the pellicle holder grooves can create mechanical perturbations that can easily break large pellicles. It was therefore necessary to physically separate and mechanically isolate the pellicle from the substrate holder in order to avoid rubbing the pellicle frame against the holding piece. This was achieved by utilizing glass wafers as ancillary supporting structures for the pellicle substrates to be wet etched. The diced substrates were affixed to the glass wafers by means of magnets prior to immersion in the KOH etchant solution, which made the fabricated pellicles impervious to mechanical perturbations at the time of dismounting the etched membranes from the substrate holder. The glass wafers were typically 200mm in diameter and resistant to KOH attack. The magnets used to mount the EUV pellicle were composed of a Iron-Neodymium-Boron alloy (FeNdB, "rare earth magnets") coated with a hydrophilic corrosionresistant layer (Nickel) which provided the necessary protection against oxidation and dissolution in KOH. Several magnets were assembled together to create a gap between the pellicle and the wafer. Mechanical isolation was achieved by means of the described assembly, since any mechanical vibration generated on the glass wafer would not efficiently propagate through the magnet stack to the pellicle frame (Figure 2).

2.8 EUV Pellicle Rinse

Once the silicon substrate exposed to the KOH etchant is completely removed, the SiNx films become freestanding, visually evidenced by its optical transparency. The removal of the ultrathin wetted membranes from the etch tank is a delicate task, as any unstable movement during withdrawal would impart an overwhelming lateral pressure that would destroy the membranes. It is therefore crucial to utilize a motorized handling system that can vertically remove the pellicle holder from the etch tank in a steady fashion without any lateral displacement that could destabilize the membranes. In this work, a mechanical arm was used to lower and raise the pellicles at variable speed, ranging from 0.1mm/min

to 120mm/min. Pellicles were usually removed from the wet etch tank at a rate of 90 mm/min.

As the etched pellicles come out of the wet etchant, a continuous film of aqueous KOH is formed on both sides of the pellicle. The integrity of this film is maintained by the high hydrophilicity of the SiNx membrane surface, which overcomes the tendency of the liquid film to break down due to surface tension. The thickness of the formed aqueous layer can be calculated using the Landau-Levich-Derjaguin equation^[29]:

$$H = 0.94 \; \frac{(\eta.U)^{2/3}}{\gamma_{LV}^{1/6} (\rho.g)^{1/2}} \label{eq:Hamiltonian}$$

where H is the liquid film thickness, η is the viscosity, U is the withdrawal speed, y_{iv} is the surface tension, ρ is the density and gis the acceleration due to gravity. Applying literature data^[30] to the above equation for aqueous KOH of concentration comparable to the one used in the present work1, an estimated liquid film thickness equal to 6µm is calculated for each side of the membrane. It is remarkable that the ultrathin SiNx pellicle is able to integrally support a liquid film about 400 times its thickness on each side of the membrane without breaking. In fact, the pellicle survives the transfer from the wet etch tank to the rinse tank only if the liquid film is not allowed to be disrupted. The pellicle is then required to be transferred swiftly to the rinse tank in order to avoid incipient drying, which can be potentially accelerated by air circulation in the exhausted environment. In fact, the presence of the ancillary glass wafers interposed between each membrane as shown in Figure 2C helps to protect them from air currents and reduces film evaporation.

The SiNx membranes are then lowered into the DI $\rm H_2O$ rinse tank at a speed of 120mm/min utilizing the same robotic arm mechanism formerly described, since identical mechanical stability considerations are in effect when immersing the pellicles in the rinse bath. The pellicles are soaked in the DI $\rm H_2O$ tank for 8-12 hours without disturbing the rinse liquid, in order to dilute and eliminate the residual KOH from the membrane surface. Shorter soak times are possible however this processing variable was not investigated in detail during the course of this work. The higher density of the aqueous KOH solution compared to DI $\rm H_2O$ helps drive the displacement of the residual etchant from the pellicle surface and replace it with the rinse liquid.

2.9 EUV Pellicle Drying

Among the forces exerted on the EUV pellicle surface during the fabrication process, the most difficult to deal with is the presence of a capillary force during the withdrawal of the membrane from

 $N \bullet E \bullet W \bullet S$

Table 1. Summary of EUV pellicle material properties, fabrication and characterization.

EUV Pellicle	Materials & Hardware	Comments
Substrate	200mm Si wafers (<100> orientation)	Double polished, pre-cleaned
Film deposition	LPCVD SiNx	20-25nm as deposited, stress=300MPa (tensile)
	$(x=1.1, NH_3 + SiH_2Cl_2, 760^{\circ}C)$	4% 3σ film thickness uniformity
Pellicle Frame	5mm width	Wafer dicing (a-Si protection layer, 500nm)
Substrate wet etch	KOH 25% wt/wt (46°C), all-glass hardware	10nm/hr - Final cool down to room temperature
Pellicle rinse	DI H ₂ O, undisturbed tank	8-12 hour soak
Pellicle drying	Novel low surface tension drying technique	isopropanol layer, 1.8mm/min withdrawal rate
Champion EUV	SiNx, thickness=16nm	EUV transmission=89.5% (single pass)
pellicle (full size)	(113x145mm)	EUV transmission uniformity= 1.9% (3σ)
ArF transmission	38% (λ=193nm; single pass)	p-polarized; incident angle ~ 69° (NA~ 0.93)
Heat load testing	EUV=1.85W/cm ² (82W source, vacuum)	Improvement in heat dissipation properties of
(tolerable peak power)	$ArF > 7.0 \text{W/cm}^2 @ 1 \text{Atm}$	EUV pellicle are required

the DI $\rm H_2O$ rinse tank. The meniscus formed at the liquid-gas-solid boundary as a result of the surface tension y_{LV} of the rinse liquid on both sides of the partially immersed membrane results in a net downward pulling force (F) described by the Wilhelmy equation^[31]:

$$F = 2.\gamma_{LV}.L.\cos\theta$$

where L is the length of the contact line along the membrane and θ is the contact angle (Figure 3).

Due to the large surface tension of water (y_{LV} =72mN/m) and the hydrophilic nature of the SiNx surface ($\theta \approx 0^{\circ}$) a high capillary force ensues, which has been demonstrated to be able to break even small EUV pellicles while being withdrawn from the DI H₂O rinse tank.

In order to overcome the membrane instability induced by the capillary force, several alternative drying techniques were considered. Most known methods that minimize the surface tension $y_{_{LV}}$ of the drying liquid require forcing a fluid flow (Marangoni drying, critical point drying), involve significant volumetric changes (freeze drying), or require specialty non-volatile chemicals that might leave a residue on the EUV pellicle surface (surfactanated rinses, Langmuir-Blodgett films). Upon testing the most amenable processes, none were able to provide a workable solution that could yield an ultrathin EUV pellicle equal to or larger than 75x75mm, due to perturbations to the drying membrane which resulted in mechanical failure before the pellicles could be fully removed from the DI H₂O rinse tank.

It was therefore necessary to devise a drying method that can lower the surface tension $y_{\scriptscriptstyle LV}$ of the liquid to be used in the final rinse step without relying on forced convection (gas flow or liquid agitation) while at the same time effectively removing the DI ${\rm H_2O}$ rinse from the membrane surface, in order to successfully fabricate the ultrathin membranes. Consequently, a novel drying procedure was developed, consisting on forming a floating water-miscible organic layer on top of the DI ${\rm H_2O}$ rinse liquid, after the EUV pellicle has been soaked in the DI ${\rm H_2O}$ rinse bath. The organic layer is composed of a low surface tension fluid such as an alcohol, which remains stagnant during the EUV pellicle withdrawal process. The DI ${\rm H_2O}$ is displaced from the membrane surface as the EUV pellicle is withdrawn from the rinse tank and traverses through the stagnant alcohol layer, thus being dried under low surface tension conditions while emerging in the atmospheric environment.

The best organic solvents to implement the new drying process are those featuring low surface tension, good miscibility with water, high viscosity and density lower than water, such as light alcohols. Suitable alcohols for this purpose are isopropanol (IPA) and n-propanol² which are fairly inexpensive, do not pose a significant health risk and can be safely disposed of. It is noteworthy that being completely miscible with water, such solvents can form a distinctively separate top layer that is largely stable for a few hours due to their unfavorable mixing inertia, afforded by their high viscosity and density difference with H_2O . Typically, a 30-50mm thick floating alcohol layer enabled the displacement of the DI H_2O from the membrane surface and replacement by the low y_{IV} alcohol (Figure 4).

Alongside, the vertical surface tension gradient established across the diffuse interfacial region where both layers intermix serves as a driving force for the elimination of residual dissolved salt (KOH) in a similar fashion as shown for the Marangoni drying technique^[32].

An additional parameter that needs to be taken into account at the time of drying the EUV pellicle is the rate of removal of the EUV pellicle assembly from the DI $\rm H_2O$ rinse as shown in Figure 4. Due to the extreme thinness of the pellicle, any macroscopic droplet that might separate from the liquid being pulled is likely to disrupt the nanoscopic membrane. It is therefore necessary to perform the extraction of the EUV pellicle at a very slow rate in order to prevent the liquid meniscus from breaking off and leaving droplets behind. Typically, the membranes were withdrawn at a nominal vertical rate of 1.8mm/min in order to prevent mechanical failure based on the described mechanism. The film pulling and inertial instability effects just described are identical to those studied in detail during the development of immersion lithography and a wealth of information is available on this topic $^{(33)}$.

2.10 EUV Pellicle Storage and Transport

Handling of the fabricated EUV pellicle is not a trivial operation due to their fragility, especially for full size specimens. A simple, cost effective, and reliable method to safely store and transport un-mounted prototype EUV pellicle samples was devised, consisting on sandwiching the fabricated membranes between top and bottom protective dummy covers, each consisting of a quartz plate (1.6mm thick), and separated by magnetic spacers similar to those described in Section 2.7. The entire assembly is packaged in a gel-pak box, which provides added protection and mechanical isolation and can be easily mounted and dismounted (Figure 5). The packaging concept can be adapted to arbitrary pellicle areas and can be expanded to accommodate multiple vertically stacked specimens [34].

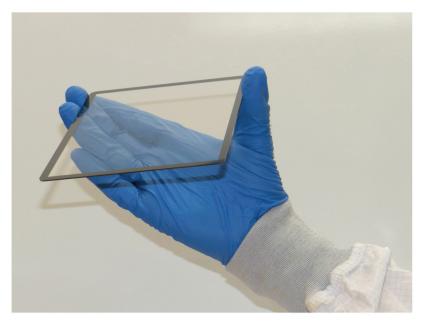


Figure 9. Full size wrinkle-free EUV pellicle based on unsupported SiNx membrane. EUV transmission= 87.3% (single pass); membrane thickness= 19.6nm; pellicle area= 113x145mm.

3. EUV pellicle characterization

3.1 EUV Transmission

EUV pellicle thickness was experimentally measured utilizing a VASE in transmission mode and applying the set of optical constants determined for the as-deposited SiNx films in Section 2.2. A separate optical constant determination for the SiNx material after KOH etch was performed on the pellicle frame yielding results identical to the as-deposited SiNx. A comparison of the SiNx film thickness values before and after wet etching indicates that a ~5nm total thickness loss is induced during the substrate wet etch. This value is only slightly higher than the ~4nm film thickness loss recorded for Si $_3$ N $_4$ under comparable wet etch conditions and is due to the small increase in silicon content in the case of the non-stoichiometric SiNx.

Given that the SiNx-based EUV pellicle is composed of a single material, the EUV transmission can be straightforwardly predicted if its atomic composition, mass density and thickness are known. A $Si_3N_{3,3}H_{0,7}$ stoichiometric formula having a N:Si ratio=1.1 and including the presence of H to compensate for the Nitrogen deficiency was used to describe the overall atomic composition. A literature value was assigned to the mass density of the ultrathin SiNx film (3.0 g/cm³), reported for a LPCVD SiNx coating having a N:Si ratio identical to the one utilized in this work[35]. EUV transmission (λ=13.5nm) was calculated using IMD software^[36], producing results equivalent to those obtainable from the CXRO database^[37]. Calculated EUV transmission agreed with experimentally measured values within 2%, the variability ascribed to small differences in SiNx density and N:Si ratio as a result of wafer position inside the LPCVD furnace[38]. Exemplary EUV transmission data along with the accomplished pellicle size is shown in Figure 6 (left).

Full size champion EUV transmission equal to 89.5% (single pass) was obtained for a 16.0nm thick pellicle. Full size pellicles with thickness below 16nm and EUV transmission above 90% (single pass) are within the realm of possibility; however their

fabrication has not been attempted yet.

EUV transmission uniformity for a full size pellicle was indirectly measured. A 200mm wafer coated with the low stress LPCVD SiNx without any backside patterning was immersed in aqueous KOH for the duration of the wet etch process, in which case no EUV pellicle was created due to the absence of exposed silicon. The wafer was rinsed and dried and a SiNx thickness map was obtained across the full wafer. The SiNx thickness was then narrowed to the area of the wafer corresponding to a full size EUV pellicle and converted into EUV transmission values (Figure 6, right). The EUV transmission variability calculated in such way was equal to 1.9% (3σ), which is larger than the preliminary EUV transmission spatial non-uniformity requirement (0.2%, 3σ) for high volume manufacturing[3]. It is noticeable that the largest contribution to the EUV transmission variability across the measured area is located near the periphery of the inscribed pellicle area. It is therefore anticipated that a pellicle manufacturing process would benefit from the use of larger wafers (e.g. 300mm), which should reduce the observed wafer edge effects and improve transmission uniformity.

3.2 ArF Transmission

The use of EUV photomasks in a semiconductor manufacturing environment requires their periodic inspection starting with its fabrication, but also during transport, storage, cleaning and usage. The proposed use of a polycrystalline silicon-based EUV pellicle would preclude the straightforward inspection of EUV masks due to its opacity at DUV wavelengths, thus requiring removal of the EUV pellicle every time a mask needs to be inspected. Additionally, the development of defect inspection tools based on actinic radiation ('at-wavelength') is lagging and is not expected to enter the commercial market for a few years [39]. Therefore, the use of EUV pellicles that are semi-transparent to ArF radiation (λ =193nm) would allow the inspection of EUV masks without the need to dismount the pellicle.

The SiNx pellicle herein described is particularly suitable for this



purpose. The transmittance (T) and reflectance (R) of a 20nm SiNx membrane was measured using a VASE with s- and p-polarized light at λ =193nm through a range of incident angles (Figure 7). It is noticeable that the SiNx pellicle achieves a maximum transmittance of close to 40% (single pass) of the incident light when using 193nm p-polarized light at incident angles around 70°, while at the same time minimizing light reflectance. Such high incidence angles are compatible with the high numerical angle (NA) illumination available in commercial mask inspection tools using ArF radiation. A detailed investigation of through-pellicle inspection of EUV masks including defect detection capabilities will be the subject of a future report.

3.3 Heat Load Testing

The extreme thinness of the SiNx-based EUV pellicle makes it susceptible to large temperature excursions during EUV exposure. Practical limitations to the use of a SiNx membrane as EUV pellicle are imposed by the transient thermal expansion and tensility loss of the material during exposure, while the vacuum environment inside the EUV scanner restricts heat dissipation to radiative cooling only. For the case of mask defect inspection using ArF radiation sources, the power requirements are more modest and only high brightness is required, when compared to the EUV scanner.

The ultimate peak power that a SiNx membrane can tolerate was experimentally determined utilizing an ArF laser exposure system as shown in Figure 8. The EUV pellicle (SiNx, 20nm) was placed inside the exposure chamber and evacuated to $<10^{-5}$ torr in a $\rm N_2$ environment. The laser power was gradually increased while the integrity of the pellicle was visually monitored. Pellicle failure under vacuum exposure conditions was observed at an equivalent total EUV peak power incident on the pellicle equal to 1.85 W/cm² (equivalent to a 82W EUV source power). When the same experiment was conducted at atmospheric pressure, the SiNx membrane was able to withstand the highest available power from the ArF laser, corresponding to a total ArF peak power incident at the pellicle equal to 7.0 W/cm², due to the additional conductive cooling provided by the surrounding atmospheric pressure environment.

The heat load results indicate that a SiNx membrane used as an EUV pellicle would be marginally compatible with the 80W EUV sources currently deployed and improvement in the heat dissipation properties of the EUV pellicle material is required. A complete analysis leading to the results included in this section is beyond the scope of this work and will be provided in a follow-up publication.

4. Summary and Conclusions

The fabrication of a full size EUV pellicle based on an ultrathin unsupported silicon nitride membrane has been carefully described in this work and is summarized in Table 1. Champion EUV transparency equal to 90% (single pass) and the absence of visible wrinking was achieved for free-standing SiNx membranes featuring low tensile stress and thickness equal to 16nm. Specialized processing hardware featuring a hydrophilic surface was developed as a means to control the formation of gas bubbles that could disrupt the stability of large-area pellicles during wet etch, and an original drying method was devised in order to overcome the capillary forces exerted on the EUV pellicle during the withdrawal from the DI H₂O rinse tank. The SiNx pellicles are characterized by 193nm transparency close to 40% when using ppolarized light at high incident angles, compatible with the illumination conditions found in advanced reticle inspection tools based on ArF radiation, thus making it possible to perform through-pellicle EUV mask inspection. The use of SiNx-based EUV pellicles is found to be marginally compatible with an 80W EUV source, based on heat load testing under vacuum conditions and therefore requires further improvement. Alternative pellicle materials with improved heat dissipation properties and concurrent EUV and DUV transmission are currently under investigation.

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Industry Briefs

■ Lam Research to Acquire KLA-Tencor

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Lam Research (LRCX) and KLA-Tencor (KLAC) announced that Lam Research is to acquire all outstanding KLA-Tencor shares in a cash and stock transaction. The move, unanimously approved by the boards of directors of both companies, will create a combined company with \$8.7 billion in pro forma annual revenue. The combined company expects to realize \$250 million in cost savings within 18 to 24 months of closing, and anticipates gaining approximately \$600 million in incremental revenue by 2020 through improved differentiation of each company's products and new capabilities.

The transaction is expected to close in mid-calendar year 2016 subject to customary conditions, including the adoption by KLA-Tencor's stockholders of the merger agreement and the approval by Lam Research's stockholders of the issuance of shares in the transaction. Given their complementary product lines and the industry benefits, the companies believe that they will be able to obtain the regulatory approvals.

Lam president and CEO Anstice concluded, "We have tremendous respect for the company KLA-Tencor employees have built over nearly 40 years — their culture, technology, and operating practices. I have no doubt that our combined values, focus on the customer, and complementary technologies will create a trusted leader in our industry, capable of creating significant opportunity for profitable growth and in turn delivering tremendous value to all of our stakeholders. This is the right time for the right combination in our industry."

■ Continued Annual Growth for Silicon Shipment Volumes, Wafer Shipments Forecast to Increase in 2015, 2016, and 2017

SEMI Global Update

SEMI recently completed its annual silicon shipment forecast for the semiconductor industry. This forecast provides an outlook for the demand in silicon units for the period 2015–2017. The results show polished and epitaxial silicon shipments totaling 10,042 million square inches in 2015; 10,179 million square inches in 2016; and 10,459 million square inches in 2017. Total wafer shipments this year are expected to exceed the market high set in 2014 and are forecast to continue shipping at record levels in 2016 and 2017.

"2015 has been a record-breaking year for silicon shipments, attributed primarily to larger diameter wafers," said Denny McGuirk, president and CEO of SEMI. "The outlook for the next two years is measured, but continues on a modest growth path."

All data cited is inclusive of polished silicon wafers, including virgin test and epitaxial silicon wafers shipped by the wafer manufacturers to the end-users and do not include non-polished or reclaimed wafers

■ ASML to Ship Only Three More EUV Systems in 2015

optics.org, 14 Oct 2015

ASML says that it will ship only four laser-driven extreme ultraviolet (EUV) lithography systems this year, although it is still confident about the ultimate success of the technology. The reason is that the customers are prolonging the current nodes and the nodes that they are about to ramp. ASML's lead customers already have two or three EUV systems in-house that they can use to fine-tune future processes. The technology was on track for a volume production insertion at the 7 nm node or equivalent. That insertion appears key to the semiconductor industry regaining its alignment to historical scaling advances. While EUV tool productivity had progressed as expected – one customer has exposed 15,000 silicon wafers in four weeks with the new equipment, and met key imaging and overlay requirements – further improvements in EUV source "availability" were needed.

As ASML continues to hone EUV technology, rival Gigaphoton claimed that its laser-driven sources had demonstrated an average output power of 60 W over a 24-hour period. Though similar to ASML's approach, Japan-headquartered Gigaphoton's tin droplet generator produces smaller droplets. When hit by a "pre-pulse" solid-state laser and then the main carbon dioxide drive laser, those droplets produce a broadband emission including EUV light. Gigaphoton says that it will start operation of a high-output pilot unit for EUV by the end of this year, and is still targeting an output power of 250 W for production insertion – necessary for making chips economically.

In comparison, ASML's latest NXE:3350B tools are fitted with colossal new drive lasers from Germany's Trumpf that will produce 125 W of EUV source power. However, the fact that a second potential vendor of EUV sources is making good progress should give some encouragement to chip makers concerned about ASML being the sole provider of the technology. The customers want to be [back at] Moore's law, and EUV is most likely the enabler to get them on a two-year cadence again.



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