APPLICATION NOTE

CONTROLLING VAPOR CONDENSATION AND DOWNSTREAM DEPOSITS IN DEPOSITION AND ETCH TOOLS

INTRODUCTION

The thin film deposition and etch technologies used in semiconductor devices, LED, MEMS, barrier films and many other advanced manufacturing processes require technologies for accurate and precise temperature control of the "wetted" surfaces, from chemical precursor delivery to exhaust gas handling systems. In both deposition and etch technologies, condensable vapors and reactive chemistries can produce condensates and/or solid deposits on the internal surfaces of precursor feed lines, process chambers, instruments and lines attached to the chamber, exhaust management system, valves, and other "wetted" areas of the system. As well, other sources can coat these areas through material transfer and deposition by one of the adhesion methods. When precursors are not kept in a liquid or gas state, solids or condensation change precursor delivery rates and/or gas conductance that shift the process and process control parameters. While process control algorithms can compensate to some degree for these changes, the drift in control characteristics can often induce undetected changes in film parameters that could be out of specification from run-to-run or system-to-system impacting product yield. Additionally, limiting or avoiding material deposits within the exhaust lines can significantly reduce maintenance downtime requirements.

BACKGROUND

Deposition processes such as chemical vapor deposition (CVD) and atomic layer deposition (ALD) often use gas or vaporized sources which are delivered to a process chamber. Through chemical reactions or physical deposition a film is deposited on a substrate which has properties suitable for the particular step in the device process. The use of solid sources has historically been limited since it proved difficult to maintain constant composition and precise flow control of the vapor from solid precursors. However, newer deposition technologies such as ALD are beginning to employ more solid source precursors, some of which must be heated to temperatures as high as 300°C to produce appropriate vapor pressures [1]. Once heated to gas form, the precursors need to remain at or above the boiling point for proper metered delivery when gas flow control is used. Common precursors such as $Si(OC_2H_5)_4$ (TEOS) and newer ALD precursors like tanatalum(V) ethoxide (liquid) ($Ta(OC_2H_5)_5$) and trimethylaluminum (liquid) ($Al_2(CH_3)_6$) need 150°C to remain in gas phase when at atmosphere. Other precursors such as tetrakis(dimethylamido)hafnium (solid) (TDMAH, $Hf[N(CH_3)_2]_4$), tetrakis(diethylamido)titanium(IV) (liquid) (TDEAT, $Ti[N(C_2H_5)_2]_4$), trimethylgallium (liquid) ($Ga(CH_3)_3$) require lower temperatures for metered vapor delivery.

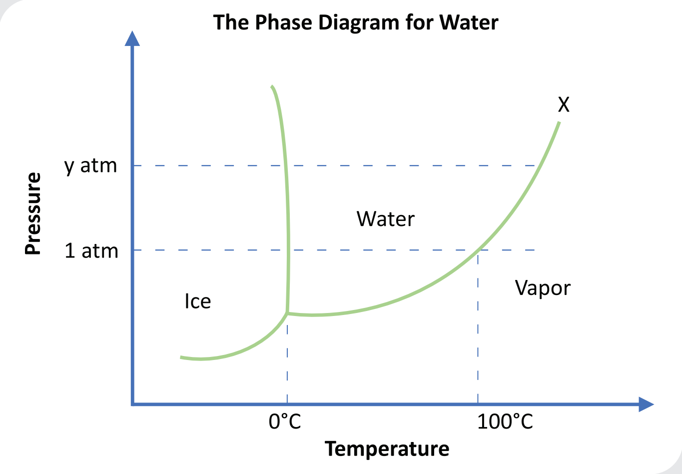


Figure 1 - Triple point phase diagram for water showing three states: solid, liquid and vapor.

High pressure gases such as silane (SiH_4) and oxygen (O_2) are easily delivered to process chambers, whereas liquid and solid precursors such as tetraethoxysilane (liquid) (TEOS,$Si(OC_2H_5)_4$), tungsten hexafluoride (liquid) (WF_6), $Ti[N(C_2H_5)_2]_4$), and TDMAH require the source and delivery lines be heated in order to provide proper vapor pressure so the precursor remains in the vapor phase in the delivery lines. Historically, some liquid precursors have been delivered using diffusion fed bubbles in a gas bubbler system, however, precursor delivery rates can vary with liquid level in such arrangements, and this has resulted in a gradual discontinuation of the approach. Whether a material is a gas, liquid or solid at a given combination of temperature and pressure is governed by the material's triple point curve (Figure 1). This curve can be used as a guide for controlling the pressure, volume (flow) or temperature conditions required to ensure adequate precursor vapor pressure in source vessels and to maintain the precursor as a vapor in reagent delivery lines when liquid or solid precursors are employed.

These curves can be used to understand the best conditions needed to avoid the formation of solid deposits in the vacuum conductance and exhaust lines. Changing precursor flow (and consequently delivery pressure) can impact deposition rates and film properties. Precursors are normally maintained in the vapor phase by controlling the source vessel and delivery line temperatures rather than by varying flow or pressure. Thermal monitoring and temperature control of source vessels and delivery lines must therefore be both highly accurate and precise. Temperatures in the source vessel must be high enough to deliver sufficient vapor to the process chamber while simultaneously being the coolest part of the vapor delivery system to ensure that should precursor condensation occur it will happen preferentially in the source vessel. Simultaneously, the vapor delivery lines must maintain a tightly-controlled and constant temperature over their entire length to avoid internal condensation with concomitant changes in flow or pressure that can impact deposition rates and film properties.

The chemistry of deposition processes ranges from simple pyrolysis (e.g., decomposition of silane to produce polysilicon) to complex reactions involving up to four precursors (e.g., the reaction of TEOS with: trimethylphosphite ($P(OCH_3)_3$), trimethylborate ($B(OCH_3)_3$), and oxygen (O_2) producing a borophosphorous silica glass (BPSG films). Typically, CVD processes convert significantly less than 100% of the process precursor in the processes chamber, and therefore significant amounts of unreacted precursor are sent through the lines to the pump and other exhaust management systems. As well, in many CVD processes (but not in ALD processes), once at process temperature, precursor compounds undergo gas phase reactions that produce highly reactive gas-phase intermediate compounds. The best understood example of this occurs in the pyrolysis of TEOS to produce silicon dioxide. When TEOS is introduced into the process chamber it undergoes the following gas-phase decomposition reaction [2]:

$$Si(OC_2H_5)_4 \longrightarrow Si(OC_2H_5)_3OH + C_2H_4$$

Since this reaction is endothermic by only 10 kcal/mol, almost all of the TEOS going into the deposition chamber (typically at ~720°C in low pressure CVD TEOS) is rapidly converted in the gas phase to $Si(OC_2H_5)_3OH$. The OH group in this molecule makes the intermediate much more reactive than pure TEOS. These intermediates, along with any unreacted precursor, are pumped out of the deposition chamber and into the exhaust lines of the system where they continue to react even up to and through the vacuum pumps. These reactions produce gas phase particulates and non-stoichiometric solid oxide deposition on the walls and other surfaces. These deposits can build up and create flow constrictions that eventually impact the overall conductance of the system and, ultimately, the yield of the process. These deposits are also a particle source within the system. The stainless steel or aluminum surfaces within the process system and other system components have thermal expansion coefficients that are very different from those of any film that coats the surface, and result in high levels of stress between the surface and the coating.

This stress causes the coating to flake off as the surface expands and contracts from temperature and pressure changes. Particles are generated when the loosely adhered coating is stressed enough that it loses adhesion with the surface. Additionally, particulates produced by gas phase nucleation can also lodge in the uneven surfaces of the stressed film. These loosely adhering particles can be transported back into the process chamber during gas cycling, causing unwanted and unpredictable particle excursions in the process. Heating the exhaust lines and other downstream components resolves this issue since it keeps the precursors or byproducts in the vapor phase until they reach the waste treatment unit, and the constant temperature of the components reduces the flaking of any deposited material due to film stress and thermal cycling.

Etch processes have similar issues with reagent delivery and the accumulation of deleterious build-up in the exhaust lines thereby affecting conductance. Source gases for etching are usually fluorine- or chlorine-containing molecules such as carbon tetrachloride (CF_4), nitrogen trifluoride (NF_3), sulfur hexafluoride (SF_6), carbon tetrachloride (CCl_4), chlorine (Cl_2), boron trichloride (BCl_3) or dichlorodifluoromethane (CCl_2F_2). Some of these sources (i.e., CCl_4, BCl_3) must be heated to deliver sufficient vapor to the process, and therefore require tightly-controlled and uniform source vessel and delivery line heating for the same reasons cited earlier for CVD and ALD liquid precursors. While etch source gases do not normally produce solid deposits, the products of etch reactions can form deposits on downstream surfaces. For example, the plasma etching of silicon using CF_4 proceeds according to:

$$CF_4 + e^- \longrightarrow \bullet CF_3 + F\bullet$$
$$Si + 4F\bullet \longrightarrow SiF_4$$

Here the dot by the chemical formulation denotes a radical fragment. In any etch process that uses CF_4 or other fluoro- or chlorofluoro-carbons, $\bullet CF_3$ and other radical fragments can sequentially react to produce polymers as illustrated below for a CF_4 based etch.

$$CF_4 + e^- \longrightarrow \bullet CF_3 + F\bullet \xrightarrow{+\bullet CF_3} C_2F_6 + F\bullet \xrightarrow{+\bullet CF_3} C_3F_8 + F\bullet \xrightarrow{+\bullet CF_3} C_4F_{10} + F\bullet \xrightarrow{+\bullet CF_3} \text{etc.} \xrightarrow{+x\bullet CF_3} C_nF_{2n+2}$$

Teflon-like polymer build-up, due to this or a similar reaction sequence, is observed in MEMS manufacturing using the Bosch etch process. Chlorine-based metal etching processes such as aluminum etch can also produce downstream deposits, in this specific case crystalline aluminum trichloride, $AlCl_3$ [3]. The vapor pressure curve in Figure 2 shows that $AlCl_3$ is a solid at room temperature (25°C) at ambient pressures as low as sub-millitorr. Thus, in an aluminum etch process, with unheated vacuum lines, most of the downstream surface will be coated with aluminum trichloride when the process pressure is in the millitorr range or unless the byproduct is trapped close to the source. Many other etch processes that use fluorocarbon and inorganic halide gases produce either polymeric fluorocarbon or solid halide salt deposits on downstream surfaces.

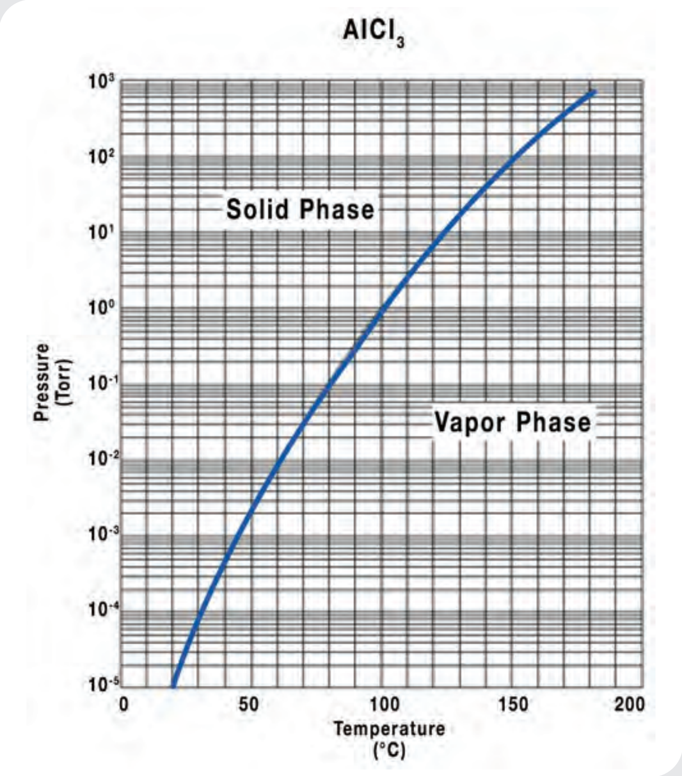


Figure 2 - $AlCl_3$ vapor pressure curve.

(a) (b)

Figure 3 - LPCVD silicon nitride system showing (a) heating tape tracing vs. (b) conformal molded polymer electric heaters.

The use of liquid sources and the presence of unwanted solid deposition in the downstream components of thin film deposition and etch tools, requires a temperature control system that can maintain precise uniform temperatures or temperature profiles in different system components having strongly varying topographies. Hardware such as gauges, piping, slit valves and gauge tree manifolds often have complex bends and tight corners that must all be heated to a uniform temperature. Historically, this need was first met using conventional electrical heat tape tracing, maybe with some insulation. However, while it is low cost and relatively simple to install, there are serious problems in using this heating method in deposition and etch equipment. Heating tape cannot cross over itself or over another heating tape, since this produces localized overheating and destruction of the tape. For this reason, heat tracing complex topographies invariably presents a tradeoff between tape positioning, the avoidance of cross-over and the elimination of cold/cool spots on the heated surface. This tradeoff produces subjective choices that impact the temperature distribution over the system component being heated that strongly depends on repeating the same wrapping pattern and tape uniformity. No two installations yield exactly the same thermal result and this can impact tool-to-tool repeatability. Non-uniform wrapping produces cold or cool spots in the gas delivery components or other heating applications especially when the heat tracing with periodic insulation is employed (due to difficulties in avoiding heat sinks at points of attachment, etc.) and producing condensation problems that impact vapor flow control. Secondly the placement of the thermocouple, to monitor temperature, needs to be strategically placed to reflect the proper temperature reference. Often, heat tape only has one location for the thermocouple which is not always ideal. Therefore, the component being heated could experience coating issues because of the temperature uniformity.

More recently, conformal, molded polymer electric heater jackets have been developed that are far superior to heat tracing. These are flexible jackets, quick and simple to install, that apply more uniform and consistent heat than can be achieved using electrical heat tape tracing. Figure 3 provides a comparison of the use of heat tracing tape with conformal polymer heaters for the back end of a LPCVD silicon nitride system. Since the development of these polymer heater jackets, temperature control systems for delivery and conductance line service have been developed that provide significant improvements in deposition and etch system yield along with reduced maintenance costs.

FEEDLINE, PROCESS CHAMBER AND VACUUM FORELINE MAINTENANCE

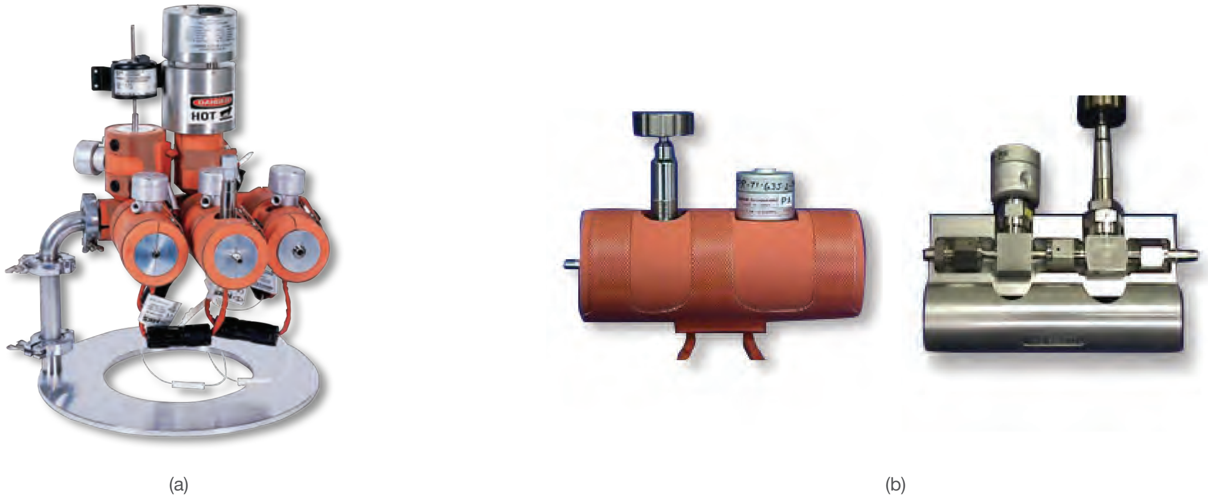
Precursor Source Vessel and Feedline Heating

Precursor source vessels are easy to heat but requirements for ALD systems are trending toward less than 2°C temperature control. Precursor feedlines and the associated manifolds and valve and sensor assemblies present more difficult topographies where conventional heat taping approaches encounter significant difficulties in maintaining uniform line temperature. MKS Instruments has developed designs for source delivery and gauge tree heating using conformal heaters and component enclosures that effectively solve this problem. Figure 4(a) shows a heated gauge tree assembly incorporating a manifold, multiple valves, a Baratron® pressure sensor and silicone heaters. Figure 4(b) shows how the heating of complex shapes is accomplished by machining an aluminum block so that there is tight contact between the heater and the aluminum block which creates an oven. MKS also offers heaters that can fit in tight spaces without the use of such blocks.

In-Situ and Off-Line Foreline Cleaning

Undesirable coatings in process chambers and on downstream surfaces in deposition and etch systems can be addressed using a number of different approaches. Single-wafer deposition and etch tools typically employ some variant on in-situ cleaning to remove deposits from the internal surfaces in process chambers. These can employ chemistries that are either thermally induced, plasma activated or utilize chemically reactive precursors depending on the inherent capabilities of the process tool. This approach to cleaning critical internal system surfaces is broadly employed utilizing existing capabilities of activating the cleaning component through one of the chemical channels of radical generation, heat or reactive chemical reaction. Typically the cleaning process utilizes little or no ancillary equipment additions.

The removal of solid coatings from downstream vacuum exhaust lines is more problematic since these are passive system components with no inherent capability to promote chemistries that can remove coatings. In-situ chemical cleaning analogous to that employed for process chambers has been evaluated for the removal of deposits in exhaust lines. It is not widely used, however, since these components cannot be heated to the temperatures needed for typical in-situ cleans, and this makes it very difficult to completely clean all of the surfaces in this way. In-situ cleans have been evaluated employing chemistries that are more effective at the achievable temperatures in a vacuum environment and compatible with the exhaust components, however,



(a)

(b)

Figure 4 - (a) Heated gauge tree assembly; (b) heater approach for complex shapes

these have introduced condensable vapors into the vacuum lines that result in prolonged system recovery times. The use of switchable binary exhaust systems can allow disassembly of the non-operational sections for off-line cleaning without system interruption. Such solutions tend to be cost prohibitive, however, since the two systems must be the same so there is no process shift from conductance changes.

SOLUTION

MKS Instruments provides a proven, unique solution for avoiding deposition on vacuum conductance lines — the Virtual Wall™ Gas Barrier Device. The Virtual Wall is inserted into the exhaust near the outlet of the chamber, in the inlet of abatement systems and other unique applications. The unique gas introduction pattern produces a gas barrier (boundary layer) reducing the ability of the effluent gas from contacting the walls of the line. This system has demonstrated its effectiveness in reducing or eliminating particle contamination due to particle generating reactions and particles generated from flaking of coatings in the pumping forelines, effluent abatement systems, and pump exhaust lines. The continuous unidirectional gas flow generated by the Virtual Wall keeps any particles in the pump forelines moving downstream to locations far enough from the chamber that they no longer constitute a threat of particle contamination due to backstreaming to the chamber.
A noted major success for particle reduction and wall coating reduction is on Low Pressure CVD (LPCVD) TEOS systems where the combination of the Virtual Wall, exhaust line configuration and a trap have increased yields significantly. Applications also exist for other processing systems. The length of Virtual Wall that can be used in pumping forelines has limits imposed by the impact of additional gas flow into the exhaust. Additional gas increases the low end pressure capability of the pump and changes the pressure differential between the process pressure and the base pressure. The Virtual Wall, when combined with a trap, provides one of the best solutions on the market for extending the time between cleans. While the Virtual Wall and trapping can resolve line coating on short distances, the combination will always be less than 100% efficient, which means deposition will still coat the parts downstream but at a much slower rate. One of the most widely used methods for further controlling the rate of build-up of these deposits is to heat the lines.

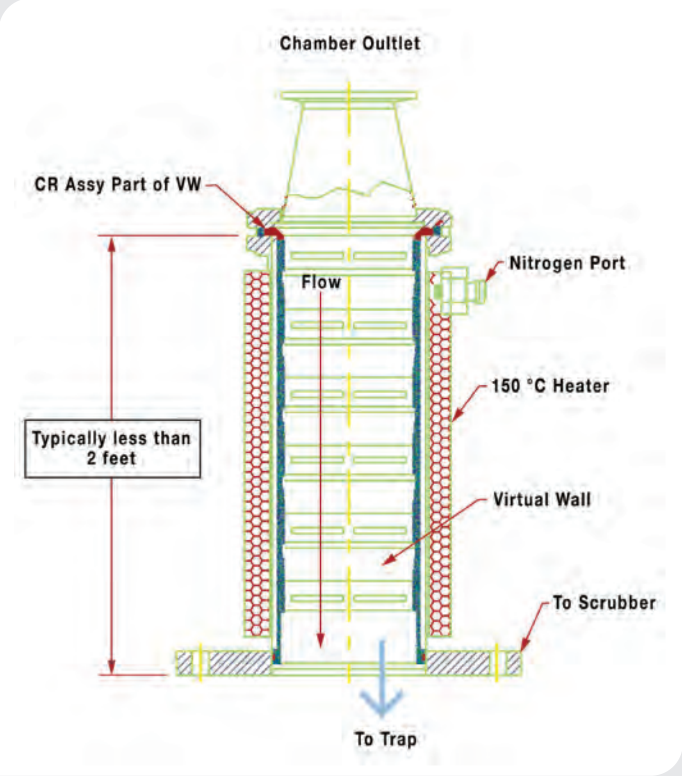


Figure 5 - The MKS Instruments Virtual Wall™ Gas Barrier Device.

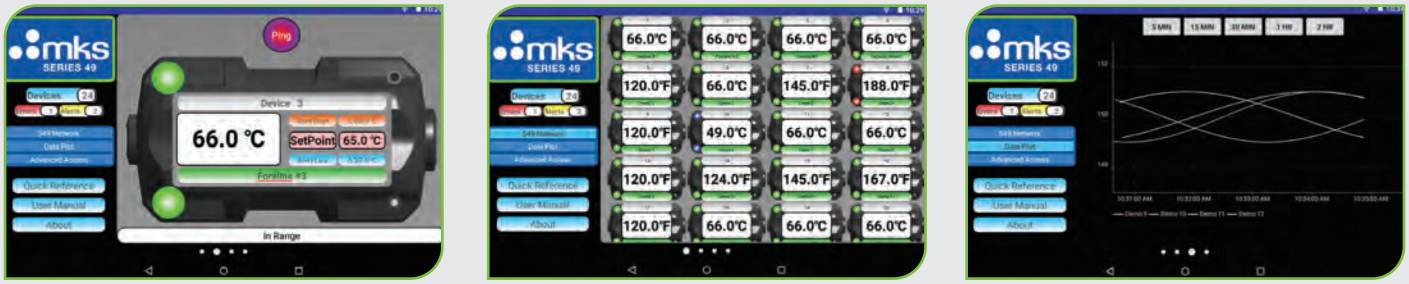
Heating critical parts of a deposition or etch system is an effective way to control the condensation of source materials or downstream deposition of solids due to excess precursor and byproduct reactions. Historically, heaters used in these applications employed individual control systems that incorporated a thermocouple at the ideal location to monitor the temperature. Using a single controller with a single heater that contains a thermocouple is the best configuration. When multiple heaters are employed and there is only a single controller, without the capability of handling additional temperature inputs and individual zone control, a dangerous situation can occur. The reason this is unsafe is if a control system is running two or more heaters in a master-slave control scheme and master thermocouple fails or power is lost to the master heater (with the master thermocouple), the controller sees the temperature as too low and will increase the power to all the heaters in an effort to keep the master thermocouple at set point. Since the master thermocouple fails to show that the temperature of the heated object has reached set point, the power remains on until there is either other component failure stopping the application of power or the heaters get so hot they reach the very real potential of a fire. Improperly employed master-slave heater configurations can encounter problems with uneven heating. If the master thermocouple is positioned on a large mass of metal, the slave heaters will run hotter than set point because of the mass of the material being heated. More power needs to be applied to the heater that is heating the large part but, since there is no need for such a long power application for smaller parts due to lower mass, the small parts get hotter. Conversely, if the master thermocouple is located on the smaller mass of metal, those parts with greater mass will not achieve set point, since the smaller mass achieves set point before sufficient energy is added to the system to raise the greater mass to set point.

MKS Instruments has developed advanced, comprehensive heater systems that prevent damage and hazardous run away conditions in heated line installations. The Series 49UL Thermal Management System (Figure 6) provides a comprehensive thermal management system that combines an intuitive user interface for intelligent and flexible control with two advanced heaters with new jacket materials offering high energy efficiency. Additionally, the MKS Series 49UL controller is an Underwriters Labs (UL) recognized component and carries a NEMA 2 housing rating. The UL recognition means the controller has undergone extensive evaluation and testing to meet the requirements and guidelines recognized around the world.

The Series 49UL Thermal Management System employs the Series 49UL advanced controller which allows fully adjustable set points, a high/low temperature alert, power switching and functions such as datalogging and a single temperature set point broadcast function which allows all heaters on a line to be set to a temperature with one input rather than having to do that multiple times. The controller also has communications capabilities utilizing Modbus RTU RS485 protocol where the process tool can access registers and addresses enabling on-tool control and monitoring of the heater system. The configuration

① Series 49UL User Interface Application
② Series 49UL Heater Control Module
③ Series 49UL PTFE Heater Jacket
④ Series 49UL Polyimide Heater Jacket
⑤ Series 49UL LTA+ Control Module

Figure 6 - The MKS Series 49UL Thermal Management System.



(a) (b) (c)

Figure 7 - The MKS Series 49UL User Interface System Display: (a) Single-node Screen; (b) Multi-node Screen; (c) Data Plot Screen.

is such that all controllers in the line are accessed from a single convenient location because of the network accessible configuration. Some of the key additions to the controller function are redundant temperature sensing and diagnostic capabilities, PCB temperature sensing, solid state no-arc relays, and a re-settable safety limit circuit. For those applications where the controllers are at a distance, LED’s are employed to show controller condition and with the 360° viewing, it is simple to find an offending controller or heater visually. The Series 49UL Controller can also be customized for OEM configurations and an optimized master-slave configuration that reduces the chance of an unsafe condition.

The Series 49UL User Interface System is an integrated remote digital communication and diagnostic solution that enables detailed control, diagnostic feedback and data acquisition using a tablet and easily downloadable, Android-based software application (app). The user interface app contains a sequential auto-addressing function which automatically increments the address on consecutive controllers for ease of set up, and prevents duplication of addresses. Network configurations can be saved and loaded for multiple OEM tool installations. The app allows full access to all Series 49UL Controller functions in an intuitive set up for control, monitoring, diagnostics and parameter adjustment. The multi-function user screen (Figure 7) allows the user to monitor the control function, temperature and alarm states of either single or multiple heaters in a network. The user interface also allows the user to access variable time-scale plots of heater temperatures, tuning and diagnostics.

In a fab that employs multiple deposition and etch tools, the energy used for heating can be in the thousands of kilowatt hours, resulting in a significant cost factor. MKS Instruments’ proprietary Series 49UL PTFE (Teflon®) woven-shell and Series 49UL polyimide heater jackets, shown in Figure 6, help to reduce energy costs. These materials provide better insulation properties than silicone foam and heat tape heaters. The heater jackets are much thinner and lighter and with superior properties retain more heat than the materials used in older heater jacket designs. This results in a 30% reduction in power consumption compared to older designs (Figure 8). The advanced materials in these heaters allows their use in cleanroom applications that demand ultra-low particulate generation, low outgassing (low VOC emissions) and high temperatures (up to 250°C).

Control of heater jackets is another area that can be optimized for energy savings in the fab. The MKS Series 49UL Controller has two energy saving functions that further reduce energy consumption. Idle mode allows the user to turn off all the heaters in a network with a single command generating energy savings while the tool is undergoing maintenance or in extended idle. A single command restarts the system while retaining all of the set points. As well, the broadcast function can be used to send a lower set point to all controllers in the network using a single command. This can reduce energy use while minimizing heat up and restart time. Figure 8 compares the energy usage for the current methods of heater jacket use with the energy used in similar systems employing MKS’ advanced materials heater jackets (32.6% energy saving) and with similar systems employing MKS' advanced materials and MKS Series 49UL advanced power management protocols (35.7% energy saving).

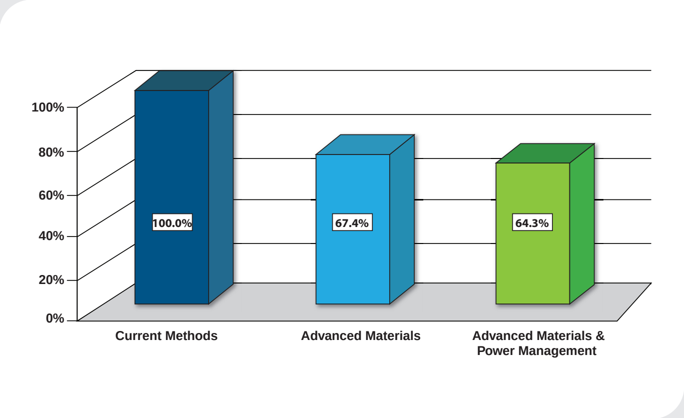


Figure 8 - Representative heater power usage for 180°C set point (3" diameter, 35 feet long, 240 VAC).

It features control functions that enhance the system safety while simultaneously reducing the cost of heater installations. By monitoring multiple temperature points on a single heater the Series 49UL system accurately controls at low temperature set points with superior temperature uniformity across the length of the heater. The system's broadcast function allows a single broadcast of a set point to be directed to each heater controller in a system, making set point changes easier. Operation and maintenance of heaters in the Series 49UL system is facilitated through the use of data logging and error checking functions. Combining the Series 49UL Thermal Management System with other MKS products such as the Virtual Wall, Automation Platform and traps along with MKS expert design engineer support will provide longer system uptime with concomitant increases in production and revenue.

CONCLUSION

MKS Instruments offers a series of advanced heaters and heater control systems well-suited for use in the different applications in ALD, CVD and etch in semiconductor device fabrication as well as many other deposition applications for a multitude of films. The new heaters are thinner and more conformal, use less energy than conventional heat tracing/insulation installations, and have superior control of temperature uniformity. The Series 49UL Thermal Management System simplifies maintaining accurate and precise temperature control while simultaneously providing improved safety functions and more comprehensive communications options. It incorporates digital communications that enable detailed control of single heaters and networks of heaters using a tablet and easily downloadable, Android-based software.

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