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WHITE PAPER

Technology that extends the life of quartz thermocouple sheaths in ASM® EPSILON® reactors up to three times longer

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In many epitaxial processes, deterioration and failure of the quartz sheath protecting thermocouples (TCs) is the determining factor when designing maintenance schedules and can be the cause of non-scheduled maintenance activities. Such unplanned events of premature TC failure costs the Fab valuable production time and affects the maintenance budget. In the interest of providing the semiconductor industry with a solution that could drastically increase the usage/lifetime of these TCs, and significantly reduce premature failures, Conax Technologies began an 8-year venture to develop an advanced TC sheath coating technology.

Conax Technologies partnered with a University, renowned for its specialty in Glass and Ceramic Engineering. Together, Conax Technologies and the University systematically evaluated a series of solutions to extend the life of quartz TC sheaths within the challenging process ambient created by the specifics of epitaxial processes. After years of work, a coating engineered specifically for the semiconductor quality quartz sheaths, compatible with the epitaxial process, and exceeding expectations for service life, was achieved. As a result, Conax Technologies filed with the US Patent Office, and EtchDefender™ is a Patent-Pending design.

Due to the complexity of the epitaxial process relative to temperatures, gas compositions, gas flow rates, and thermal cycling conditions, simplistic simulation of this process was deemed inadequate. Therefore, the final qualification phase involved use of functional EtchDefender™ TCs in a real production environment. Qualification was performed over an excessive period at a major European Fab producing wafers for the semiconductor industry using multiple ASM® EPSILON® reactors. The Fab used both standard and EtchDefender™ center TCs where the only difference between the two was the EtchDefender™ coating. All center TCs were run under identical processing conditions. The wafers produced were

Conax Premium Quartz Coated with EtchDefender™ **After Use** | Conax Premium **Before Use** | Conax Premium Quartz Uncoated **After Use**

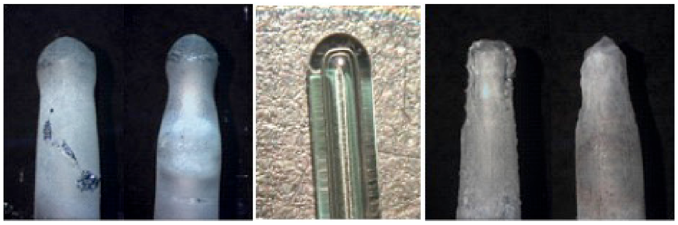

Figure 1. (left) Image of 2 EtchDefender™ TCs run at a Fab in an ASM® EPSILON® Reactor. (center) Example of the Standard quartz sheath before population with the thermoelements and prior to running in the reactor. (right) Image of 2 Standard TCs run at the same Fab to failure under identical process parameters.

closely monitored using surface photovoltage (SPV) and secondary ion mass spectrometry (SIMS) technology to inspect and detect any potential contaminants of the processed wafers due to the EtchDefender™ coating. The Fab did not experience any changes in wafer quality while running with the EtchDefender™ TCs.

EtchDefender™ TCs were removed during planned maintenance after processing 11,200 wafers and were still functioning at the time of removal. Standard TCs were run to the same wafer count, but all showed open circuit or failure of the sheath. Images of both the EtchDefender™ and Standard TCs, after service in the EPSILON® Reactor, can be seen above in Figure 1.

Standard Sheath Deterioration

Thermocouples used within the ASM® EPSILON® Reactor are protected with quartz sheaths. Quartz sheaths function to protect the thermoelements during wafer processing, however, frequent and continued thermal cycling at typical operating temperatures causes devitrification and structural damage. The density change (up to 20%) associated with devitrification (quartz to cristobalite phase transition) causes stresses to build in the sheath and microcracks to form. The level of Cl in the deposition gases and presence of HCl during the in-situ chamber clean and wafer etching results in chemical attack of the microcracks within the sheath. This accelerates the ablation rate of the sheath by the high velocity process gases and presents as severe pitting of the sheath. The pitting leads to holes in the sheath that transverse the quartz providing a pathway between the chamber environment and the interior of the quartz sheath.

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EtchDefender™ TECHNOLOGY

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Standard Thermoelement Deterioation

Once the sheath degradation occurs as described above, H2 molecules will diffuse through the protective quartz sheath. As the quartz (SiO_2) breaks down, H_2 combining with O to form H_2O, the Si is then free to attack the platinum thermoelement forming platinum silicide (PtSi). The formation of PtSi will: 1) cause calibration drift in the thermoelements; 2) cause embrittlement of the thermo-elements at the grain boundaries; and 3) cause a lower melting point in the negative leg of the TC, allowing for wire slip and eventual open circuit condition.

EtchDefender™ Sheath Deterioration

The EtchDefender™ coating creates a surface that is resistant to ablation during epitaxial processing. While the characteristic phase transition will take place within the quartz under typical operating temperatures and thermal cycling, EtchDefender™ impedes the destructive chemical attack that accelerates ablation of the quartz and eventual TC failure. The reduced ablation rate achieved with the EtchDefender™ coating is clear in Figure 1. Instead of pitting, a smooth surface results from the ablation process and quartz loss is a fraction of that seen from standard quartz sheaths in an ASM® EPSILON® reactor. As a result, EtchDefender™ center TCs were run 3X longer than standard ground and diffused TCs and removed prior to failure during a planned maintenance event.

EtchDefender™ Qualification: Thermal Shock Analysis

Quartz used for TC sheaths has a relatively low coefficient of thermal expansion (CTE). When the EtchDefender™ technology is used, an interface between the quartz surface and the EtchDefender™ coating is created. For best performance, EtchDefender™ should be continuous and have a closely matched CTE to that of the quartz. To evaluate continuity and any difference in CTE, both quartz and EtchDefender™ sheaths underwent thermal shock testing by furnace heating to 550°C for 30 mins; before cooling in air or being quenched in water at room temperature. After quenching, CTE matching was assessed, visually and using scanning electron microscopy (SEM), for characteristics, including hazing, crazing, spalling, delamination, and particle generation relative to the standard sheaths. None of the aforementioned indications of a CTE mismatch occurred in any of the sheaths which indicated EtchDefender™ is continuous with the quartz TC sheaths.

EtchDefender™ Qualification: Thermal Cycling Analysis

To further evaluate the EtchDefender™ technology and interfaces in an environment relevant to the semiconductor industry, EtchDefender™ sheaths were cycled at 1200°C under vacuum. Both standard and EtchDefender™ sheaths were loaded into a room temperature furnace at atmospheric pressure; the furnace was sealed and evacuated to reach a vacuum of 10^{-3} Torr, then heated to 1200°C over a period of 6 hrs. The furnace was held at temperature for 24 hours then turned off and allowed to cool to room temperature. Once cooled, the furnace was brought back to atmospheric pressure, and sheaths were removed. This was repeated five times. Sheaths were analyzed visually and using SEM for changes in coating characteristics, such as hazing, crazing, spalling, delamination, and particle generation relative to the standard sheaths. None of the sheaths exhibited the afore mentioned characteristics giving further indication of the robust nature of the EtchDefender™ technology and interfaces and their viability for use over large temperature ranges in atmospheric or vacuum semiconductor processing environments.

EtchDefender™ Qualification: FactSage Decomposition Simulations

Simulations to predict any potential decomposition of the EtchDefender™ coating was completed using the following criteria:

Chemical species:	processing gases including: H_2, HCl, Cl_2, $SiHCl_3$, SiH_2Cl_2, SiH_4 solids including: Si, SiC (coated, graphite susceptor), SiO_2, refractory oxide
System pressure:	1 ATM
HCl partial pressure:	10-100%
Temperature:	800-1200°C
Refractory oxide content:	0-100%
Results:	The total amount of decomposition did not depend on refractory oxide content therefore, results are shown using 100%. The worst-case decom-position corresponded to 100% HCl at 1 ATM; simulation results using these parameters are listed.
	5% decomposition @ 800°C
	0.3% decomposition @ 1200°C
Possible ablated species:	Si, SiC (coated, graphite susceptor), SiO_2, refractory oxide, SiO, refractory silicates, refractory-processing gas compounds

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Relative to Wafer Contamination:

As discussed above, the process of ablation is greatly reduced on TCs with the EtchDefender™ coating. Therefore, in comparison to standard TCs, ablation products from EtchDefender™ TCs that are potential contaminants will be reduced. These ablation products will likely be analogous to those produced with standard sheaths or readily converted into harmless oxides. While simulations were not run to assess the potential deposition of the ablated species on a wafer, data from the Fab formerly and currently using EtchDefender™ TCs suggests zero deposition of ablation products on the wafer. In addition, the Fab has not detected any effect on process gases within their ASM® EPSILON® reactor using the EtchDefender™ TCs. With the introduction of new material into a complex system, especially at high temperatures, thermodynamic anomalies are possible and, as the simulations suggest, there may be minimal decomposition. However it is apparent that, if occurring, these species are effectively removed and neutralized through the exhaust management system.

About Conax Technologies

Conax Technologies is a designer and manufacturer of standard and custom engineered temperature sensors, compression seal fittings and feedthroughs, probes, sensors, wires, electrodes and fiber optic cables. The company is headquartered in Buffalo, New York, with locations on the U.S. west coast, Canada, Europe and Asia.