DYNAMICS OF PULSED DC DISCHARGES USED FOR PACVD - EFFECT OF BIPOLAR VOLTAGE PULSES

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Abstract

In recent years the PACVD (plasma-assisted chemical vapour deposition) technique using pulsed d.c. discharges has attracted much attention, because this technique allows to increase the reactor size easily. To produce homogeneous coatings it is necessary to obtain a temporally and spatially uniform plasma. In unipolar operation mode of the power supply the spreading of the discharge is slow. We used a fast CCD camera to record the light emission of the discharge. Therefore, we were able to observe the spreading of the discharge during one pulse. The current/voltage characteristic of the pulses was monitored with a digital oscilloscope. In the present work we studied the effect of using bipolar pulses. We investigated the effect of the duration of the additional positive pulses as well as the pulse-off times between the pulses. Our experiments show an improvement of the ignition and spreading behaviour of the discharge by the use of bipolar voltage pulses.

1. Introduction

Nowadays pulsed direct current (d.c.) glow discharges are commonly used in plasma-assisted chemical-vapour deposition (PACVD) systems designed for the deposition of hard coatings. Experience shows that the dynamics of the discharge has a significant influence on the homogeneity of the coatings, especially in larger PACVD systems. Studies of the development of plasmas relevant for the production of titanium nitride (TiN) coatings using titanium tetrachloride (TiCl₄) as a feed stock gas have shown that under certain loading conditions the spreading of the discharge during the pulse is too slow [1-5]. The discharge ignites at the beginning of each pulse at one spot and spreads from there across the reactor [5]. The spreading of the discharge as expressed in an increase of the coverage of the substrate plate (cathode) with a negative glow is accompanied with an increase of the discharge current. The spreading is completed when the whole substrate is covered with a negative glow of constant luminosity and the discharge current reaches a saturation value. Without TiCl₄, an immediate spreading of the discharge after ignition was observed, as expressed in a total and time-independent coverage of the substrate plate with a negative glow. In this case the discharge current reaches the saturation value immediately after ignition and remains constant during the rest of the pulse.
Recently, we demonstrated that the ignition and spreading behaviour can be very much improved by the superposition of short high voltage pulses at the beginning of each conventional pulse [6, 7].

In the present work we investigate another possibility to get a better performance of the ignition and spreading of the discharge, the bipolar operation of the voltage power supply.

2. Experimental details

Our experimental setup shown in Fig. 1 consists of a commercially available plasma system manufactured by Rübig GmbH [8], a fast video system and a voltage and a current probe with digitizing oscilloscope. As similar hot wall reactors are described elsewhere [5, 9], no further details are shown in Fig. 1, only essential features will be presented here.

![Diagram of experimental setup](image)

**Fig. 1.** Schematic illustration of the experiment setup with reactor, pulsed d.c. power supply, oscilloscope and video system.

The deposition chamber with an inner diameter of 50 cm and a height of 90 cm has five viewpoints which permit the entire volume to be seen from outside. The discharge is maintained by a pulsed d.c. power supply. In unipolar operation mode of the power supply, the pulsed d.c. voltage is applied to the substrate plate which acts as cathode. The walls of the reactor chamber serve as anode. The power supply can also be run in bipolar operation mode. During the positive pulses the substrate plate is the anode and the vessel is the cathode. The pulse duration of the negative and the positive pulse are separately programmable. However, the pulse pauses between the positive and the negative pulses cannot be varied independently. As a substrate a steel cylinder placed in the middle of the substrate plate was used.

The measurement of the discharge current is performed using an active current probe (Fluke 80i-110s AC/DC current probe). Discharge current is monitored by a digitizing oscilloscope (Fluke 105B Scopemeter SeriesII).

Experiments were performed with a gas mixture consisting of hydrogen (H₂), nitrogen (N₂), argon (Ar) and titanium tetrachloride (TiCl₄). The parameters were choosen for typical titanium nitride (TiN) deposition conditions as listed in Table 1.
Table 1. Process parameters.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure</td>
<td>3 mbar</td>
</tr>
<tr>
<td>Substrate temperature</td>
<td>450 °C</td>
</tr>
<tr>
<td>Wall temperature</td>
<td>500 °C</td>
</tr>
<tr>
<td>Discharge voltage</td>
<td>450 V</td>
</tr>
<tr>
<td>Pulse-on time neg. pulse</td>
<td>200 µs</td>
</tr>
<tr>
<td>Pulse-off time</td>
<td>100 - 400 µs</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>200 slh</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>27 slh</td>
</tr>
<tr>
<td>Argon</td>
<td>8 slh</td>
</tr>
<tr>
<td>Titanium tetrachloride</td>
<td>0.6 slh</td>
</tr>
<tr>
<td>Pulse-on time pos. pulse</td>
<td>10 - 200 µs</td>
</tr>
</tbody>
</table>

3. Results and discussion

Former investigations revealed large differences between plasmas with and without an electronegative species, e.g. TiCl₄ [5]. In unipolar operation mode of the power supply, an immediate spreading of the discharge after ignition was observed in the absence of TiCl₄. In the presence of TiCl₄, the spreading of the discharge along the cathode (substrate surface) occurs with a certain delay. The reason is the limited conductivity of the plasma at the beginning of each pulse. This yields a non-uniform plasma power density in front of the substrates and therefore a spatially varying exposure time of the surface to the plasma.

In bipolar operation mode measurements with positive pulses of 10, 25, 50, 100 and 200 µs duration and a pulse-off time of 100, 200 and 400 µs, have been performed.

In Fig. 2 the very high current during positive pulses is not shown in order to get an uniform axis scale and a good view on the development of the discharge current during the negative pulse. For reference Fig. 2 (a) shows the development of the discharge current in the unipolar mode (only negative pulses). Fig 2 (b) to (f) show the discharge current wave forms for different duration of the positive pulse. It can be seen that with increasing duration of the positive pulse the current wave form during the negative pulse improves, as expressed in a faster increase of the discharge current after ignition.

Fig. 3 shows the discharge current wave forms for different pulse-off times. With increasing pulse pause between the pulses only a small degradation of the negative pulse can be observed.

The video system observations show that for a duration of the positive pulses of 50 µs or more, the discharge in the negative pulses ignites faster than in the unipolar operation mode [5].

A possible explanation of the beneficial effect of the bipolar pulses might be as follows. During the positive voltage pulses the ratio of the surfaces of cathode to anode is inverted compared to the case of the negative voltage pulse. Because of the large surface of the cathode during a positive voltage pulse the discharge current and accordingly the plasma power density is significantly higher. So one can assume that at the end of the pulse-off time more charge carriers remain and hence, the density of charge carriers is high enough for a better ignition of the plasma at the beginning of the negative pulse. Therefore, the duration of partial coverage of the substrate is lower and a greater uniformity of the plasma can be maintained during the negative pulse.
Fig. 2. Discharge current waveform in a plasma containing TiCl₄ for unipolar mode (a) and bipolar mode with different duration of positive pulses: (b) 10 µs, (c) 25 µs, (d) 50 µs, (e) 100 µs and (f) 200 µs.
The negative voltage pulses are showing positive current values.
Fig. 3: Discharge current wave forms in a plasma containing TiCl$_4$ for different pulse-off times between pulses.
(a) 100 µs
(b) 200 µs
(c) 400 µs
The negative voltage pulses are showing positive current values.

4. Conclusions
Our experiments clearly demonstrated that the uniformity of the discharge is improved by running the power supply in bipolar operation mode. Apparently with positive pulses an increase of the conductivity of the plasma at the beginning of each negative pulse can be obtained. Thus the duration of a partial coverage of the substrate surface which leads to a spatial inhomogeneous deposition of the coating is shorter.
Acknowledgements

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References