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Instructions: Diener Hexamethyldisiloxane (HMDSO) Plasma-Enhanced Chemical Vapor Deposition (PECVD)

by Thomas C Parker

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14. ABSTRACT
This report describes the procedure to set up a modified Diener plasma-enhanced chemical vapor deposition system using a liquid hexamethyldisiloxane (HMDSO) precursor. The user, via gas control, determines the chemistry of the deposited thin films. The composition is controllable from approximately 50 atomic percent carbon to less than 1 atomic percent carbon continuously. The control is realized by balancing the ratios of the partial pressures of the input gasses, argon and/or oxygen, with the HMDSO partial pressure. This permits the deposition of polymer thin films to silicon dioxide films with a single nontoxic precursor.

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1. Introduction

This report describes the procedure to set up a modified Diener plasma-enhanced chemical vapor deposition system using a liquid hexamethyldisiloxane (HMDSO) precursor. The user, via gas control, determines the chemistry of the deposited thin films. The composition is controllable from approximately 50 atomic percent carbon to less than 1 atomic percent carbon continuously. The control is realized by balancing the ratios of the partial pressures of the input gasses, argon (Ar) and/or oxygen (O), with the HMDSO partial pressure. This permits the deposition of polymer thin films to silicon dioxide films with a single nontoxic precursor. The ability to deposit thin films with varied chemistry reduces the overall complexity of deposition tools and hence reduces costs. The ability to use a single nontoxic and non-chlorofluorocarbon precursor to deposit organic and inorganic films lowers both environmental and work safety barriers to industrial application to Army material systems.

2. Procedures

The chamber preparation, maintenance, and process to deposit thin films with controlled chemistry in a Diener plasma system follow:

1) Wipe the interior of the vacuum chamber with particle-free wipes and an alcohol such as methanol, isopropanol, or ethanol.

   It is important to wipe down the interior surfaces of the vacuum chamber to remove particulates and poorly adhered material.

2) Place a cleaned stainless screen in front of the pumping port at the back of the chamber (Fig. 1).

Fig. 1 Deposition chamber with the screen in place

The screen is meant to prevent plasma formation on the ungrounded pump inlet screen. The screen must be protected by a faraday shield to prevent the subsequent deposition on the pump port screen. Try to keep the temporary screen flush against the back of the chamber, using the aluminum sample plate to push the screen in place.

3) The chamber is now ready to introduce samples. To minimize the particles on the sample surface, it is recommended to mount the samples horizontally, if possible.

4) The chamber can now be pumped down to find its base (lowest) pressure. This requires running the system in "manual mode". Pump the system until it reaches its base pressure. This is typically around 0.02–0.03 mbar.

5) Ensure all gas valves are in the off/closed position, as shown in Figs. 2 and 3.

Fig. 2 **Gas line pumping valves**

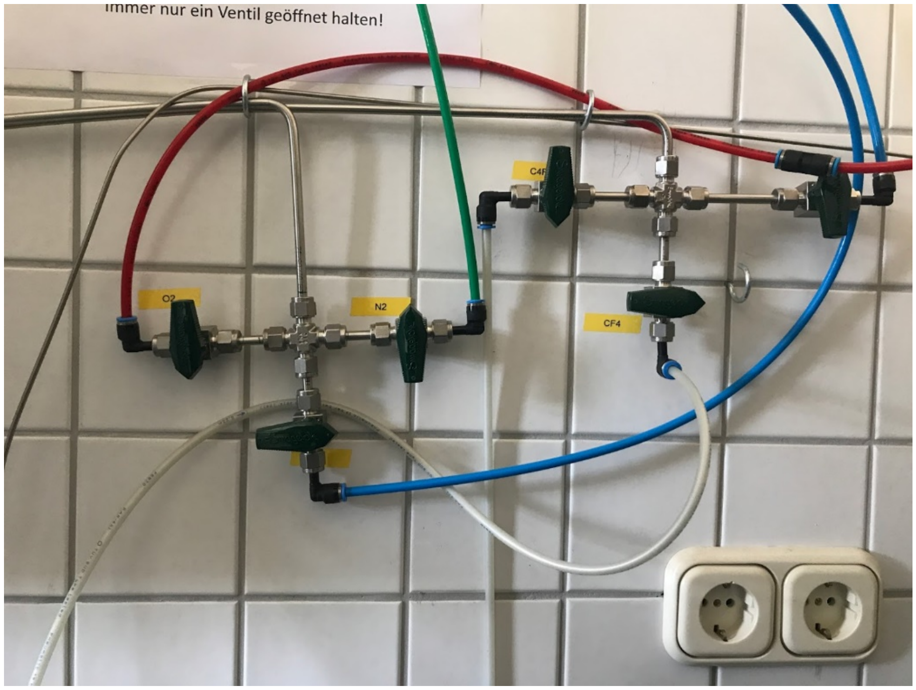

Fig. 3 Gas supply valves

Turn on the system to power the vacuum pump. The gas line pumping valves shown in Fig. 2 must each be opened and pumped to remove any residual gas in the supply lines. Pump each line (separately) for 1 min, ensuring to return the valve to the closed position.

Now that the supply lines are under vacuum, the gas supply valves shown in Fig. 3 should be opened for the desired film chemistry (based on the parameters in Table 1).

Table 1 HMDSO/gas parameters to control film chemistry

HMDSO (Δ mbar)	Gas (Δ mbar)	O:C ratio	SCCM	Film composition	Film character
0.0	0.33	0.35:1	200 (Ar)	$SiO_{0.6}C_{1.7}$	Polymer
0.13	0.20	2.04:1	65 (O_2)	$SiO_{1.32}C_{0.9}$	More polymer like
0.13	0.33	3.04:1	120 (O_2)	$SiO_{1.57}C_{0.29}$	More silica like
0.06	0.33	6:1	130 (O_2)	$SiO_{1.75}C_{0.008}$	Silica

Note: C = carbon, SiO_x = silica.

6) The partial pressures of each gas/vapor must now be done to achieve the desired film chemistry (see Table 1).

a) The HMDSO pressure and gas pressures must be measured/set before each deposition to ensure the correct film chemistry. While in the manual mode, open the HMDSO valve on the touch panel. Open the manual valve just above the glass flask that contains the HMDSO. Now, adjust the needle valve to achieve a change (Δ mbar = pressure – base pressure) of 0.06 to 0.13, depending on the desired film chemistry. Next close the HMDSO valve on the touch panel, leaving the needle valve and manual valve in place. Again, pump down the system to its base pressure. Once the base system pressure is reached (0.02–0.03 mbar), the user can now set the gas flow to achieve the correct pressure for the desired film chemistry (see Table 1). If different film chemistries are desired, the necessary parameters can be calculated with a "path to excel-file".

7) With the gas parameters now set, the system is ready for the deposition of the desired film chemistry as determined by the input gasses. The overall system pressure during the deposition should be 0.40–0.60 mbar. If the pressure keeps increasing above 0.65 mbar, it indicates that the screen has become coated and the pumping port is becoming too obstructed. The deposition should be stopped if the pressure goes above 0.65 mbar as the plasma will become unstable. A deposition power of 30% was found to lead to a stable plasma for all conditions listed in Table 1. Deposition rates were on the order of 10 nm/min. In Fig. 4, a photo of a deposition in progress demonstrates the appearance of a stable plasma.

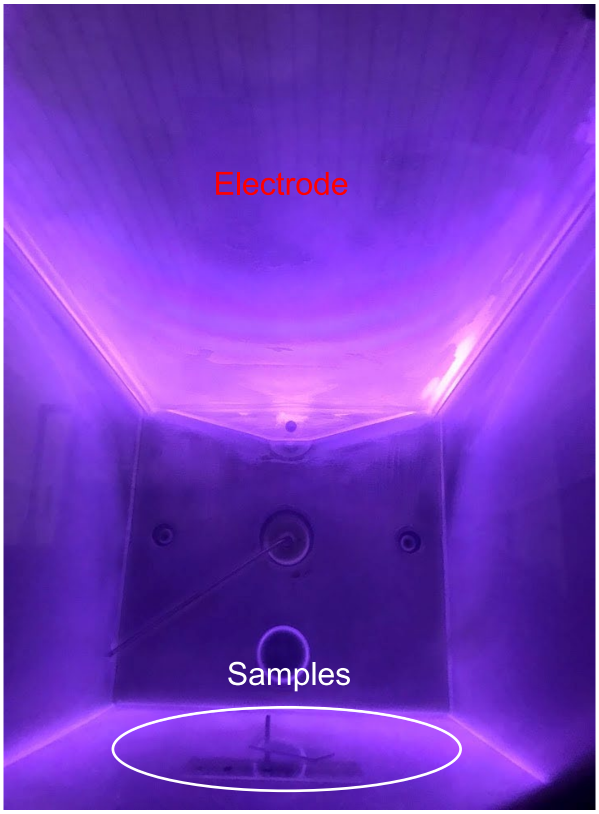

Fig. 4 View of a plasma-enhanced deposition in progress

8) Once the deposition is completed, remove your samples. Clean the chamber (per Step 1). Remove the screen and clean it as well for later reuse.
9) If desired, one can perform additional plasma cleaning of the chamber by running a program with oxygen plasma.
10) After finishing all processes, please close all valves in the gas/pumping line.

3. Conclusion

The process and procedure to deposit thin films in a Diener plasma system have been described. A method of controlling the thin-film chemistry through the O-to-C ratio of the input gasses was described. The method to control the O-to-C ratio for four film chemistries, ranging from organic to inorganic, was listed in Table 1.

List of Symbols, Abbreviations, and Acronyms

Ar	argon
C	carbon
HMDSO	hexamethyldisiloxane
O	oxygen
PECVD	plasma-enhanced chemical vapor deposition
SiOx	silica

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