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Plasma surface interaction in integrated circuit production and biomedical applications♣

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A review is presented of different techniques whereby non-equilibrium plasmas are optimized and adjusted to suit the needs of modern technologies. The reactive nature and high energy of ions allow plasmas to etch profiles or change the structure of the surface, leading to functional applications ranging from micro (nano)-electronics to biomedical applications. Non-equilibrium plasmas provide the means to develop a number of new technologies, by fine tuning the properties of fluxes of particles hitting the surface, and fundamental studies of plasmas provide the means to realize such optimizations of plasma sources.

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1. Introduction

Numerous novel technologies spring every day from the physics of solids, and in many cases such technologies are closely related to the application of plasma physics. Standard techniques such as implantation, plasma assisted chemical vapour deposition and sputtering will not be discussed here, we shall focused on the application of non equilibrium plasmas in further advances in nano-electronics [1], in nano-structuring and in other aspects of forthcoming nanotechnologies where plasmas may be used for massively parallel self-organized nano-structuring and in biomedical applications.

In this paper, we review recent studies of plasma surface interactions motivated by two possible applications of plasmas in the treatment of solid state surfaces, and the development of the related novel technologies.

In plasma etching of nanostructures in dielectrics for integrated circuit production, where it is necessary to control charging of the nanostructures to avoid damage, to couple plasma models and models of the development of surface profile due to plasma etching in order to optimize plasma processing technology and to control roughness if one wants a plasma to have the same role in nanotechnologies as it has in micro electronics and in current nano-electronics. The treatment of SiO_2 and organic dielectrics is discussed, and optimal plasmas for etching will be evaluated.

In the interaction of plasmas with organic materials, including living tissue, it is important to establish the basic interaction mechanisms, before one may proceed to optimize further the applications such as the treatment of textiles or biomedical applications. Plasma treatment of organic materials and living tissue is superficial, and leaves the bulk properties unchanged, but it may change the surface properties significantly and may add new functionalities that were not available before.

A common front between the two topics mentioned here is that of the nanostructuring of surfaces, which is one of the main foundations of future mass produced nanotechnologies and their products. Control of plasma induced surface roughness is essential in such applications. On the other hand, the critical issues from the point of view of plasma physics are the control and optimization of multi-frequency low pressure capacitively coupled and inductively coupled plasmas, and the development of atmospheric non-equilibrium plasma sources. Micro discharges are perhaps the most promising category amongst the atmospheric pressure discharges, but require further fundamental studies.

We discuss here some topical research issues that have bearings on future technologies and that have been associated with the activities of one group of authors.

2. Plasma etching in nano-electronics

Plasma etching is one of the basic steps used in semiconductor processing for the fabrication of electronic devices, since anisotropic etching allows high-fidelity pattern transfer and the processing can be performed with relatively low heating of the surface and structures [1].

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Capacitively Coupled Plasmas (CCP) are the most commonly used plasma sources to provide etching of dielectrics, which is the most important and frequent step in the production of integrated circuits (IC) [1]. Non-equilibrium (low temperature) Inductively Coupled Plasmas (ICP) have been used mostly in the plasma etching of polysilicon, metal and in the plasma ashing of the photoresist, three of the key steps in ultra large scale integrated (ULSI) technologies.

The key issues for further improvement of the plasma processing tools are still numerous, and some will be discussed in this section.

2.1 Two frequency operation of rf plasma reactors, and control of the ion energy

At the moment, CCP tools dominate the plasma etching market. In particular, pulsed two and even three frequency operation is regarded as a topical research issue. A few years ago, a question of how to achieve functional separation between the plasma driving source and the source for accelerating the ions was raised, and some results were offered on the basis of experiments [2]. The first experiments revealed that much higher frequencies than the standard 13.56 MHz must be used to produce the plasma, in order to achieve functional separation for CCPs. The situation is less critical for ICPs [3] where efficient functional separation of a plasma and biasing sources for ICP may be achieved by using the standard industrial frequency of 13.56 MHz, and biasing at around 1 MHz [3].

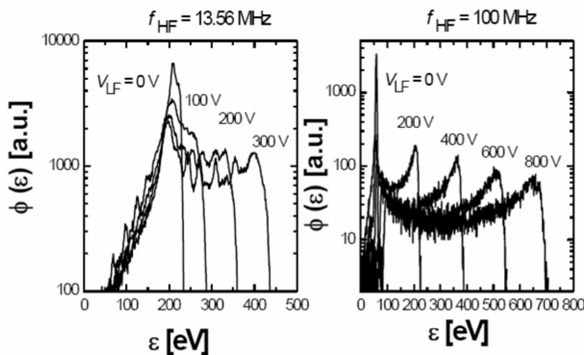

Fig.1. Energy distribution functions of ions for a) 13.56 MHz and for b) 100 MHz high frequency voltages. The results were obtained for different amplitudes of the low frequency "biasing" voltage [4].

Understanding of the functional separation may be achieved by employing numerical simulations [4]. In our calculation for CF_4, based on the Particle in Cell (PIC) simulation code with Monte Carlo collisions [4], we were able to determine the ion energy distribution functions and their dependence on a low frequency biasing voltage, as shown in Fig 1. For the higher "plasma forming" frequency of 100 MHz, there are two distinct peaks in the Ion Energy Distribution Function (IEDF), at a low and a maximum available energy. The IEDF for the energies between two peaks has a minimum, and thus one may expect that the flux of high energy ions will be controlled very effectively by the biasing voltage.

2.2. Charging of nano-structures in dielectric materials, during plasma etching

As etching of dielectrics is the most frequent and demending step in the production of integrated circuits, the more complex circuits of future generations (65 nm, 45 nm and smaller) will require even larger numbers of interconnect layers, and transitions from the current 9 layers to near 20 will be carried out soon. This will pose more and more difficult requirements on the etching of the dielectrics. On the other hand, charging of the dielectrics affects the efficiency of etching and the profile and thus for high aspect ratios, the structures (depth to width ratio). In this case, the etching rate is very much reduced. In addition to the aspect ratio dependent etching rate, there are other associated issues, such as microloading, etchstop, notching, and many more. Most of these problems have been strongly associated with charging of the bottom of the nanostructures [5]. As the density of deposited charges increases, so does the potential and eventually (at the aspect ratio of 7 [5]) it will become equal to the potential that corresponds to the energy of the ions. At this point, no ions will reach the surface and etching will stop. Electrons leave the plasma with relatively high energies, and thus have very anisotropic distribution as they slow down in the sheath, so they are deposited mainly at the sidewalls and cannot reach the bottom, which prevents other electrons from reaching the bottom of the structure and neutralizing the charge. Negative ions from the plasma have low energies, so normally they cannot cross the sheath which was created to slow down the much more energetic electrons.

Experiments with pulsed plasmas, where the plasma sustaining source is pulsed while the biasing voltage remains constant, gave excellent improvements regarding the defects that may be associated with charging. Makabe and co-workers [6] have been able to show that in the afterglow phase, a double layer forms that may push negative ions into the nano-trench and will improve the neutralization of positive charges. In addition to etching defects, the very large potentials on the dielectric of the transistors may cause breakdown of the very thin dielectrics of the MOSFET during manufacturing.

A general solution to the problem of charging would be to use fast neutrals instead of ions. Such fast neutrals may be formed in the gas phase by charge transfer collisions, which has been proposed by one of the present co-authors in 1991 [7]. A similar proposal, with the additional neutralization at surfaces of narrow tubes, was given more recently and was shown to give excellent results without the damage due to charging [8,9]. We may expect that the need for purely fast neutral etching will arise for technologies beyond 45 nm.

2.3. Etching of organic dielectrics

The new steps in the miniaturization of IC components, epitomized in 65 nm, 45 nm and 32 nm technologies, require the replacement of SiO_2 by low-k dielectrics, in order to reduce the RC time constants and increase the frequency of operation. In addition to porous inorganic dielectrics [10], one of the primary contenders for the low-k dielectric materials are organic materials that have values of k in the range 2.0-2.8. These materials require a completely different plasma chemistry, as has been observed in recent experiments [11,12]. A mixture of oxygen, nitrogen and hydrogen [11] is the best choice for this purpose. This is an additional benefit, as there is a lot of pressure on the microelectronics industry to replace fluorocarbons, due to their high global warming potential.

We have studied the etching in rf ICP of a prototype low-k dielectric (which is known by its commercial name SiLK) by employing Optical Emission Spectroscopy coupled with Computer Tomography [1,13]. The emission of the products of etching with the bias voltage in a two frequency system depends linearly on the bias potential. It has a significant value at zero bias, but it also increases with the bias [12]. It is very different from the etching of SiO_2, where there is a threshold at high energies and a non-linear dependence on the bias, with a maximum and a slight decrease at the energies beyond 1 keV.

These results cannot establish directly the mechanism of etching, but they are consistent with the well established proposal [11] that hydrogen atoms are the reactive species and that sidewall passivation (by nitrogen) will lead to anisotropic etching.

2.4. Modeling of etching profiles

Development of the new generation of plasma tools usually proceeds through empirical development. Having in mind the price of the individual tools, it has become necessary to develop complex plasma simulation systems that start from atomic and molecular collisions, proceed through plasma kinetics and self consistent field calculations, describe the sheaths between the plasma and electrodes, and describe how plasma affects the surface by employing surface reactions [14,15]. The last stage, critical for the analysis of applications, is the calculation of the etched profile and the relating calculations of the field development inside the substrate [15]. Different profile simulators have been developed to understand various artifacts seen in real processing conditions, such as microtrenching, sidewall bowing, undercut and sidewall roughening. In principle, two approaches are chosen, either of which discretizes the volume of the substrate and removes one box at a time [16] or one employs the level set method [17].

The idea behind the level set method is to represent the surface of the substrate by a function $\varphi(t, \mathbf{x})$, the so called level set function. The initial surface is defined by $\{\mathbf{x} \mid \varphi(0, \mathbf{x}) = 0\}$. The evolution of the surface with time is caused due to the effect of effective "forces" or fluxes of particles reaching the surface. The velocity of the point on the surface normal to it $V(t, \mathbf{x})$ is the so-called velocity function. This function is determined by physical models of the ongoing processes; in the case of etching by the fluxes of incident particles, their dependencies on different parameters, such as the angles, and by subsequent surface reactions. At a time $t > 0$, the surface is the function $\varphi(t, \mathbf{x})$. This all leads to the level set equation:

$$\frac{\partial \varphi}{\partial t} + V(t, \mathbf{x})\left|\nabla \varphi\right| = 0. \quad (1)$$

In Fig. 2, the results obtained for a test calculation performed with a constant velocity function $V = V_0 = 5$ *nm/s* (a purely isotropic etching case) are shown. The resulting etch profile is isotropic, with equal lateral and axial spreads, exactly what is not wanted in nano-electronics. In Fig. 3, the evolution of the etching profile, when the etching rate is dependent on the angle to the surface, $V=V_0 \cos\theta$, is presented. This is the simplest form of the angular dependence, but it describes the ion enhanced chemical etching correctly. In this case, we expect that the horizontal surfaces move downward, while the vertical surfaces are not affected.

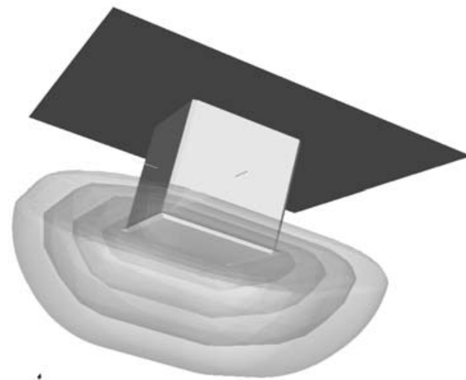

Fig. 2. Isotropic etching - etching profiles at t =0, t = 5 s, t = 10 s, t = 15 s and t = 20 s [17].

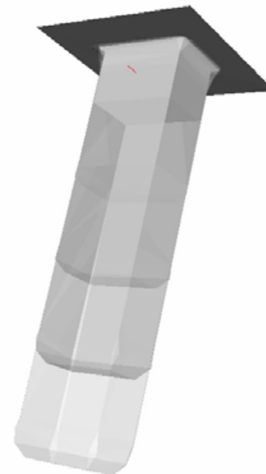

Fig. 3. An example of anisotropic etching. Optimized Lax-Friedrichs scheme etching profiles for $V=V_0 \cos\theta$ at t = 0, t = 30s, t = 60s, t = 90s and t = 120s [17].

In application of the level set method, it is critical to provide a realistic velocity as a function of the angle of collision, and other properties of the incoming ions. It is also critical to integrate the calculation with realistic characteristics of the high energy ions obtained from the plasma simulation and fluxes of radicals. If handled properly, the level set method gives profiles much faster than other techniques, it is easy to define the correct angle to the surface and its tangent. In addition, calculations can be stable, provided that the mesh size is appropriately chosen. Thus, the level set method is the best option for a quick integration to complex plasma modelling codes, for simulation of industrial etching tools.

2.5. Surface roughness in plasma etching

One of the limiting factors in applications of plasma etching in nanotechnologies in general will be the control of the plasma induced roughness or perhaps control of the surface roughness by plasma etching. The requirement is that the surface roughness should not exceed 5%-10% of the dimension, and this immediately has a bearing on 32 nm technologies, as the current roughness achieved by standard technology is of the order of 4-5 nm. There are some indications that fast neutral etching may produce much smoother surfaces [18]. We have applied the level set method to study the surface roughness, and here we consider the roughening of nano-composite materials during plasma etching for two etching modes (isotropic and anisotropic). In the past few years, the formation of surface roughness during the etching process, and its control, have become a point of interest [19]. However, in the majority of these studies, etching of homogeneous materials with a constant etch rate throughout the whole volume has been considered. Nano-composite materials, however, often consist of components with very different etch rates [20].

Possible future applications of nano-composite materials in nanotechnology (such as: mechanically reinforced lightweight components, non-linear optics, battery cathodes and ionics, nano-wires and sensors) require control of the surface roughness; both a reduction or an increase. The properties of nano-composite materials depend not only on the properties of their individual constituents, but also on their morphology and interfacial characteristics. For example, a higher roughness may be sought in order to improve the wetability of the material and its affinity to metallization [21].

In our simulation [22], the composite material consists of two phases (polymer and graphite nano-particles) randomly distributed and represented by a 3D cubic lattice. The evolution of the surface morphology during anisotropic etching for two values of the etch rate ratio s (s=2 and s=20) are considered, as representatives of the two possible components of the nano-composite material. The results shown in Fig. 4 were obtained for a large value of s=20, and a concentration p=0.1 [22].

The etch rate is higher in the case of the isotropic process, as compared to the anisotropic one. At the same time, during the isotropic process, the higher s leads to a higher overall etch rate.

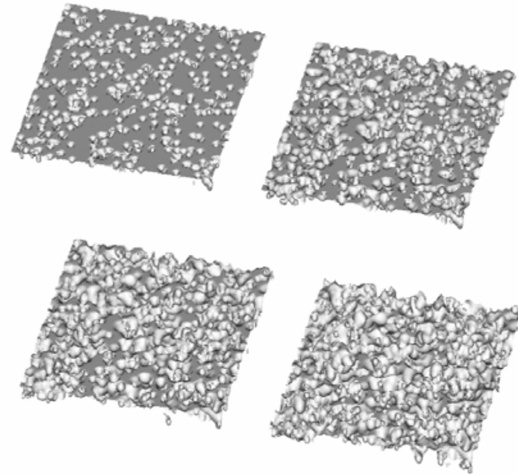

Fig.4. Images of roughening during isotropic etching of a surface of nano-composite material (in arb. units). The material is composed of two phases: a) s=20 and p=0.1 [22].

The rms roughness w quantifies the vertical roughness, and is defined as a standard deviation of the height fluctuations along a profile. Fig. 5 shows [22] the time evolution of the roughness. It first increases with the etching time and later reaches saturation.

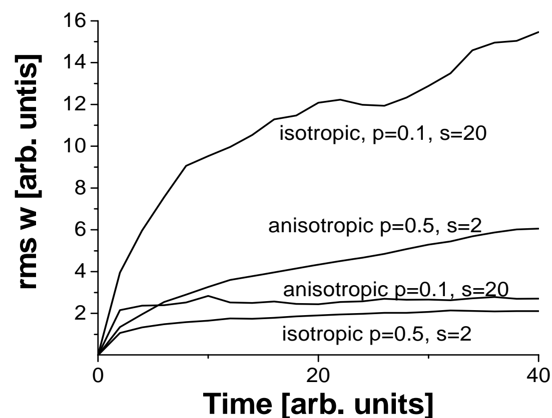

Fig.5. Dependence of the rms roughness w on etching time for two etching modes, different values of the etch rate ratio (s=2 and s=20) and the different fractions of the easily etched material (p=0.5 and p=0.1)[22].

It was found that the etching of nano-composite materials depends strongly on the etch rate ratio, s. At the same time, it depends weakly on the volume fraction of the easily etched phase p. The roughest surface appears during the isotropic process, when the etch rate ratio is large. We have proceeded to study, in a similar fashion,

the sidewall roughness, which occurs due to the co-existence of the thin layer of the photoresist and the substrate.

In the application of the level set method to surface roughness, the mesh size has to be chosen to represent properly the small structures. Thus, perhaps, modeling of the overall surface development and modeling of the development of roughness have to be performed separately, by choosing quite different mesh sizes [22].

3. Micro discharges and other atmospheric pressure discharges, and their role in nano-technologies

Non-equilibrium plasmas have proved to be able to produce chemically reactive species at a low gas temperature, while maintaining highly uniform reaction rates over relatively large areas [1]. Such plasma systems are normally run at pressures below 1 Torr, as the most favourable conditions for non-equilibrium may be found at low pressures and operation close to or below the Paschen minimum [23]. In this case, however, it is necessary to use vacuum systems, thereby making systems more complex and expensive. In addition, the complex equipment is just one part of the problem, as many targets such as living tissues or most organisms cannot be treated at all under low pressure conditions. Alternatives operating at large atmospheric pressures apparently offer a resolution to this problem, but this may not be the case in most situations. As most large size glow discharges operate in rare gases, and such discharges may be unstable due to sparking, low pressure discharges are still an option for many materials [1]. The most promising large scale atmospheric plasma source is an atmospheric-pressure diffuse coplanar surface discharge [24], which has the advantage of also producing relatively small amounts of ozone [25]. In particular, it is important that this system may operate in atmosphere, as it does not require a rare buffer gas.

3.1. Micro Discharges

The most studied forms of discharges that offer both the flexibility of non-equilibrium plasmas and yet operation in the stable regime of the Paschen minimum is that of micro discharges, where the dimensions of the system have been reduced to 10 μm - 500 μm, to secure operation under non- equilibrium conditions. For the sake of stability, those discharges usually operate under a hollow cathode geometry [26], or at least what appears to function under the hollow cathode effect but often does not [27]. It is, therefore, of importance to study the parallel plate micro discharges and test the scaling by *pd* (pressure times gap between the electrodes) in order to verify the breakdown mechanism [28,29] and whether it is possible to apply standard low pressure phenomenology like that of the Townsend theory [30].

In particular, it is interesting to verify the j/p^2 scaling (where j is the current density) [28] and test the possible application of the Townsend dark discharges, which under normal pressures (Torr-cm dimensions) have too low a current density to be of practical importance. In Fig. 6, we show the test of scaling where standard phenomenology [30] was found to function at small gaps (down to 50 μm) but tests could not yet be made down to the dimensions required to allow operation at atmospheric pressure (10 μm).

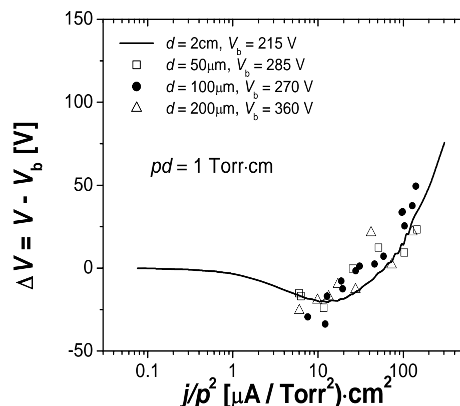

Fig. 6 Volt-Ampere characteristics for a pd = 1 Torr cm, where the discharge voltage has been scaled to the estimated breakdown voltage [28].

So far, it has been possible to achieve stable operation in the Townsend regime down to 200 μm and pressures of the order of 100 Torr. In the Townsend regime, the electric field is more or less uniform and the properties of the ions hitting the cathode may be controlled very accurately [31]. While the first applications of arrays of micro discharges [32] are focused on light sources, one should test the possibility of atmospheric surface treatment of materials, especially if it is possible to achieve etching of SiO_2 or plasma ashing of the photo resist. Thus, studies of micro discharges in RF fields [33] and of RF breakdown are essential for further advances [34].

To conclude, micro discharges hold the promise for solid state physics surface technologies of realizing atmospheric pressure technologies that allow all the advantages of low pressure non-equilibrium discharges, such as good control of the properties of particles hitting the surface, good control and stability of the discharge and simpler realization, as they require only higher pressures. However, micro discharges hold the greatest promise for the treatment of living tissues and biomedical applications in general.

4. Biomedical applications of plasmas

At present, much effort is invested in creating 'bio-compatible' plasma sources operating sometimes at low pressure, but mainly at atmospheric pressure. Such sources must produce non-thermal plasmas, operating at ambient

pressure and temperature, and should not pose any electrical or chemical hazards. We have studied the treatment of seeds under low pressure plasma conditions [35], but the set can be only applied to living tissues that can survive vacuum. Exceptional results were achieved in facilitating germination of the Empress tree (Paulownia tomentosa) seeds, and the hypothesis explaining the results is that recombination of nitrogen and oxygen atoms produced by the plasma are subsequently adsorbed, leading to the formation of NO at the surface. NO is known to trigger efficiently germination of these seeds. Most living tissues cannot be submitted to vacuum, and thus the key issue is to develop plasma sources that may operate at the atmospheric pressure, with all the benefits of the non-equilibrium plasmas.

4.1. Plasma needle

A plasma needle [36] is one of the atmospheric pressure sources that can be used for treatment of living matter which is highly sensitive when it comes to low pressure or high temperatures (above 40°C). In general, a plasma needle belongs to the category of micro discharges, as it often has sub-millimeter dimensions. A plasma needle consists of a central tungsten wire (0.5 mm in diameter) which is placed in a ceramic (OD 2 mm) tube, which is inside a glass tube (OD 6 mm). The tungsten wire is connected to a 13.56 MHz power supply, and acts as the powered electrode. The flow of the buffer feed gas (we have used helium in all experiments [37]) allows gas breakdown at a somewhat lower voltage as compared to the pure atmospheric gas, while the atmosphere mixes with the buffer gas in the region of the plasma, allowing the generation of a large number of radicals.

For practical applications on living tissues, it is particularly important to know the electrical characteristics of the plasma needle, i.e., the power transmitted to the plasma. Derivative probes were developed for that purpose by Puač et al. [37] to measure the current and voltage waveforms and determine the power inserted close to the plasma. It was found that powers of less than 0.5 W may be supplied in a stable fashion, allowing plasma formation and yet no appreciable signs of heating of the gas. The low powers were produced in a controlled fashion by using a step up transformer and a dummy load with the RF source operating at 30 W to 150 W. Less then 1% is delivered to the plasma itself.

Two modes of operation were observed, one in which the plasma is independent of the nearby surface, and the other in which it strongly affects the plasma profile.

The plasma acts as a well-controllable source of chemical energy that can be deposited on the surface. There are numerous indications that active plasma radicals play an important role in inducing specific reactions of living cells and bacteria. We have managed to apply mass spectrometry to a plasma needle operating at atmospheric pressure. We found that with a very small production of O_3, the dominant molecule was NO and the dominant radicals were N and O [38]. Ions were detected, but due to a large number of collisions their energy dependence could not be established to test the potential combined radical-ion mechanisms, such as those found in plasma sterilization.

4.2. Treatment of the living cells and tissues

The effects of these plasmas on living cells and tissues have been studied on numerous occasions in the recent literature. Dependent on the plasma conditions, several refined cell responses, such as necrosis, apoptosis or separation of cells from the tissue were induced in mammalian cells [39]. Furthermore, applications of plasma treatment in dentistry and deactivation of bacteria have been intensively studied. It appears that plasma treatment may find numerous biomedical applications [40].

We have studied the plasma treatment of plant tissues, as the properties of plant membranes are quite different from those of animals. We have analyzed the interaction of the plasma needle with gametophytes and calli, as representatives of small multicellular plant organisms that may be grown and analyzed in a controlled fashion. Cell death (necrosis) of the Polypodium prothalium occurred after high doses (power-treatment time) of plasma. As for calli, it was shown that plasma treatment induces the weight gain for the monocotyledons. While further analysis is required to elucidate the mechanisms of interaction, one may expect that the effects are triggered by the abundant radicals and reactive molecules produced by the plasma needle [38]. In Fig. 7, we show the weight gain of the calli treated by plasma, as compared to the control group. The gain occurred even when some necrosis could be observed at the tips and sharp corners of the tissue.

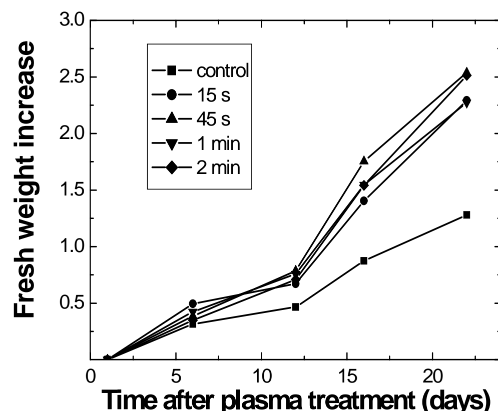

Fig. 7. Increase of the fresh weight (FW) of Fritillaria imperialis calli as a function of plasma treatment. The calli (about 5 mm diameter) were exposed to plasma for the indicated time, and the fresh weight increase was recorded for three weeks [37].

The plasma needle has the potential to be one of the most widely used sources of atmospheric pressure non-equilibrium (non-thermal) plasmas for biomedical applications. It may also compete with the presently considered plasma sources for sterilization, treatment of materials and even control of pollution.

In general, non-equilibrium plasmas offer a range of applications in biomedicine, due to their ability to treat the material only superficially without affecting the bulk, the possibility to have a small heating of the background gas compatible with living cells, and the fact that the reactants and their properties may be made in a wide range of well defined and controllable conditions. Nevertheless, a lot of work is needed concerning the exact mechanisms of interactions, in order to optimize plasmas further.

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