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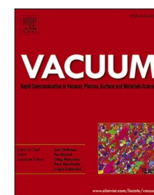

Short communication

Vacuum technical investigation of a novel reactor for the continuous coating of flexible web material

Philipp Alizadeh [*], Rainer Dahlmann

Institute for Plastics Processing (IKV) in Industry and Craft at RWTH Aachen University, 52074, Aachen, Germany

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ABSTRACT

This short communication presents the concept and validation of a vacuum reactor designed for Plasma-Enhanced Chemical Vapor Deposition (PECVD) inline coating of flexible web material. Three airlocks are utilized in order to ensure uninterrupted transportation of the film in and out of a consistently evacuated reactor. Four distinct pressure areas are monitored by respective sensors. Pressure differentials were achieved, which are a prerequisite for minimizing plasma excitation and coating deposition outside the process envelope.

The study highlights leakage flow dynamics, with approximately 70 % attributed to Pumping Unit I, 29 % to Pumping Unit II, and only 1 % to the coating module. The reactor's configuration effectively reduces pressure equalization between zones by "throttled short-circuits". Conductance values for the airlocks and process envelope are discussed, with geometric adjustments recommended for improved flow choking. A base pressure (without process gases) of 4.9 Pa in the process envelope was observed, enabling PECVD at pressures greater than 15 Pa.

1. Introduction

PECVD technology has rapidly established itself in the field of hollow containers in recent years. PET beverage bottles can now be coated with a throughput of several tens of thousands of bottles per hour [1]. However, the technology is much less widely used in the flexible packaging sector. The reasons for this are primarily process-related, as standard systems on the market only work discontinuously. A discontinuous work step in the continuous process chain of film processing means higher costs compared to hollow body coating. Additionally, the mitigation of parasitic coatings is more complex since the plasma is not enveloped by the substrate, unlike in hollow body coating.

This poses a significant challenge for the desired transformation to a circular economy. Flexible packaging accounted for the largest market share in the global packaging sector in 2019, ahead of PET bottles, at around 28 % [2]. Significant growth is expected in the barrier packaging sector as the requirements for recyclability and product protection continue to increase. Consequently, the establishment of PECVD technology to implement the EU's sustainability goals is of great interest, particularly in the area of flexible packaging.

The aim of the work presented here is to investigate the vacuum properties of a new type of coating reactor, which was built to enable the continuous coating of flexible plastic substrates using the PECVD process [3]. In contrast to closed vacuum chambers, the vacuum must be generated using sequentially arranged airlocks that ensure a pressure reduction from atmospheric pressure to the required pressure level of the PECVD process. Therefore, the validation of the vacuum concept is the first milestone in the commissioning of the reactor and the necessary prerequisite for maintaining the PECVD process.

1.1. Reactor design

Based on the coating concept for hollow bodies, the plasma is enclosed within a process envelope. This is achieved by the envelope having an increased pressure (20–40 Pa) compared to the surrounding volume (<10 Pa). According to the ignition conditions for microwave plasmas, analogous to Paschen's law, this results in preferred ignition conditions within the envelope [4,5]. This is accompanied by significantly increased coating deposition rates and greatly reduced parasitic coating compared to conventional roll-to-roll systems.

Furthermore, continuous processing of the film is ensured by the use of three airlocks. The process chamber remains in an evacuated state while flexible substrates can be continuously transported in and out of the chamber through slits. The slits are designed to tolerate a headspace

* Corresponding author. IKV, 52074, Aachen, Germany.
E-mail address: publications@ikv.rwth-aachen.de (P. Alizadeh).

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for contactless conveying of the substrate, so as not to damage an applied coating. The tolerated leakage flows thus represent a stark contrast to conventional, discontinuous vacuum systems. The vacuum condition of the prototype is examined in more detail below.

The reactor has three airlocks and a process chamber, as well as a process envelope. The plastic film to be conveyed has a width of 400 mm and a thickness of 12 µm. Correspondingly, the gaps in the process envelope have a width of 402 mm, a height of 0.1 mm, and a length of 90 mm. A technical drawing is shown in Fig. 1.

In Fig. 1 a sectional view of the reactor modules from above is displayed. The film is positioned vertically in the viewing direction and is transported twice through the process envelope with the aid of a guide roller. In contrast to the sketch, Airlock I is equipped with a fixed cross-section that, for reasons of dimensional accuracy, consists exclusively of aluminum components that cannot be adjusted. The green lines indicate the slots for the film feed-through.

Regarding the process envelope, the film is fed in and out via slits on the process gas pipes. Analogue to the airlocks, the slots form a flow channel with a length of approx. 10.5 mm, a height of approx. 0.14 mm and a width of 404 mm. The walls of the envelope are curved to favor full contact of the film and to avoid air pockets between the film and the wall. This results in the channels having a decreasing height towards the inside of the envelope. Due to the pressure gradient between the envelope and the surrounding volume, this results in a diffuser that favors turbulence in order to reduce the conductance of the slits through turbulent flow behavior. The dimensions (l x w x h) of the envelope are approx. 424 mm x approx. 32.7 mm (average wall distance) x 404 mm. The resulting volume is approx. 5.6 L.

2. Results and discussion

The vacuum reactor concept is shown in Fig. 2. The technical vacuum is created using three pumping units with nominal suction capacities of 265 m^3/h, 1000 m^3/h and 2000 m^3/h as well as a turbomolecular pump (TMP) with a suction capacity of 1600 l/s. The pumping units are arranged according to the vacuum requirements with an increasing capacity in the direction of the coating chamber.

The process envelope, which is designed to contain the plasma during PECVD coating, is directly connected to pumping unit III. The surrounding volume of the process chamber is evacuated by the TMP. The pressure is kept lower in the surrounding volume compared to the process envelope in order to limit plasma excitation and coating deposition outside of the process envelope. The airlock modules also have separate pressures, resulting in four separate pressure areas. Each of those areas is equipped with a respective pressure sensor. The areas correspond to the first (p_1) and second (p_2) airlock modules, the process chamber (p_3) and the process envelope (p_4).

The most important leakage flows are represented by the q_{pV} flows through the airlocks ($q_{pV_a\rightarrow 1}$, $q_{pv_1\rightarrow 2}$, $q_{pv_2\rightarrow 3}$), into the process envelope ($q_{pv_3\rightarrow 4}$) and into the pumping units ($q_{pv_1\rightarrow I}$, $q_{pv_2\rightarrow II}$, $q_{pv_4\rightarrow III}$, $q_{pv_3\rightarrow TMP}$). The inlet pressure of the TMP is generated with the aid of pumping unit III, which also evacuates the process envelope. Pirani pressure sensors (TPR 280, Pfeiffer Vacuum) with a measuring range of $5 * 10^{-2}$ to 100,000 Pa were used to determine the pressures p_1 and p_2. Capacitive pressure sensors (CMR 363, Pfeiffer Vacuum) with a measuring range of 10^{-1} to 1100 Pa were used to determine the pressures p_3 and p_4.

With the help of the measured basis pressures p in the evacuated state of the system without the feed of process gases and under consideration of the suction capacities of the pumps S, an initial approximation can be found for the mass flows conveyed by the pumps (Eq. (1)) [6]. For this purpose, the simplifying assumption is made that no pipe friction losses occur and consequently the suction capacity for the measured pressure can be read from the underlying curves. The suction capacity curves can be found in the appendix.

$$q_{pV} = S\cdot p \quad \text{Eq. 1}$$

The resulting values can be found in Table 1.

The total volume flow of the leakage into the reactor can be estimated at approx. 125 slm. Pumping unit I accounts for approx. 70 % and pumping unit II for a further approx. 29 %. Consequently, only approx. 1 % of the total leakage flow is attributable to the coating module including the process envelope.

As both the TMP and the process envelope are evacuated by pumping unit III, the configuration can be interpreted as a “throttled” short circuit. The pressure equalization between pressure range III and IV is throttled by the conductance values of the film slots of the process envelope, so that $p_4 > p_3$ applies. With regard to the total leakage flow, the q_{pV} flow of the TMP is not added to the volume flow balance due to this short circuit.

The following equation set (Eqs. (2)–(5)) can be established for the q_{pV} flows of the airlock elements and the process envelope:

$$\text{Airlock I: } q_{pV_a\rightarrow 1} = q_{pV_tot} = q_{pV_1\rightarrow 2} + q_{pV_1\rightarrow I} \quad \text{Eq. 2}$$

$$\text{Airlock II: } q_{pV_1\rightarrow 2} = q_{pV_2\rightarrow 3} + q_{pV_2\rightarrow II} \quad \text{Eq. 3}$$

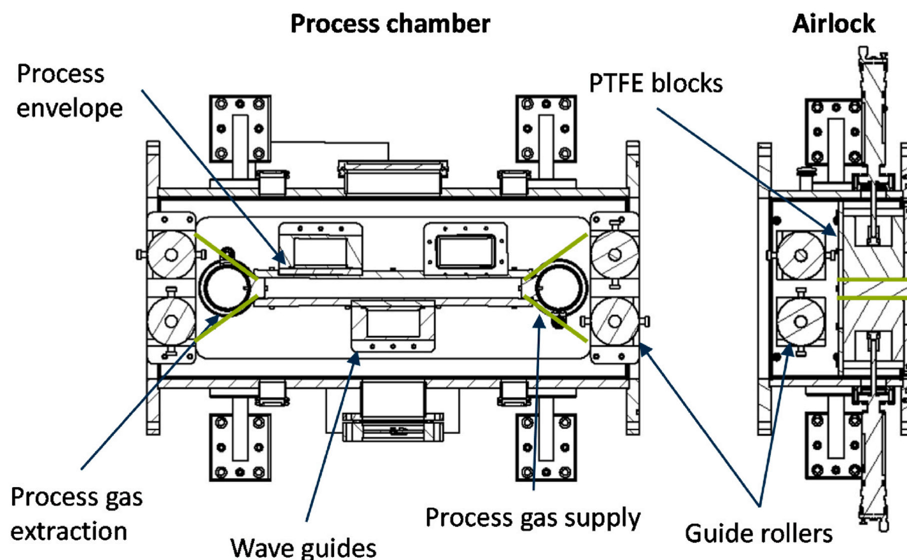

Fig. 1. Technical drawing of the reactor prototype.

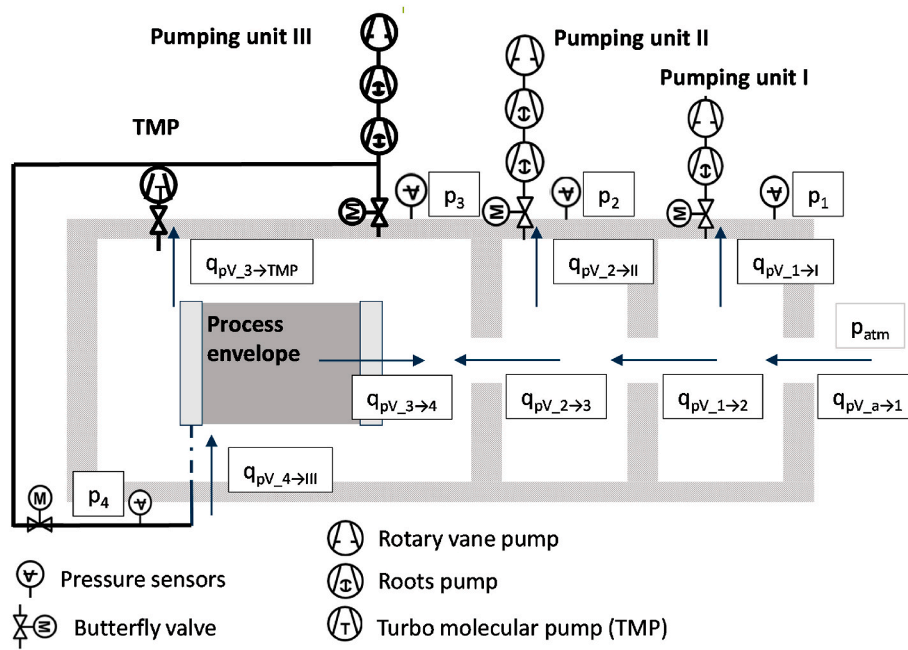

Fig. 2. Vacuum reactor concept.

Table 1
Resulting pressures, q_{pV} flows and suction capacities of the pumping units.

Area	Atmosphere	Pressure area I	Pressure area II	Pressure area III	Pressure area IV	Total
Subscript	atm, a	1, 1 → I	2, 2 → II	3, 3 → TMP	4, 4 → III	tot
S [m^3/h]	–	150	900	4320	1500	–
p [Pa]	101,320	3551	242	1.48	4.87	–
q_{pV} [sccm]	–	87,599	35,860	(1051)	1201	124,660
Percentage	–	70.27	28.77	–	0.96	100

Airlock III: $q_{pV_2\to 3} = q_{pV_4\to III}$ Eq. 4

Process envelope: $q_{pV_3\to 4} = q_{pV_3\to TMP} - q_{pV_4\to III}$ Eq. 5

As a result of external friction between the gas particles and the wall surface and internal friction between the gas particles, flow resistance occurs, which manifests itself in pressure differences. Conductance C is determined in order to assess the flow behavior. Using the q_{pV} flows of the airlocks and the process envelope as well as the pressures, C can be calculated according to equation Eq. (5) [6]:

$$q_{pV} = C \cdot \Delta p = C \cdot (p_1 - p_2)$$ Eq. 6

The results are displayed in Table 2.

The conductance of a single slit for airlock I is approx. 3.88 m^3/h at an average pressure of approx. 52.44 Pa. As Airlocks II and III are based on the concept of movable PTFE blocks, as opposed to Airlock I, significantly higher conductance values can be observed. These are due to the limited dimensional accuracy in the production of the PTFE components and the fitting dimensions that are difficult to realize as a result. Consequently, in addition to leakage via the pre-set airlock gap of 0.1 mm, additional leakage flows via unwanted gaps in the airlock housing are a probable cause.

Another significantly higher conductance value is observed for the process envelope, which occurs under significantly lower prevailing pressures ($p_{m,3\to 4} = 0.18$ Pa). This can be explained in particular by the cross-sectional geometry of the film slit. In contrast to the airlocks, the gap height of 0.1 mm only occurs at certain points. For comparison, the airlocks have a gap length (l) of approx. 90 mm. To promote the desired effect of vacuum blocking or laminar-turbulent transition, longer channels are advantageous [7]. In addition, the walls of the process envelope are also made of PTFE, which affects the dimensional accuracy.

It should be noted that the leakage flow of the process envelope ($q_{pV_3\to 4}$) has a negative value, as the flow escapes from the envelope into the ambient volume. Although the value of 150 sccm is very small relative to the other flows, in absolute terms it is of a relevant order of magnitude with regard to an employment of process gas flows. Geometric adjustments to the duct cross-section to reduce the conductance values and to promote blocking would be adequate improvement measures. With regard to any contamination that could be introduced into the process due to the leakage flow, the pressure gradient between the shell and the coating module should be viewed positively. As the area of

Table 2
Determined q_{pV} flows and conductance values of the airlocks and the process envelope.

Element	Subscript	q_{pV}-flow [sccm]	Conductance C [m^3/h]	Number of slits [–]	Conductance C of single slit [m^3/h]
Airlock I	a → 1	124,660	7.76	2	3.88
Airlock II	1 → 2	37,062	68.14	2	34.07
Airlock III	2 → 3	1201	30.35	2	15.17
Process envelope	3 → 4	−150	268.98	4	67.24

lowest pressure is Pressure area III, any impairment of the process due to contaminants introduced via the airlocks can be ruled out with a high degree of certainty. In order to increase the purity of the process envelope and consequently the coatings to be deposited, the envelope can be flushed with high-purity inert gas using the gas supply system before coating.

The validation of the vacuum concept is rated as a partial success. Particularly with regard to the functionality of the airlock systems, adjustability must be dispensed with due to the achievable manufacturing tolerances of the PTFE components. However, using a fixed channel in Airlock I, a base pressure of 4.9 Pa was achieved in the process envelope, which allows the PECVD process to be carried out at a process pressure of 15–40 Pa without restriction. Further research will be conducted in order assess a PECVD barrier coating on PET film in the reactor prototype presented here.

CRediT authorship contribution statement

Philipp Alizadeh: Writing – original draft, Validation, Formal analysis, Data curation, Conceptualization. **Rainer Dahlmann:** Supervision, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare no financial or commercial conflict of interest.

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Data availability

Data will be made available on request.

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