(54) Title: METHODS AND APPARATUS FOR FORMING GASEOUS ORGANOSILICON COMPOUNDS

(57) Abstract: The present application is directed to a method for producing, from a solid organosilane compound, a mixture of gaseous organosilicon compounds having a desired molar ratio, the method comprising the step of pyrolysing the solid organosilane while maintaining a predetermined pressure which is selected to provide the desired molar ratio of gaseous organosilicon compounds. Associated apparatus and control mechanisms are also disclosed.
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European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, NO, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).
METHODS AND APPARATUS FOR FORMING GASEOUS ORGANOSILICON COMPOUNDS

FIELD OF THE INVENTION

The present invention relates to methods and apparatus for forming gaseous organosilicon compounds.

BACKGROUND OF THE INVENTION

Ceramic amorphous silicon carbide has been known to be produced from polydimethylsilane (PDMS). A description of such a process can be found, for example, in "Synthesis and characterisation of polysilane precursors for silicon carbide fibers" by Cranstone et al. (J. Material Research Vol. 10, No 10, p. 2659, October 1995). In these previous efforts, focus has been kept on turning the polymer into liquid, then applying further treatment, usually at high temperature and/or pressure to turn the liquid into ceramic silicon carbide. Typically, the silicon carbide is prepared by thermal decomposition of PDMS under high pressure in an autoclave, the decomposition proceeding through a process known as a Kumada rearrangement. The efficiency of this decomposition is known as the ceramic yield, and losses to the ceramic yield occur when some of the starting mass is not turned into silicon carbide but instead into a gas. Therefore, a poor ceramic yield correlates with a higher gas yield from the polymer, a situation that conventionally has been avoided.

It has also been reported by Takamizawa et al. (U.S. Patent No. 4,461,908) that methyl hydrogensilanes such as dimethyl silane, 1,1,2,2-tetramethyl disilane and 1,1,2,2,3,3-hexamethyl trisilane can be prepared from methyl polysilane at a temperature from 350°C to 800°C.
SUMMARY OF THE INVENTION

According to one aspect of the present invention, there is provided a method for producing from a solid polysilane compound a mixture of gaseous organosilicon compounds having a desired molar ratio, the method comprising the step of pyrolysing the solid polysilane while maintaining a predetermined pressure which is selected to provide the desired molar ratio of gaseous organosilicon compounds.

According to a further aspect of the present invention, there is provided a method for producing a mixture of gaseous organosilicon compounds comprising at least 50 mol% of gaseous organosilicon compounds comprising 3 to 5 silicon atoms, the method comprising heating a solid organosilane (e.g. polysilane) compound at a temperature from 350 to 500 °C under a maintained pressure from 100 to 250 psi.

According to another aspect of the present invention, there is provided a method for producing a mixture of gaseous organosilicon compounds comprising at least 50 mol% of gaseous organosilicon compounds comprising 1 or 2 silicon atoms, the method comprising heating a solid organosilane (e.g. polysilane) compound at a temperature from 350 to 500 °C under a maintained pressure from 600 to 900 psi.

According to another aspect of the present invention, there is provided an apparatus comprising: a gas panel for connection to a gas outlet of a vessel, the gas panel comprising a valve for controlling a flow of gas from the gas outlet of the vessel through the gas panel; and a controller for controlling a heater to heat a solid organosilicon compound in the vessel at a predetermined temperature while a predetermined pressure is maintained inside the vessel by the valve, to thereby form a mixture of gaseous organosilicon compounds.
There is also provided a method comprising: determining a temperature inside a vessel containing a solid organosilicon compound; controlling a heater based on the determined temperature to heat the solid organosilicon compound at a predetermined temperature; and maintaining a predetermined pressure inside the vessel while the solid is heated, to thereby form a mixture of gaseous organosilicon compounds.

BRIEF DESCRIPTION OF THE DRAWINGS

In the accompanying drawings, which illustrate an exemplary embodiment of the present invention:

Figure 1 is a graph showing the relationship between temperature and pressure during the evolution of gas from polydimethylsilane (PDMS).

Figure 2 is a graph showing the pressure of gas within a vessel as gas is removed from the vessel and additional gases are produced.

Figure 3 is a block diagram of a system according to an embodiment of the invention.

Figure 4 is a schematic diagram of an example gas panel layout.

Figure 5 is a diagram of an example gas cabinet.

DETAILED DESCRIPTION OF THE INVENTION

It has now been discovered that pyrolysing a solid organosilane, such as a polysilane, can be carried out at a variety of maintained pressures to produce a mixture of gaseous organosilicon compounds, and that the nature and relative molar amount of the gases present in the produced mixture will be affected by the pressure selected.

In the present application:
"solid organosilane" and "solid polysilane" refer to organosilane and polysilane compounds that are solid at room temperature and pressure. In some embodiments, the solid organosilane, e.g. polysilane, is solid at room temperature and pressure.

"pyrolysing" or "pyrolysis" refers to the chemical decomposition of the solid organosilane by heating in an atmosphere that is substantially free of molecular oxygen.

"gaseous organosilicon compounds" refer to compounds comprising silicon, carbon and hydrogen atoms, which compounds are in the gas phase at 200°C and 20 psi;

"maintained pressure" refers to a state where the pressure is kept substantially stable, optionally at a selected pressure or within a selected pressure range;

"relative molar amount of the gases present in the produced mixture" refers to the relative molar amounts of the individual gases within the mixture, or the relative molar amounts of one or more specific gases in relation one or more other gases within the mixture;

Solid organosilane compound

The solid organosilane compound may, in one embodiment, be a silicon-based polymer (Si-Si backbone) with Si-C bonds that are thermodynamically stable during heating in a heating chamber. In one embodiment, the silicon-based polymer has a monomeric unit comprising at least one silicon atom and two or more carbon atoms. The monomeric unit may further comprise additional elements such as N, O, F, or a combination thereof. In another embodiment, the polymeric compound is a polysilane or a polycarbosilane.
The polysilane compound can be any solid polysilane compound that can produce gaseous organosilicon compounds when pyrolysed, i.e. chemical decomposition of the solid polysilane by heating in an atmosphere that is substantially free of molecular oxygen. In one embodiment, the solid polysilane compound comprises a linear or branched polysilicon chain wherein each silicon is substituted by one or more hydrogen atoms, Ci-C₆ alkyl groups, phenyl groups or -NH₃ groups. In a further embodiment, the linear or branched polysilicon chain has at least one monomeric unit comprising at least one silicon atom and one or more carbon atoms. In another embodiment, the linear or branched polysilicon chain has at least one monomeric unit comprising at least one silicon atom and two or more carbon atoms.

Examples of solid organosilane compounds include silicon-based polymers such as polydimethylsilane (PDMS) and polycarbomethylsilane (PCMS), and other non-polymeric species such as triphenylsilane or nonamethyltrisilazane. PCMS is commercially available (Sigma-Aldrich) and it can have, for example, an average molecular weight from about 800 Daltons to about 2,000 Daltons. PDMS is also commercially available (Gelest, Morrisville, P.A. and Strem Chemical, Inc., Newburyport, M.A.) and it can have, for example, an average molecular weight from about 1,100 Daltons to about 1,700 Dalton. PDMS is known as a polymer able to yield polycarbosilane. Use of PDMS as a source compound is advantageous in that (a) it is very safe to handle with regard to storage and transfer, (b) it is air and moisture stable, a desirable characteristic when using large volumes of a compound in an industrial environment, (c) no corrosive components are generated in an effluent stream resulting from PDMS being exposed to CVD process conditions, and (d) PDMS provides its own hydrogen supply by
virtue of its hydrogen substituents and yields dense amorphous SiC at temperatures as low as 500°C.

In another embodiment, the solid organosilane source may have at least one label component, the type, proportion and concentration of which can be used to create a chemical "fingerprint" in the obtained film that can be readily measured by standard laboratory analytical tools, e.g. Secondary Ion Mass Spectrometry (SIMS), Auger Electron Spectrometry (AES), X-ray photoelectron spectroscopy (XPS).

In one embodiment, the solid organosilane source can contain an isotope label, i.e. a non-naturally abundant relative amount of at least one isotope of an atomic species contained in the solid organosilane source, e.g. C\textsuperscript{13} or C\textsuperscript{14}. This is referred to herein as a synthetic ratio of isotopes.

Mixture of gaseous organosilicon compounds

In one embodiment, the mixture of gaseous organosilicon compounds comprises one of more gases selected from a gaseous silane, a gaseous polysilane, or a gaseous polycarbosilane. In another embodiment, substantially all of the gaseous organosilicon compounds produced within the mixture comprise from 1 to 4 silicon atoms. By gaseous silane is meant a compound comprising a single silicon atom, by gaseous polysilane is meant a compound comprising two or more silicon atoms wherein the silicon atoms are covalently linked (e.g. Si-Si), and by gaseous polycarbosilane is meant a compound comprising two or more silicon atoms wherein at least two of the silicon atoms are linked through a non-silicon atom (e.g. Si-CH\textsubscript{2}-Si).

In a further embodiment, the gaseous organosilicon compound can be a gaseous polycarbosilane of formula:

\[
\text{Si(}\text{CH}_3\text{)}_n \text{(H)}_m - [\text{(CH}_2\text{)}_p \text{Si(}\text{CH}_3\text{)}_q \text{(H)}_r \text{]}_x - \text{Si(}\text{CH}_3\text{)}_n \text{(H)}_m'.
\]
wherein \( n, m, n', \) and \( m' \) independently represent an integer from 0 to 3, with the proviso that \( n + m = 3 \) and \( n' + m' = 3 \), \( p \) and \( q \) independently represent an integer from 0 to 2, with the proviso that \( p + q = 2 \) for each silicon atom, and \( x \) is an integer from 0 to 3.

Examples of gaseous silanes and gaseous polycarbosilanes include trimethyl silicon, tetramethyl silicon,
\[
[\text{Si(CH}_3\text{)}(\text{H})_2]-\text{CH}_2-[\text{Si(\text{CH}_3)}_2(\text{H})], \quad [\text{Si(\text{CH}_3)}_2(\text{H})]-\text{CH}_2-
\]
\[
[\text{Si(CHs)}_2(\text{H})], \quad [\text{Si(CH}_3\text{)}_3]-\text{CH}_2-[\text{Si(CH}_3\text{)}_2(\text{H})], \quad [\text{Si(\text{CH}_3)}_2(\text{H})]-\text{CH}_2-
\]
\[
[\text{Si(CH}_3\text{)}_2]-\text{CH}_2-[\text{Si(CH}_3\text{)}_3], \quad [\text{Si(CH}_3\text{)}_2(\text{H})]-\text{CH}_2-[\text{Si(CH}_3\text{)}_2]-\text{CH}_2-[\text{Si(CH}_3\text{)}_2(\text{H})], \quad [\text{Si(\text{CH}_3)}_2(\text{H})]-\text{CH}_2-[\text{Si(\text{CH}_3)}_2]-\text{CH}_2-[\text{Si(\text{CH}_3)}_2(\text{H})], \quad [\text{Si(CH}_3\text{)}_2]-\text{CH}_2-[\text{Si(CH}_3\text{)}_2]-\text{CH}_2-[\text{Si(CH}_3\text{)}_2(\text{H})], \quad [\text{Si(CH}_3\text{)}_2(\text{H})]-\text{CH}_2-[\text{Si(CH}_3\text{)}_2]-\text{CH}_2-[\text{Si(CH}_3\text{)}_2(\text{H})], \quad [\text{Si(CH}_3\text{)}_2]-\text{CH}_2-[\text{Si(CH}_3\text{)}_2]-\text{CH}_2-[\text{Si(CH}_3\text{)}_2(\text{H})], \quad [\text{Si(CH}_3\text{)}_2(\text{H})]-\text{CH}_2-[\text{Si(CH}_3\text{)}_2]-\text{CH}_2-[\text{Si(CH}_3\text{)}_2(\text{H})].
\]

**Method**

The production of a mixture of gaseous organosilicon compounds from a solid organosilane is achieved through a pyrolysis step, which can encompass one or more different types of reactions within the solid. In one embodiment, different types of reactions are promoted depending on the temperature selected for the pyrolysis step.

In the embodiment where the solid organosilane source is a polysilane, one possible reaction leads to the formation of Si-Si crosslinks within the solid polysilane, which reaction usually takes place up to about 375°C. Another possible reaction is referred to as the Kumada rearrangement, which typically occurs at temperatures between about 225°C to about 350°C, wherein the Si-Si backbone chain becomes a Si-C-Si backbone chain. While this type of reaction is usually used to produce a non-volatile product, the Kumada re-
arrangement can produce volatile polycarbosilane oligomers. While the amount of gaseous species produced by way of the Kumada rearrangement competes with the production of non-volatile solid or liquid polycarbosilane, the production of such species, while detrimental to the overall yield, can prove a useful aspect of the gas evolution process in that any material, liquid or solid that is left is in some embodiments turned into a harmless and safe ceramic material, leading to safer handling of the material once the process is terminated.

The heating of the solid polymeric source in the heating chamber may be performed by electrical heating, UV irradiation, IR irradiation, microwave irradiation, X-ray irradiation, electronic beams, laser beams or the like. In one embodiment, the solid organosilane is heated in a vessel to a suitable temperature, for example from 100 to 650°C, from 300 to 450°C, or from 350 to 420°C. A higher temperature will normally increase the rate at which the mixture of gaseous organosilicon compounds is produced from the polysilicon compound.

The solid organosilicon compound may be added to the heating vessel in a batch or continuous manner as a powder, pellet, rod or other solid forms. In batch addition, the solid organosilicon compound may be added, for example, in an amount in the range of from 1 mg to 10 kg, although larger amounts may also be used.

In one embodiment the vessel is purged, optionally under vacuum, after the solid organosilicon compound has been added to replace the gases within the vessel with an inert gas, such as argon or helium. The vessel can be purged before heating is commenced, or the temperature within the vessel can be increased during, or prior to, the purge. The temperature within the vessel during the purge should be
kept below the temperature at which evolution of the gaseous organosilicon compounds commences to minimise losses of gaseous organosilicon compounds.

In one embodiment, the vessel is heated at a rate of up to 150°C per hour until the desired temperature is reached, at which temperature the vessel is maintained. In another embodiment, the temperature is increase to a first value at which pyrolysis proceeds, and then the temperature is changed on one or more occasion, e.g. in order to vary the rate at which the mixture of organosilicon compounds is produced or to vary the pressure within the vessel.

Once the vessel has attained a suitable temperature, the pressure within the vessel is maintained at a predetermined pressure or within a predetermined pressure range that is selected to provide the desired molar ratio of gaseous organosilicon compounds in the produced mixture.

In one embodiment, the major contributor to the pressure within the vessel is the mixture of gaseous organosilicon compounds prepared by the process, while a minor contributor to the pressure is the gas present in the vessel before heating of the solid organosilicon compound is commenced, for example the inert gas introduced during the optional purging step. In order to regulate the pressure within vessel, the temperature of the vessel can be varied, or gas can be removed from the system. The temperature affects the pressure within the vessel e.g. by expanding the gases present in the vessel and by increasing the rate at which new gaseous organosilicon species are produced from the solid polysilicon compound. Variations in temperature can therefore be used to increase or decrease the pressure within the vessel. The pressure within the vessel can also be reduced by releasing gases therefrom.
Generally, maintaining a high pressure favours the production of gaseous organosilicon species having a lower number of silicon atoms, while maintaining a lower pressure favours the production of gaseous organosilicon species having a higher number of silicon atoms. This previously unknown relationship having now been discovered, it will be possible for a person skilled in the art to determine, through non-invention experimentation, which pressure to maintain in order to produce a mixture of gaseous organosilicon compounds having the desired relative amounts of gaseous species.

The overall pressure within the vessel, and the facility with which the pressure within the vessel will be controlled, can also be affected by the volume of the vessel used. In one embodiment, use of a larger vessel will promote a more stable pressure within the vessel when a portion of the gaseous organosilicon compounds is removed from the vessel, along with a more stable exit pressure for the gases removed from the vessel.

In one embodiment, the pressure is maintained from 50 to 900 psi. In another embodiment, the pressure is maintained from 100 to 250 psi or from 600 to 900 psi, each pressure range promoting the production of different mixtures of gaseous organosilicon species. When production of gaseous organosilicon compounds according to the method of present invention is carried out at lower pressures, e.g. from 100 to 250 psi, the production of gaseous compounds having larger molecular weights is promoted. Accordingly, in one embodiment pyrolysing the solid polysilane at a pressure from 100 to 250 psi produces a mixture of gaseous organosilicon compounds comprising at least 50-mol% of gaseous organosilicon compounds having 3 or 4 silicon atoms. Conversely, when production of gaseous organosilicon
compounds according to the method of present invention is carried out at higher pressures, e.g. from 600 to 900 psi, the production of gaseous compounds having larger molecular weights is promoted. Accordingly, in one embodiment pyrolysing at a pressure from 600 to 900 psi produces a mixture of gaseous organosilicon compounds comprising at least 50-mol% of gaseous organosilicon compounds having 1 or 2 silicon atoms.

Release and processing of gas mixture

Once the pressure within the vessel has reached a value within the selected pressure range, a portion of the gases within the vessel can be released, while insuring that the pressure within the vessel remains within the selected pressure range.

In one embodiment, the gases released are further processed, e.g. the gas mixture can be used in a deposition process, the mixture can be admixed with further gases, or the gas mixture can be stored.

For example, the vessel can be fitted with a relief valve and a storage reservoir such that the pressure within the vessel cannot rise above that set by the relief valve, effectively determining the maximum pressure within the vessel. The volume of the storage reservoir, which can be e.g. from 20 to 50 litters, will be determined by the pressure setting of the relief valve and the gas flow requirements of the intended use for the gaseous organosilicon compounds.

In a further example, the mixture of gaseous organosilicon compounds can be mixed with one or more further gases. The one or more further gases may be obtained already in gas form and admixed with the produced gas mixture outside the vessel, or the gases can produced by heating, optionally in
the vessel together with the solid polysilicon compound, a solid or liquid source comprising any number of elements, such as N, O, F, B, P, or a combination thereof. For example, the reactant gas may be produced by heating a solid source comprising phosphorous such as triphenylphosphine \((C_6H_5)_3P\); a solid source comprising nitrogen such as tris (pyrazol-1-yl) methane; or a solid source comprising boron such as borane t-butylamine \((CH_3)_3CNH_2\).BH_3, triethanolamineborate \(B(OCH_2CH_2)_3N\), borane dimethylamine \((CH_3)NH\cdot BH_3\), or triphenylboron \(B(C_6H_5)_3\). In another example, the reactant gas may be produced by heating a liquid source comprising fluoride such as difluorobenzene \((CeH_4F_2)\); a liquid source comprising phosphorous such as triethylphosphine \((C_2H_5)_3P\), dimethylphenylphosphine \((CH_3M C_6H_5)P\), or tris (trimethylsilyl) phosphine \([(CH_3)_3Si]_3P\); or a liquid source comprising boron such as tris (trimethylsiloxy) boron \([(CH_3)_3SiO]_3B\). In still another example, the reactant gas may be a nitrogen based gas such as NH_3, N_2, or NCl_3; an oxygen based gas such as CO, O_2, O_3, CO_2; a fluorine based gas such as CF_4, C_4F_8, CH_2F_2, NF_3, C_2F_6, C_3F_9, CHF_3, C_2F_4, C_3F_6, or a combination thereof; a boron based gas such as BH_3, B_2H_6, BCl_3, B_2Cl_6; or a phosphorous based gas such as PH_3 or PCl_3. In an embodiment, the reactant gas may also comprise Al, B, Ge, Ga, P, As, N, In, Sb, S, Se, Te, In and Sb.

**Apparatus and control methods**

Embodiments of the present invention have been described above primarily in the context of gas production methods. Other embodiments include, for example, apparatus and associated control methods, as described in further detail below.

It will be apparent from the foregoing that embodiments of the invention produce a gas or gas mixture from a solid
organosilane. The gas can be used, for example, as an input to a deposition tool of some kind, such as a CVD or PECVD deposition tool. An apparatus and gas cabinet including various physical hardware components, control methods used to obtain the gas, and an associated software implementation of the control method are all described below.

Figure 3 is a block diagram of a system according to an embodiment of the invention. In general terms, the overall system shown in Figure 3 includes a gas source, to create the gas, and a deposition tool. As shown, the gas source includes a heated pressure vessel in which the gas is produced and a gas panel for conditioning the gas.

The input to the deposition tool, in the example shown, includes an array of mass flow controllers (MFC) feeding a mixing manifold. Each MFC controls the flow of a specific gas. However, in other embodiments, the MFCs and the mixing manifold might be included in a gas cabinet at the gas source to allow a single gas mixture to be delivered to the deposition tool. A mixture of the output of the gas source with other gases, such as ammonia or nitrogen, may be used to give a desired film produced by the deposition tool, for instance.

One specific example layout of components of a gas panel is discussed below. It is to be noted that other layouts could deliver substantially the same functionality and meet the requirements of various implementations and applications. For example, the relative locations of components may be taken into consideration for minimising dead spaces between valves on the gas panel and/or minimising dead space between the gas source and the deposition tool.
Thus, it should be appreciated that the example layout described below is not intended to be restrictive, but rather illustrative of one specific embodiment.

Figure 4 is a schematic diagram of an example gas panel layout. The gas panel includes a nitrogen inlet, a vent outlet, a purge gas inlet shown in the drawing as an argon inlet, a process gas outlet, and various valves for controlling gas flow through the gas panel. A pressure gauge, pressure sensors, and a vacuum generator with integrated valves are also provided in the example gas panel.

Although the gas panel is connected to vessel components in the example shown, the vessel components and the gas panel could be provided separately. For example, a gas panel and controller, described below, might be sold with a gas cabinet. The vessel components could be separately acquired by a customer. The vessel components in the example shown include a sealable vessel itself, for holding a solid sample from which gas is to be produced, as well as gas inlet and outlet valves, a relief valve, a pressure sensor, temperature sensors, and a heater.

The table below lists specific devices that could be used to implement the various components shown. The valve P1, for example, could be an air actuated diaphragm valve. This type of device and the other examples listed in the table are intended solely for illustrative purposes, and the present invention is in no way limited thereto.

<table>
<thead>
<tr>
<th>LABEL</th>
<th>DESCRIPTION</th>
<th>FUNCTION</th>
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<tbody>
<tr>
<td>P1</td>
<td>Air Actuated Diaphragm Valve.</td>
<td>Vent outlet isolation.</td>
</tr>
<tr>
<td>P2</td>
<td>Air Actuated Diaphragm Valve.</td>
<td>Ar inlet isolation.</td>
</tr>
<tr>
<td>P3</td>
<td>Air Actuated Diaphragm Valve.</td>
<td>Crossover isolation.</td>
</tr>
</tbody>
</table>
In operation, the gas panel introduces gases into and/or carries gases out of the vessel. Gas flow through the gas panel is controlled using the valves. Some of the valves M1, M2 are manually operated, and others are automatically controlled by a controller, such as the valves P1, P2, P3, P4, P5, P6, P7, and P9. The check valves SV1, SV2, the pressure regulators R1, R2 are not explicitly controlled, but operate to open and close under certain pressure conditions.

The pressure gauge G1 provides a visual indication of pressure in the gas panel, and the pressure gauge G2 provides a visual indication of pressure in the vessel. The

<table>
<thead>
<tr>
<th>LABEL</th>
<th>DESCRIPTION</th>
<th>FUNCTION</th>
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<tbody>
<tr>
<td>P5</td>
<td>Air Actuated Diaphragm Valve.</td>
<td>Vessel pump/purge isolation.</td>
</tr>
<tr>
<td>P7</td>
<td>Air Actuated Diaphragm Valve.</td>
<td>Vessel inlet isolation.</td>
</tr>
<tr>
<td>P9</td>
<td>Air Actuated Diaphragm Valve.</td>
<td>Vessel outlet isolation.</td>
</tr>
<tr>
<td>SV1</td>
<td>Check Valve.</td>
<td>Ar inlet check valve.</td>
</tr>
<tr>
<td>SV2</td>
<td>Check Valve.</td>
<td>Vessel check valve.</td>
</tr>
<tr>
<td>Heater</td>
<td>Heater.</td>
<td>Vessel heater.</td>
</tr>
<tr>
<td>G1</td>
<td>Pressure Gauge.</td>
<td>Vessel pressure gauge.</td>
</tr>
<tr>
<td>G2</td>
<td>Pressure Gauge.</td>
<td>Ar gas pressure gauge.</td>
</tr>
<tr>
<td>SP1</td>
<td>Pressure Sensor.</td>
<td>Vessel pressure sensor.</td>
</tr>
<tr>
<td>SP2</td>
<td>Pressure Sensor.</td>
<td>Purge/vacuum pressure sensor.</td>
</tr>
<tr>
<td>SP3</td>
<td>Pressure Sensor.</td>
<td>Process gas outlet pressure sensor.</td>
</tr>
<tr>
<td>R1</td>
<td>Pressure Regulator.</td>
<td>Ar gas pressure regulator.</td>
</tr>
<tr>
<td>R2</td>
<td>Pressure Regulator.</td>
<td>Process gas outlet pressure regulator.</td>
</tr>
<tr>
<td>T1</td>
<td>Temperature Sensor.</td>
<td>Vessel temperature sensor.</td>
</tr>
<tr>
<td>T2</td>
<td>Temperature Sensor.</td>
<td>Heater temperature sensor.</td>
</tr>
<tr>
<td>V1</td>
<td>Vacuum Generator (with integral Air Actuated Valve and Check Valve).</td>
<td>System vacuum generator, with N2 purge/vacuum drive isolation and N2 supply check valve.</td>
</tr>
</tbody>
</table>
pressure sensors SP1, SP2, SP3 similarly measure pressure, but may instead provide respective indications of measured pressure to system components such as a controller. Indications of measured temperatures within the vessel and at the Heater may be provided by the temperature sensors T1, T2.

One function of the gas panel, and in particular the valve P4, is to control a flow of gas from the gas outlet of the vessel to the process gas outlet. The valve P4 can thereby be used to control pressure within the vessel. In one embodiment, a controller, which is implemented at least in part in software in some embodiments, controls the Heater to heat a solid organosilicon compound in the vessel at a predetermined temperature, illustratively from 350 to 500°C, while a predetermined pressure, illustratively from 100 to 900 psi, is maintained inside the vessel by the valve, to thereby form a mixture of gaseous organosilicon compounds.

Such a controller may control both the Heater and the valve P4 to maintain desired temperature and pressure conditions. The controller may receive indications of a temperature and a pressure inside the vessel from the sensors T1 and SP1, and control the Heater and the valve P4 based on the sensed temperature and pressure, for example.

The controller may also in some embodiments control an outlet valve of the vessel, if one is provided. The apparatus shown in Figure 4 includes a vessel outlet valve at the top right of the vessel, which can be controlled by the controller to control a flow of gas from the gas outlet of the vessel to the gas panel.

Gases from the vessel can be vented through the gas panel and the vent outlet. The valve P6 provides one possible vent path through the gas panel, and another path is
provided through the relief valve SV2 of the vessel and the
gas line connected to that relief valve. Whereas the valve
P6 is under automatic control in the example shown, the
relief valve SV2 allows gas to flow from the vessel to the
vent outlet when the pressure inside the vessel exceeds a
maximum pressure.

Prior to a gas production cycle, it may be desirable to
purge the vessel. In the example shown in Figure 4, argon
is used as an inert gas for purging the vessel. The valve
P2 is automatically controlled to control a flow of the
argon gas from the purge inlet to the gas inlet of the
vessel. The manual valve M1, the check valve SV1 and the
pressure regulator R1 are also involved in introducing a
purge gas into the vessel. During a purge cycle, the vacuum
generator V1 is automatically controlled to generate a
vacuum pressure. Nitrogen from the N2 inlet is used to
drive the vacuum generator V1, a Venturi pump in one
embodiment. Purging of the vessel is described in further
detail below.

The apparatus shown in Figure 4, including the gas panel
and/or the vessel, may be implemented in a gas cabinet. Gas
panel, controller, and/or vessel components are mounted to a
housing of the cabinet in some embodiments.

Figure 5 is a diagram of an example gas cabinet, which
includes a gas panel, which may be the gas panel shown in
Figure 4, and a control unit. The gas cabinet in Figure 5
includes an air vent inlet at its lower left-hand side, and
an air vent outlet at the top, from which air might be
extracted, for example, at a rate of about 100 cfm. The
control unit may be operatively coupled to a PC or other
system, illustratively through a Universal Serial Bus (USB)
or other type of wired or wireless connection.
The particular example layout shown in Figure 5 is intended solely for the purposes of illustration. Sizes, shapes, layouts, and other physical characteristics are of course subject to change between different implementations or applications.

Other components may also be provided, even though not explicitly shown in Figure 5. A user interface may be provided, for example, in the form of a control interface panel on the control unit, that both receives inputs from a user and provides outputs to a user. A PC, shown in Figure 5 as coupled to the control unit, represents another form in which a user interface could be provided.

The various control functions described herein could be implemented in the control unit, or in some embodiments in a separate component such as the PC or a control terminal that is operatively coupled to the control unit.

A connector between a gas cabinet and the control unit could take any of various forms. Those skilled in the art will appreciate that connector layouts and types may vary between different embodiments.

Several example operating scenarios of a gas panel will now be considered in detail. In particular, the following numbered sequence describes operations for setting up the gas source, including loading. Alarms during set-up and operation, as well as other modes of operation, are subsequently described.

The following is an example instruction set for charging the vessel and bringing into use:

<table>
<thead>
<tr>
<th>Step</th>
<th>Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>(Start)</td>
</tr>
</tbody>
</table>
All valves closed.

Verify if the starting mode is hot or cold mode. Hot mode may occur after pressing the emergency button or an electrical power supply failure.

If the vessel pressure is above "minimum pressure restart" AND the vessel temperature is above "minimum temperature restart", then this is a hot mode restart; go to step 12.

If not, cold mode restart; go to step 3.

If the vessel pressure is above "maximum pressure to purge" open valve P6 and outlet valve P9 of the vessel to vent the vessel so as to lower the pressure to "maximum pressure to purge", illustratively 10psi; go to step 4.

If not, go to step 4.

Close valve P6 and outlet valve P9 of the vessel.

Put the vessel in recharge mode, in a solid source sample or cartridge can be replaced.

Wait until the temperature on the temperature sensor T1, illustratively a thermocouple, inside the vessel reads lower then "min temp temperature to unload".

Start the vacuum Venturi V1.

Open valve P1, verify minimum vacuum after a predetermined time on SP2.
If so, open valve P5, P3 and the inlet valve P7 of the vessel.

Otherwise, close all valves and raise alarm "low vacuum".

(Purge the vessel)

Open inlet valve P7 of the vessel. Wait to get "vacuum for good purge", then close valves P5, P1.

(Refill the vessel)

Open valve P2 and wait fill time or pressure for purge on SP1, then close valve P2.

Repeat steps 5 to 7 for "purge repeat" time.

Close the inlet valve P7 of the vessel and valve P3.

If in recharge mode, go in step 17.

If in charged mode, go to step 10.

Remove the charge mode, place in ready mode.

Ramp heating with maximum ramp of 150°C per hour to 500°C for 3 hours, and then ramp down with 150°C/hour to 450°C. Stay at this temperature until "low pressure" is reached.

Open the outlet valve of the vessel.

If the pressure sensed by pressure sensor SP1 goes lower than a minimum, "low pressure" is reached; go to 13.
When the temperature of 400°C has been reached for 1 hour, reheat vessel.

If the pressure sensed by pressure sensor SP1 goes lower than the minimum, then "low pressure" is reached; go to 13.

Open outlet valve P9 of the vessel.

Open valve P4.

If the pressure sensed by pressure sensor SP1 goes lower than minimum, "low pressure" has been reached; go to 13.

When "low pressure" is reached, close valve P4.

Stop heating.

Open valve P6 and wait until "purge pressure max" is reached at pressure sensor SP3.

Close valve P6.

Remove the ready mode and place in recharge mode.

Go to step 4.

(Recharge vessel)

Prompt operator to unscrew vessel cover and remove.

Wait for feedback.

Prompt operator to close and remove vessel liner.

Wait for feedback.
Remove the recharge mode, and place in charge mode.

Prompt operator to place new liner in vessel and open new liner.

Wait for feedback.

Prompt operator to install new seal and close vessel cover.

Wait for feedback.

Prompt operator to replace vessel cover bolts.

Wait for feedback.

Prompt operator to close door of cabinet.

Wait for feedback.

Remove the recharge mode and place in charge mode.

Go to step 5.

Other possible modes of operation may include a maintenance mode to allow opening of all the valves. A purge mode in which the valve P6 and the outlet of the vessel are opened, possibly with warning, could also be provided. In a standby mode, the process gas would not be being drawn from the unit or the unit is ready to be driven into gas producing mode. For example, the vessel might be charged with a solid sample and pre-heated but it is not yet hot enough to release process gas. One purpose of sitting at a pre-heat temperature would be to lessen the time to reaching the temperature at which the process gas would be released from the solid polymer.
These modes and/or possibly others may be implemented in a controller and executed automatically when selected by a user. An automatically executed mode might have control points or delays to allow an operator to perform certain tasks, such as removing the vessel cover, removing a liner, etc. Automatic operations associated with a current mode may then proceed upon receipt of feedback from the operator, as will be apparent, for example, from steps 17 to 22 above.

Various interlocks could also be provided. For example, it may be desirable to not permit opening of the valve P3 when the pressure sensed by SP1 is above 100psi and outlet valve P9 of the vessel is open. Other possible interlocks may include, for example, preventing the inlet valve of the vessel from being opened if the pressure sensed by SP1 is above 100psi, preventing the valve P4 from being opened if SP3 is not at a proper operating pressure, preventing the valve P6 from being opened if the valve P4 is open, and preventing the Heater from being turned on if the system is not in ready mode.

Alarms are also contemplated, and may include any or all of the following, among others:

- If low purge pressure, turn on a "low purge pressure" alarm light and wait until this is resolved.

- If the temperature is above "high temperature inside" on T1 or "high temperature heater" on T2, stop the heating, and provide a temperature alarm. A user may then press a reset button or provide some other input to restart the above sequence.

- If the pressure goes above a maximum at one or more of the pressure sensors, then a "high pressure alarm" is raised and the heating is stopped. When a user presses a
reset button or provides another input, the above sequence could be restarted.

- If the cabinet door is opened, heating is stopped, the vessel outlet and inlet valves are closed, and a door open alarm light is illuminated until the door is closed.

- If any power failure, raise an alarm to provide an indication of the failure, and reset to step 1.

The status of various valves and components under different example modes of operation are listed in the table below.

<table>
<thead>
<tr>
<th>Mode of operation</th>
<th>Step 1</th>
<th>Step 2</th>
<th>Step 3</th>
<th>Step 4</th>
<th>Step 5</th>
<th>Step 6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Recharge Vessel</td>
<td>P1</td>
<td>P2</td>
<td>P3</td>
<td>P4</td>
<td>P5</td>
<td>P6</td>
</tr>
<tr>
<td>Vessel Pressure</td>
<td>close</td>
<td>close</td>
<td>close</td>
<td>close</td>
<td>close</td>
<td>close</td>
</tr>
<tr>
<td>Evacuate Vessel</td>
<td>open</td>
<td>close</td>
<td>close</td>
<td>close</td>
<td>close</td>
<td>open</td>
</tr>
<tr>
<td>Purge Vessel</td>
<td>close</td>
<td>close</td>
<td>close</td>
<td>close</td>
<td>open</td>
<td>close</td>
</tr>
<tr>
<td>Pump/Charge Vessel</td>
<td>open</td>
<td>close</td>
<td>close</td>
<td>close</td>
<td>close</td>
<td>close</td>
</tr>
</tbody>
</table>

The table below defines example alarm options.
The tables below define inputs and outputs for one embodiment of a gas panel controller.

### DIGITAL INPUTS (CPU)

<table>
<thead>
<tr>
<th>I/O</th>
<th>ID</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>X2</td>
<td>DS1</td>
<td>Door Switch-1</td>
</tr>
<tr>
<td>X5</td>
<td>EGO</td>
<td>Local EGO</td>
</tr>
</tbody>
</table>

### DIGITAL OUTPUTS (CPU)

<table>
<thead>
<tr>
<th>ID</th>
<th>Output Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>Transistor (NPN) Vac Vent</td>
</tr>
<tr>
<td>P2</td>
<td>Transistor (NPN) Argon process</td>
</tr>
<tr>
<td>P3</td>
<td>Transistor (NPN) Crossover inlet outlet vessel</td>
</tr>
<tr>
<td>P4</td>
<td>Transistor (NPN) Process outlet</td>
</tr>
<tr>
<td>P5</td>
<td>Transistor (NPN) Venturi isolation</td>
</tr>
<tr>
<td>P6</td>
<td>Transistor (NPN) Process vent</td>
</tr>
<tr>
<td>P7</td>
<td>Transistor (NPN) Pressure vessel inlet</td>
</tr>
<tr>
<td>P9</td>
<td>Transistor (NPN) Pressure vessel outlet</td>
</tr>
<tr>
<td>V1</td>
<td>Transistor (NPN) Venturi</td>
</tr>
</tbody>
</table>

### DIGITAL OUTPUTS (SLOT 1)

<table>
<thead>
<tr>
<th>I/O</th>
<th>ID</th>
<th>Output Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Y20</td>
<td>HORN</td>
<td>Transistor (NPN) HORN</td>
</tr>
<tr>
<td>Y21</td>
<td>CR2</td>
<td>Transistor (NPN) WARNING</td>
</tr>
<tr>
<td>Y22</td>
<td>CR3</td>
<td>Transistor (NPN) ALARM</td>
</tr>
</tbody>
</table>
Additional operating modes and operations that may be involved in using a gas cabinet that incorporates a gas panel and vessel are described below.

As will be apparent from the above sequence of operations, the pressure vessel is opened to recharge the solid source polymer from which gas is produced. After the polymer inside the pressure vessel has been heated, to above 200°C for instance, a drop in the pressure inside the vessel as measured by the pressure sensor SP1 to below a certain value may be an indication that the source polymer is close to exhaustion. In one embodiment, 40 psi is the suggested minimum operating pressure. The operations below outline the steps to open the pressure vessel, including cool down and removal of source gas.

Emptying the Pressure Vessel

Step 1 - Cool down the pressure vessel

- Turn off the vessel heat supply Heater, using a power switch on the control unit, for example. The cool-down time of the vessel can potentially be accelerated by
loosening insulation around the vessel and removing the Heater.

Step 2 - Remove Gaseous Polymer Fractions from the Pressure Vessel

5 • Ensure that the temperature inside the pressure vessel is below 200°C, as indicated on T1.

• Ensure that the vacuum supply provided by the vacuum generator V1 is greater than a minimum, illustratively 100 psi.

10 • Place the software control system in the 'manual valve controls' operating mode.

• Open P1, to activate the vacuum generator V1.

• Allow the pressure indicated on SP2 to stabilise. This is the 'base pressure'.

15 • Pump down the pressure vessel by opening P5 and P7.

• Allow SP2 to reach within a certain range, illustratively 2 psi, of the 'base pressure'.

• Close P1, P5 and the vacuum generator V1.

20 Step 3 - Fill the pressure vessel with argon, illustratively at 40 psi

• Ensure that the argon inlet is pressurised to >40 psi and the manual valve M1 is open.

• Open P2.

25 • Let the pressure inside the pressure vessel (SP1) to stabilise to purge pressure, illustratively 40psi.
Close P2 and P7.

Step 4 - Conduct the 'pump-purge' steps a number of times, illustratively six times

Assuming a 20:1 dilution each cycle, this should give > 60 million: 1 dilution.

Step 5 - Open the pressure vessel

Ensure that the temperature inside the vessel is below 80°C on T1.

Initial commissioning operations may also be supported. This may involve, for example, checking the operation of the controller and possibly other electronics, leak testing the pipe work, valves, and pressure vessel, and verifying operation of the heater up to operating temperature.

After preparing a gas cabinet for operation, which may include, for example, connecting the gas cabinet to an electrical supply (240 to 260 Volts, 20 Amps in one embodiment), to a compressed air (or nitrogen) supply, illustratively at 130 psi, for the vacuum generator V1, to an argon or other inert gas supply (at a minimum of 40 psi in one embodiment) for purge gas, and to an extraction unit, which operates at 400 cfm and provides a scrubbing function in one embodiment, initial commissioning may proceed as follows.

Step 1 - Installing and connecting the electronics/control unit

Place the electronics/control unit, illustratively onto the top or side of the gas cabinet, ensuring that the connections and leads enter into the cabinet.
• Connect CDA outlets (in the electronics/control box) to the appropriate valves. The CDA outlets control valves in the gas panel and the vessel inlet and outlet valves. Different numbers of valves and different types of connections than CDA outlets may be provided in other embodiments.

• Pass heater wires through the cabinet and into the electronics/control unit, to provide for control of the Heater.

• Attach the electronics/control unit to the cabinet using fasteners, illustratively a nut and bolt through each of four holes in the corners of the electronics/control unit, with two washers between the cabinet and the electronics/control unit in each position to a separation of 1 mm, reducing thermal contact.

Step 2 - Check the operation of the valves

• Place a PC, which is configured with control software and is connected to the electronics/control unit in one embodiment, in manual operation.

• Operate each valve with the CDA line disconnected at the valve, to ensure that the correct CDA line for the valve operates. All valves should be normally closed in one embodiment.

Step 3 - Conduct a pressure drop test

This should be conducted using the supply from the vacuum generator V1, a Venturi pump in one embodiment, to give the maximum possible gas pressure. Each time an instruction is given to observe the pressure drop on a pressure transducer, it should be observed, illustratively for 1 minute and not drop by more than 1 psi according to one possible testing
scheme. Other observation periods and/or pressure drops could be used in other embodiments, such as a 5 psi drop over one hour, or other values that are reasonable to verify that there are no leaks.

5
• Open an internal valve in V1 until the pressure on SP2 stabilises.
  • Close valve V1 and observe the pressure on SP2.
  • Close manual valve M1.
  • Open valves P5, P3, P2 and manual valve M1 until the pressure on SP2 stabilises.
  • Close valve V1 and observe the pressure on SP2. This is now performing a leak test on the gas panel.
  • Close valves P3, P2 and manual valve M2.
  • Open valve V1 and valve P7 until the pressure on SP2 stabilises.
  • Close valve V1 and observe the pressure on SP2. This is now performing a leak test on the pressure vessel.
  • Close all valves.

Step 4 – Initial heating of pressure vessel

20 The pressure vessel is heated at this stage without any source polymer loaded, but with an overpressure of the Venturi gas. This is to achieve the following:
  • Test the operation of the Heater, temperature measurement and control.
  • Ensure that the vessel is leak-tight at a higher pressure and operating temperature.
Clean up the vessel by out-gassing to give a standard starting point for the operation.

Next steps:

- Place the PC in manual operation.
- Check the set points of all alarms and warnings on the PC.
- Remove the lid of the pressure vessel.
- Clean the gasket and inside the pressure vessel, illustratively with acetone and clean-room wipes.
- Ensure that there is no grease on the connector to the thermocouple feed through in the vessel lid.
- Replace the gasket for the pressure vessel.
- Replace the pressure vessel lid.
- Install lid bolts, and tighten to 250 foot-pounds, on alternate sides of the vessel.
- Replace thermocouple and heater jackets.
- Ensure that the heater and vessel thermocouples are correctly connected to their respective wires into the electronics/control unit. The vessel thermocouple (T1) might display on a display of the electronic/control unit, whereas the heater thermocouple (T2) displays only on the PC in some embodiments.
- Fill the vessel with gas from the Venturi line (the N2 inlet in Figure 4) to a certain pressure, >100 psi in one embodiment.
- Open valves V1, P5, P7.
Observe the pressure rise in the vessel (SP1).

Close valves V1, P5, P7.

Ensure that the Heater HT1 is switched on.

Select a temperature, illustratively 420°C, on the heater display on the electronics/control unit.

Observe the temperature rise on the PC and the heater display.

Ensure that the display on T2 rises faster than that on T1.

Observe the pressure inside the vessel to ensure that it remains within safe limits and does not reduce.

Step 5 - Cool down the pressure vessel

Select a lower temperature, illustratively <20°C, on the heater display.

Turn the Heater off.

Monitor the Heater and vessel temperatures.

The insulation jacket and Heater can be removed.

Various embodiments of the invention have been described above, primarily in the context of methods and apparatus of producing gas from a solid polymer source. Another aspect of the invention relates to control methods for controlling gas cabinet components. Such methods may be implemented, for example, in electronics of an electronics/control unit, or in software for execution by a processing element such as a microprocessor in a PC or other terminal operatively coupled to the electronics unit. Other processing elements that may be suitable for this purpose may include
microcontrollers, Field Programmable Gate Arrays (FPGAs), and Programmable Logic Devices (PLDs). Application Specific Integrated Circuits (ASICs) could also or instead be used in implementing controller functions. In terms of actual software, control software could be written in Visual Basic or Labview, for example, although the present invention is in no way limited to any particular programming language(s) or software application(s).

Given the many options for implementing controller functions, it should be appreciated that a controller may be implemented in one or more physical devices. A PC may be co-located with a gas cabinet or remotely located with some sort of communication connection to the gas cabinet, through the electronics unit for instance. Thus, references herein to a controller should be interpreted accordingly, to include controller implementations in one or more devices, which may be configured by software to perform control functions.

A control method might include determining a temperature inside the vessel, controlling the heater based on the determined temperature to heat a solid organosilicon compound at a predetermined temperature, and maintaining a predetermined pressure inside the vessel while the solid is heated, to thereby form a mixture of gaseous organosilicon compounds. The pressure is maintained in one embodiment by controlling a valve that controls a flow of gas from a gas outlet of the vessel through the gas panel.

Both temperature and pressure may be monitored in some embodiments, by receiving temperature and pressure indications from sensors. The heater and valve can then be controlled based on the temperature and pressure inside the vessel.
Venting, pressure relief, and purge functions may also be provided. These and possibly other control functions may be or become apparent to those skilled in the art, from the foregoing production method and apparatus descriptions for instance.

Although the foregoing invention has been described in some detail by way of illustration and example for purposes of clarity of understanding, it is readily apparent to those of ordinary skill in the art in light of the teachings of this invention that certain changes and modifications may be made thereto without departing from the spirit or scope of the appended claims.

For example, control of vessel pressure may be provided by a mechanism other than the valve P4. According to another embodiment, pressure control is provided using a reservoir in the gas panel and a relief valve in either the gas panel or the vessel. Such an arrangement would allow process gas to flow from the vessel to the reservoir when pressure inside the vessel exceeds a maximum pressure. The pressure in the heated vessel then cannot rise above that set by the relief valve, effectively setting the maximum pressure in the heated vessel and also determining the environmental pressure during the gas evolution process and the associated impact on the type of gases produced. The process gas is retained in the separate reservoir in this arrangement, but will not exceed the pressure set by the pressure relief valve. As the gas is drawn off from the reservoir, such as by a deposition system, the pressure in the reservoir will drop accordingly. The volume of the reservoir would be dependent on the pressure setting of the relief valve and the gas flow requirements of the deposition tool in a deposition application. 20-50 litres may be reasonable in some embodiments.
The form of a controller user interface may also vary. A Graphical User Interface (GUI) might be provided, on an electronics/control unit, a PC operatively coupled to that unit, or both. A GUI might be used to display a representation of each component of a gas cabinet, and its current position, operating status, and/or alarm(s). These representations could also provide access to control functions for those components.

It should also be noted that various functions described above could be implemented in instructions stored on a computer-readable medium.

EXAMPLES

The following examples are provided to illustrate the invention. It will be understood, however, that the specific details given in each example have been selected for the purpose of illustration and are not to be construed as limiting the scope of the invention.

EXAMPLE 1

400 grams of polydimethylsilane (PDMS) were placed in a 2 liter stainless steel vessel provided with a heating unit and a pressure sensor. Once sealed, the gas within the vessel was purged and replaced with argon. The vessel was then heated at a rate of about 150 °C per hour for three hours to about 500 °C, and the temperature was then reduced to about 422 °C. While maintaining the temperature substantially constant, gaseous products obtained were released from the vessel at 805 psi, thus reducing the pressure. Between releases of the gaseous products, the pressure within the vessel was allowed to increase as additional gaseous products were produced from the polydimethylsilane. The pressure in the vessel was
maintained between 600°C and 900 psi. After 300 hours of heating and 89 separate releases of gas mixture, representing 4848 minutes of gaseous product release, the pressure within the vessel was noted to no longer increase between releases, indicating that the PDMS load was spent. The solid product remaining in the vessel after the reaction was found to weight 261 grams, indicating that 139 grams of polydimethyilsilane were converted to gaseous organosilicon compounds during the process.

Figure 1 provides a graphical representation of the evolution of the mixture of gaseous organosilicon compounds from PDMS as the vessel containing the PDMS is heated. At about 300°C, the solid becomes liquid which can be noted by the slight change in slope of pressure. At about 400°C, a rapid rise in pressure is observed as the gas mixture evolves from the liquid.

Figure 2 provides graphical representation of the pressure within the vessel over the course of the process of Example 1 as the mixture of gaseous organosilicon compounds is released from the vessel. The points on the curve represent each release of gases from the vessel. In the exemplified process, each release represented a flow of gas to a PECVD system sufficient to complete a film deposition at a thickness of 80-100nm.

Table 1 provides the results from GC-MS analysis of the gas mixture produced from the process described in Example 1. The table also provides potential identification of the gaseous organosilicon compounds contained in the produced mixture, deduced from the GC-MS results.
<table>
<thead>
<tr>
<th>Group</th>
<th>Group %</th>
<th>Peak %</th>
<th>Elution time (min)</th>
<th>Possible structure /compound</th>
<th>Molecular Weight from Possible Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-Si</td>
<td>0.17</td>
<td>0.17</td>
<td>3 563</td>
<td>Unknown</td>
<td></td>
</tr>
<tr>
<td>1-Si</td>
<td>67.27</td>
<td>44.50</td>
<td>9.247</td>
<td>$\text{H}_2\text{C-Si-H}$</td>
<td>Molecular Weight = 74.20</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{CH}_3$</td>
<td>Inert Mass = 84</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{H}_2\text{C-Si-CH}_3$</td>
<td>Molecular Formula = $\text{C}_2\text{H}_5\text{Si}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{CH}_3$</td>
<td>Molecular Composition = C 45.66% H 13.99% Si 37.85%</td>
</tr>
<tr>
<td></td>
<td>8.30</td>
<td>9.387</td>
<td></td>
<td>$\text{H}_2\text{C-Si-CH}_3$</td>
<td>Molecular Weight = 68.25</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{CH}_3$</td>
<td>Exact Mass = 69</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{H}_2\text{C-Si-CH}_3$</td>
<td>Molecular Formula = $\text{C}_2\text{H}_5\text{Si}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{CH}_3$</td>
<td>Molecular Composition = C 54.46% H 13.77% Si 31.83%</td>
</tr>
<tr>
<td>14.47</td>
<td>Others</td>
<td></td>
<td></td>
<td>Dimethyldisilane</td>
<td></td>
</tr>
<tr>
<td>2-Si</td>
<td>32.59</td>
<td>6.30</td>
<td>11 422</td>
<td>$\text{H}_2\text{C-Si-Si-CH}_3$</td>
<td>Molecular Weight = 189.33</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{CH}_3$</td>
<td>Exact Mass = 18</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{H}_2\text{C-Si-Si-CH}_3$</td>
<td>Molecular Formula = $\text{C}_4\text{H}_6\text{Si}_2$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{CH}_3$</td>
<td>Molecular Composition = C 40.60% H 11.92% Si 47.47%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>18.87</td>
<td>13.11</td>
<td>$\text{H}_2\text{C-Si-Si-CH}_3$</td>
<td>Molecular Weight = 192.39</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{H}_2\text{C-Si-Si-CH}_3$</td>
<td>Exact Mass = 192</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{CH}_3$</td>
<td>Molecular Formula = $\text{C}_4\text{H}_6\text{Si}_2$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{CH}_3$</td>
<td>Molecular Composition = C 45.37% H 12.19% Si 42.44%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4.80</td>
<td>15 221</td>
<td>$\text{H}_2\text{C-Si-Si-CH}_3$</td>
<td>Molecular Weight = 146.58</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{H}_2\text{C-Si-Si-CH}_3$</td>
<td>Exact Mass = 146</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{CH}_3$</td>
<td>Molecular Formula = $\text{C}_4\text{H}_6\text{Si}_2$</td>
</tr>
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EXAMPLE 2

The process described in Example 1 was repeated, with the exception that the pressure within the vessel was maintained between about 100 and about 200 psi.

Table 2 provides the results from GC-MS analysis of the gas mixture produced from the process described in Example 2. The table also provides potential identification of the gaseous organosilicon compounds contained in the produced mixture, deduced from the GC-MS results.
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<th>Peak %</th>
<th>Elution time (min)</th>
<th>Possible structure / compound</th>
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<th>Molecular Formula</th>
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Example 3

The process described in Example 2 was repeated, with the exception that the pressure within the vessel was maintained below about 230 psi, and 450g of polydimethylsilane was used. The solid product remaining in the vessel after the reaction was found to weight 281 grams, indicating that 169 grams of polydimethylsilane were converted to gaseous organosilicon compounds during the process.

The citation of any publication, patent or patent application in this specification is not an admission that the publication, patent or patent application is prior art.

It must be noted that as used in the specification and the appended claims, the singular forms of "a", "an" and "the" include plural reference unless the context clearly indicates otherwise.

Unless defined otherwise all technical and scientific terms used herein have the same meaning as commonly understood to one of ordinary skill in the art to which this invention belongs.
Claims:

1. A method for producing from a solid organosilane compound a mixture of gaseous organosilicon compounds having a desired molar ratio, the method comprising the step of pyrolysing the solid organosilane while maintaining a predetermined pressure which is selected to provide the desired molar ratio of gaseous organosilicon compounds.

2. The method according to claim 1, wherein the desired molar ratio represents the molar ratio between gaseous organosilicon compounds having different numbers of silicon atoms.

3. The method according to claim 1 or 2, wherein the solid organosilane compound comprises a linear or branched polysilicon chain wherein each silicon is substituted by one or more hydrogen atoms, C1-C9 alkyl groups, phenyl groups or -NH3 groups.

4. The method according to claim 1 or 2, wherein the solid organosilane compound is polydimethylsilane (PDMS), polycarbosilane (PCMS), triphenylsilane, or nonamethyltrisilazane, preferably PDMS or PCMS.

5. The method according to claim 4, wherein the PDMS has a molecular weight from 1,000 to 1,700 g/mol.

6. The method according to any one of claims 1 to 5, wherein each gaseous organosilicon compound within the mixture is independently selected from a gaseous silane, a gaseous polysilane or a gaseous polycarbosilane.

7. The method according to any one of claims 1 to 5, wherein each gaseous organosilicon compound within the mixture comprises from 1 to 5 silicon atoms.
8. The method according to any one of claims 1 to 5, wherein the gaseous organosilicon compound mixture comprises compounds of the formula:

\[
\text{Si} \left(\text{CH}_3\right)_n \left(\text{H}\right)_m - \left[\left(\text{CH}_2\right)\text{Si} \left(\text{CH}_3\right)_p \left(\text{H}\right)_{z}\right]_x \text{Si} \left(\text{CH}_3\right)_n - \left(\text{H}\right)_m
\]

5 wherein

\(n, m, n', m'\) independently represent an integer from 0 to 3, with the proviso that \(n + m = 3\) and \(n' + m' = 3\),

\(p\) and \(q\) independently represent an integer from 0 to 2, with the proviso that \(p + q = 2\) for each silicon atom, and

\(x\) is an integer from 0 to 3.

9. The method according to any one of claims 1 to 8, wherein the pyrolysis is carried out at a temperature from 100 to 650°C.

10. The method according to any one of claims 1 to 8, wherein the pyrolysis is carried out at a temperature from 350 to 420°C.

11. The method according to any one of claims 1 to 10, wherein the pressure is maintained from 100 to 900 psi.

12. The method according to any one of claims 1 to 10, wherein the pressure is maintained from 100 to 250 psi, and the mixture of gaseous organosilicon compounds comprises at least 50 mol% of gaseous organosilicon compounds comprising 3 to 5 silicon atoms.

13. The method according to any one of claims 1 to 10, wherein the pressure is maintained from 100 to 200 psi, and the mixture of gaseous organosilicon compounds comprises at least 50 mol% of gaseous organosilicon compounds comprising 3 to 5 silicon atoms.
14. The method according to any one of claims 1 to 10, wherein the pressure is maintained from 600 to 900 psi, and the mixture of gaseous organosilicon compounds comprises at least 50 mol% of organosilicon compounds comprising 1 or 2 silicon atoms.

15. The method according to any one of claims 1 to 14, wherein the mixture of gaseous organosilicon compounds is further processed.

16. The method according to any one of claims 1 to 15, wherein the vessel is purged with an inert gas prior to heating.

17. The method according to claim 16, wherein the inert gas is argon or helium.

18. A method for producing a mixture of gaseous organosilicon compounds comprising at least 50 mol% of gaseous organosilicon compounds comprising 3 to 5 silicon atoms, the method comprising heating a solid organosilane compound at a temperature from 350 to 500°C under a maintained pressure from 100 to 250 psi.

19. A method for producing a mixture of gaseous organosilicon compounds comprising at least 50 mol% of gaseous organosilicon compounds comprising 1 or 2 silicon atoms, the method comprising heating a solid organosilane compound at a temperature from 350 to 500°C under a maintained pressure from 600 to 900 psi.

20. The method according to claim 18 or 19, wherein the solid organosilane compound comprises a linear or branched polysilicon chain wherein each silicon is substituted by one or more hydrogen atoms, C1-C6 alkyl groups, phenyl groups or NH3 groups.
21. The method according to claim 18 or 19, wherein the solid organosilane compound is polydimethylsilane (PDMS), polycarbomethylsilane (PCMS), triphenylsilane, or nonamethyltrisilazane, preferably PDMS or PCMS.

22. The method according to claim 21, wherein the PDMS has a molecular weight from 1,000 to 1,700 g/mol.

23. The method according to any one of claims 18 to 22, wherein each gaseous organosilicon compound within the mixture is independently selected from a gaseous silane or a gaseous polycarbosilane.

24. The method according to any one of claims 18 to 23, wherein the mixture of gaseous organosilicon compounds is further processed.

25. The method according to any one of claims 18 to 24, wherein the vessel is purged with an inert gas prior to heating.

26. The method according to claim 25, wherein the inert gas is argon or helium.

27. An apparatus comprising:

a gas panel for connection to a gas outlet of a vessel, the gas panel comprising a valve for controlling a flow of gas from the gas outlet of the vessel through the gas panel; and

a controller for controlling a heater to heat a solid organosilicon compound in the vessel at a predetermined temperature while a predetermined pressure is maintained inside the vessel by the valve, to thereby form a mixture of gaseous organosilicon compounds.
28. The apparatus of claim 27, wherein the controller is implemented at least in part in software for execution by a processing element.

29. The apparatus of claim 27 or 28, wherein the valve is controllable by the controller to maintain the pressure inside the vessel.

30. The apparatus of claim 29, wherein the controller is operable to receive from respective sensors indications of a temperature and a pressure inside the vessel, and to control the heater and the valve based on the temperature and pressure inside the vessel.

31. The apparatus of any one of claims 27 to 30, wherein the controller is further operable to control a flow of gas from the gas outlet of the vessel to the gas panel by controlling an outlet valve of the vessel.

32. The apparatus of any one of claims 27 to 31, wherein the gas panel further comprises a reservoir connected to the valve, wherein the valve comprises a relief valve for allowing the gaseous organosilicon compounds to flow from the vessel to the reservoir when pressure inside the vessel exceeds a maximum pressure.

33. The apparatus of any one of claims 27 to 32, wherein the gas panel further comprises:

   a vent outlet; and

   a vent valve, operatively coupled to the controller, for controlling a flow of gas from the gas outlet of the vessel to the vent outlet.

34. The apparatus of claim 33, wherein the gas panel further comprises a gas line connected to the vent outlet and for connection to a relief valve of the vessel, the gas
line allowing gas to flow from the vessel to the vent outlet when the pressure inside the vessel exceeds a maximum pressure.

35. The apparatus of any one of claims 27 to 34, wherein the gas panel further comprises:

a purge inlet for receiving an inert gas for purging the vessel; and

a purge valve, operatively coupled to the controller, for controlling a flow of the inert gas from the purge inlet to a gas inlet of the vessel.

36. The apparatus of claim 35, wherein the inert gas is argon or helium.

37. The apparatus of claim 35 or 36, wherein the gas panel further comprises:

a vacuum generator, operatively coupled to a vent outlet, to the gas inlet of the vessel, to the gas outlet of the vessel, and to the controller, the controller controlling the vacuum generator to generate a vacuum pressure during a purge cycle.

38. The apparatus of any one of claims 27 to 37, further comprising:

the vessel.

39. The apparatus of claim 38 or 39, wherein the vessel comprises a temperature sensor, operatively coupled to the controller, that senses a temperature inside the vessel.

40. The apparatus of claim 38, wherein the vessel comprises a pressure sensor operatively coupled to the controller, that senses the pressure inside the vessel.
41. The apparatus of any one of claims 27 to 40, further comprising:
   a user interface, operatively coupled to the controller, that receives inputs from a user, provides outputs to a user, or both receives inputs from and provides outputs to a user.

42. The apparatus of claim 41, wherein the user interface comprises a Graphical User Interface (GUI).

43. The apparatus of any one of claims 27 to 42, implemented in a gas cabinet comprising a housing, the gas panel and the controller being mounted to the housing.

44. The apparatus of any one of claims 27 to 42, implemented in a gas cabinet comprising a housing, the gas panel, the controller, and the vessel being mounted to the housing.

45. A method comprising:
   determining a temperature inside a vessel containing a solid organosilicon compound;
   controlling a heater based on the determined temperature to heat the solid organosilicon compound at a predetermined temperature; and
   maintaining a predetermined pressure inside the vessel while the solid is heated, to thereby form a mixture of gaseous organosilicon compounds.

46. The method of claim 45, wherein the maintaining comprises controlling a valve that controls a flow of gas from a gas outlet of the vessel through a gas panel.
47. The method of claim 45 or 46, wherein the determining comprises receiving from a temperature sensor an indication of the temperature inside the vessel.

48. The method of claim 46, further comprising:

5. determining the pressure inside the vessel,

wherein determining the temperature comprises receiving from a temperature sensor an indication of the temperature inside the vessel,

wherein determining the pressure comprises receiving from a pressure sensor an indication of the pressure inside the vessel, and

wherein controlling comprises controlling the heater and the valve based on the temperature and pressure inside the vessel.

49. The method of claim 46, wherein maintaining further comprises controlling a flow of gas from the gas outlet of the vessel to the gas panel by controlling an outlet valve of the vessel.

50. The method of claim 45, wherein maintaining comprises providing a relief valve for allowing the gaseous organosilicon compounds to flow out of the vessel when pressure inside the vessel exceeds a maximum pressure.

51. The method of any one of claims 45 to 50, further comprising:

25 providing for controlled venting the vessel.

52. The method of claim 51, wherein providing for controlled venting comprises:
providing a vent valve connected to a gas outlet of the vessel and to a vent outlet; and

controlling a flow of gas from the gas outlet of the vessel to the vent outlet through the vent valve.

53. The method of claim 51, wherein providing for controlled venting further comprises:

providing a gas line connected to a vent outlet and to a relief valve of the vessel, the gas line allowing gas to flow from the vessel to the vent outlet when the pressure inside the vessel exceeds a maximum pressure.

54. The method of any one of claims 45 to 53, further comprising:

providing for controlled purging of the vessel.

55. The method of claim 54, wherein providing for controlled purging comprises:

providing a purge valve connected to a purge inlet, the purge inlet receiving an inert gas for purging the vessel; and

controlling a flow of the inert gas from the purge inlet to a gas inlet of the vessel.

56. The method of claim 55, wherein providing for controlled purging further comprises:

providing a vacuum generator, operatively coupled to a vent outlet, to the gas inlet of the vessel, to a gas outlet of the vessel; and

controlling the vacuum generator to generate a vacuum pressure during a purge cycle.
57. The method of any one of claims 45 to 56, further comprising at least one of:

receiving control inputs from a user; and

providing outputs to the user.

58. The method of claim 57, wherein the at least one of receiving and providing comprises providing a user interface.

59. The method of claim 58, wherein the user interface comprises a Graphical User Interface (GUI).

60. A computer-readable medium storing instructions which when executed perform the method of any one of claims 45 to 59.
FIG. 1
500 gram load in the Sunbox system, heated at temperature of 422 degree inside the pressure vessel.

A. 4520 min deposition time using AMAT system (48 runs); rough consumption of polymer is 46 gram
B. 328 min of deposition using Trion system (41 runs); rough consumption of polymer is 11 gram
C. Total consumed polymer is about 57 gram
D. The points on the curve represent the deposition.
E. The part of the curve after 350 hrs need to be verified after the weekend, the pressure may go up again.

FIG. 2
FIG. 3

GAS SOURCE

Heated pressure vessel

Gas panel, containing regulators, valves, gauges to condition the gas for use by PECVD

PECVD or CVD

Mass flow controllers & mixing manifold

DEPOSITION TOOL
FIG. 4
INTERNATIONAL SEARCH REPORT

International application No.
PCT/CA2008/001504

A. CLASSIFICATION OF SUBJECT MATTER
IPC: C07F 7/08 (2006.01), BOIF 3/06 (2006.01), BOIJ 19/14 (2006.01), BOIJ 4/02 (2006.01), BOIJ 8/02 (2006.01), C07F 7/10 (2006.01), G05B 19/042 (2006.01), G05D 23/19 (2006.01), G05D 7/06 (2006.01)

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC(2006.01): C07F7/08, BOIF 3/06, BOIJ 19/14, BOIJ 4/02, BOIJ 8/02, C07F 7/10, G05B 19/042, G05D 23/19, G05D 7/06

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic database(s) consulted during the international search (name of database(s) and, where practicable, search terms used)
Delphion (all database), Canadian Patents Database, Scopus, STN

Keywords searched: silane, organosilane, organosilicon, silicon, pyrolysis, sublimation, vaporization.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>CA 2 486 867 C (SCARLETE et al.) 04 December 2003 (04-12-2003) <em>see page 4, lines 17-20 and page 7, lines 6-8</em></td>
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<td>WO 2008/104059 A1 (AWAD et al.) 04 September 2008 (04-09-2008) <em>see page 10, lines 4-10</em></td>
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Further documents are listed in the continuation of Box C.

[X] See patent family annex.

Date of the actual completion of the international search 24 October 2008 (24-10-2008)

Date of mailing of the international search report 18 November 2008 (18-11-2008)

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