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Short communication

Upscaling plasma deposition: The influence of technological parameters

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The technological parameters that control plasma processes follow several scaling rules. Hence, we must adapt inlet gas flow rate, pressure, input power and excitation frequency to the new reactor dimensions. High rates of film deposition or etching; plasma homogeneity, and reproducibility are the main objectives in any attempt to upscaling. This paper provides guidelines to transfer plasma deposition processes from small pilot reactors to large plants for industrial production.

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1. Introduction

Low-pressure and low-temperature plasmas show a wide range of applications in industry, from the deposition of thin films and coatings to surface modification or etching [1–3]. Nowadays, plasma-enhanced chemical vapour deposition (PECVD) and magnetron sputtering are the most popular deposition techniques for thin film production at industrial scale. However, early stages of research of new materials and surface processes are always performed in lab-size reactors. The already investigated processes in laboratories must be then transferred to large scale vessels. The goals consist in achieving uniform large area plasmas, increasing throughput and deposition rate to reduce production cost, and treating substrates with complex geometry. Currently, upscaling of plasma processes remains as a non-trivial question and arises as an important time-consuming step for industry in order to optimize coating and surface treatment in large reactors.

PECVD coating technique consists of film growth from chemical reactions undergone by gas precursors in glow discharge regime [4]. Some fundamental processes are defragmentation of the precursors, reactions of oxidation-reduction, hydrolysis, polymerization and transport phenomena. The main steps are depicted in the schematic representation of a PECVD system in Fig. 1 [2]. Such processes can be controlled through the experimental setup, where the shape and size of the reactor play a critical role. Comparative studies of inductively coupled (ICP) and capacitively coupled (CCP) plasmas are currently performed to optimize their application in the coating industry [5].

The effort of an elevated number of researchers has been devoted to the development of plasma technology at large scale. The sectors more interested in this field are mainly the automotive industry [6,7], the fabrication of machinery components and tools [8], optical coatings [9], polymers [10–12], as well as solar cell and flat panel displays [13–15]. As a result, an immense network of know-how concerning the upscaling of plasma processes has been generated, which is most of the times protected by patents. Different types of plasma chambers, basically batch and continuous (roll-to-roll) coaters, have been developed to increase throughput [13]. Innovative designs of plasma sources like linearly extended sources [16,17] and resonant network antennas [18], as well as the introduction of moving parts in the reactor setup, have improved substantially the homogeneity of large area plasma processes. A discussion concerning these aspects, which are of great importance to perform a successful upscaling, is out of the scope of this article.

The following sections focus on the strategies to adjust the technological (macroscopic) parameters of cold plasmas to the reactor dimensions. Since the preservation of discharge parameters is crucial to scale up plasma processes, several scaling rules must be considered. In principle, the following magnitudes should be constant [19]: partial pressures, distance between electrodes, residence time, power density, impedance, excitation frequency and substrate temperature. Their interrelationship can be expressed by means of similarity parameters, which constitute excellent tools to compare plasma processes held in different reactors. Table 1 summarizes the contents of the article, and relates the basic processes shown in Fig. 1 to the technological parameters of deposition. Although this paper focuses on plasma deposition, similar scaling rules are applicable to plasma etching processes [20].

Very large area plasma processing is a key topic in the current roadmap of low-temperature plasma technology [21]. In that context, this work constitutes a guide to upscaling of plasma processes from the point of view of the external parameters controlling the discharges. The discussion is illustrated with examples of a-C:H film deposition carried out at the Fraunhofer IST.

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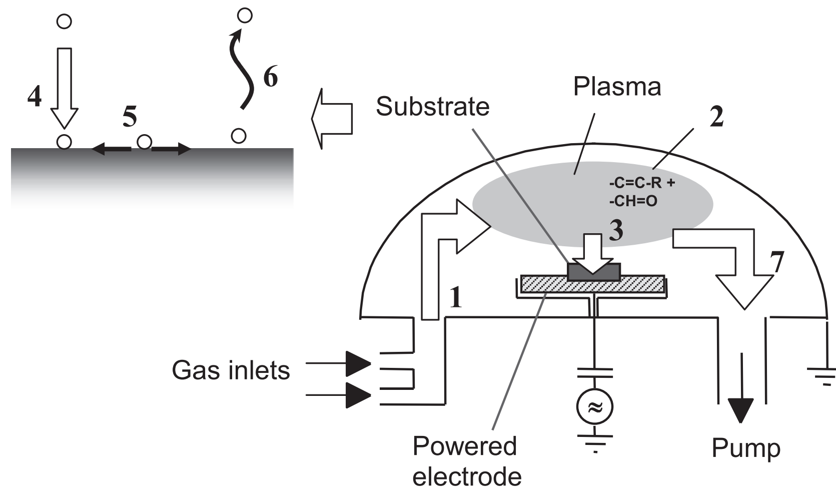

Fig. 1. Layout of a typical PECVD reactor in capacitive configuration showing the basic steps. 1: Transport of the reactants from the gas inlets to the reaction zone. 2: Reactions in vapour phase that form the gas precursors of the film and by-products. 3: Transport of the reactants and their products from the gas phase to the substrate; positive ion bombardment due to plasma sheath. 4: Adsorption of these species on the substrate surface. 5: Surface diffusion, chemical reactions and incorporation of these species on different growth sites; processes assisted by ion bombardment. 6: Desorption of the volatile by-products of surface reactions. 7: Transport of the by-products away from the reaction zone.

2. Pressure and flow dynamics

Partial gas pressure should be constant in plasma upscaling. This is a crucial parameter for the plasma chemistry because reaction rates are very sensitive to pressure. Also, film properties are generally dependent of the incident particle flux or collision rate onto the substrate, j_i, which is proportional to the pressure exerted by the corresponding specie, p_i [22]:

$$j_i = \frac{p_i}{\sqrt{2\pi m_i k_B T}} \quad (1)$$

where m_i is the mass of the i-specie, κ_B is the Boltzmann's constant and T is the gas temperature. Generally, the growth rate is proportional to the collision rate and also to the sticking coefficient of the depositing species on the substrate surface.

Gas pressure is usually measured with vacuum gauges. It is important to consider the pressure gradients between the vessel and the measurement point. Indeed, the effects of thermal transpiration must be taken into account, in particular they can be significant in large plants [23,24]. In the case of flowing systems, pressure does not scale linearly with gas flow rate [25]. Indeed, the inlet flow rate, F_i, and pressure of an individual gas are related through this empirical expression:

$$F_i = a \cdot p_i^{\,b} \quad (2)$$

where a and b are constants. From this relation, and due to the possible interactions among gases, it is evident that the law of addition of partial pressures is not valid in plasma processes. Therefore, mass spectrometry (with eventually differential pumping) is required to perform accurate measurements of partial pressures when the plasma is on. Even mass spectrometry is not straightforward in this case, since highly reactive particles might not be detected due their fast depletion in the plasma chamber.

2.1. Reactor geometry

The interelectrode gap should be kept constant if possible, since the mere consideration of the Paschen law does not guarantee that the internal plasma parameters remain constant upon variations of the separation between electrodes [19]. However, industrial chambers might present larger gaps than small reactors do, and then, the working pressure, p, scales according to $p \cdot d = ct$. The following product should be constant and can be considered as a generalization of the Paschen law at high-frequency discharges [26]:

$$p \cdot \Lambda = ct. \quad (3)$$

Λ is the characteristic diffusion length of the vessel, and it is obtained from solving the ambipolar diffusion equation in the plasma. Its value depends on the chamber geometry and dimensions, as shown in Fig. 2 [27].

The product in Eq. (3) determines fundamental plasma parameters in a first approximation, such as electron temperature and electron

Table 1
Links between technological parameters (upscaling constants) and related microscopic processes depicted in Fig. 1. The corresponding issues derived from setting upscaling constants, proposed solutions, monitoring techniques and similarity parameters are also indicated.

Parameters	Processes Fig. 1	Issues	Solutions	Monitoring	Similarity parameters
Pressure	1,2,3,7	Interelectrode gap	Paschen law	Vacuum gauge	Kn
		Pressure gradients	Gas inlet distribution	QMS	s/λ_ι
Residence time	2,7	Inactive zones	Stronger pumps	Pressure evolution	Da
		Dust + contamination	Gas inlet distribution	OES	Pe
Power density	2,3,5	Plasma edges	Sample arrangement	Langmuir + IV probe	W/F
(V_{sh}, I/A)		Arcing	Pulsed power	RFEA	n_e/F
Frequency	2,3,5	Standing waves	Lower frequency	Oscilloscope	L/λ
		Plasma position	Electrode shaping		
		Ion bombardment	Dual frequency		
Substrate temperature	4,5,6	Non-uniformity	Distributed heaters	Pyrometer	SZD
			Cooling down	Thermocouple	

Acronyms and variables: QMS: quadrupole mass spectrometry; OES: optical emission spectroscopy; RFEA: retarding field energy analyzer; SZD: structure zone diagram; V_{sh}: sheath voltage; I: electric current; A: electrode area; Kn: Knudsen number; s: sheath thickness; λ_i: ion mean free path; Da: Damköhler number; Pe: Péclet number; W: input power; F: gas flow rate, n_e: electron density; L: characteristic reactor size; λ: excitation wavelength.

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