CHAPTER 5

EVOLUTION OF THE ELECTRICAL PARAMETERS ON RF DISCHARGE

5.1 Introduction

The presence of dust particles in the plasma has a dual effect in the surface processing industry. On one hand, the particle contamination can drastically increase the risk of device failure in microelectronics production [54]. On the other hand, properties like a uniform size distribution or enhanced chemical activity, can be useful in ceramic industry for sintering or in the fabrication of hard coatings [55] and solar cells [56]. For these reasons the early detection of particle formation and their controlled growth is necessary for the development of more efficient surface processing techniques. Unfortunately, the detection of particles in the nanometer range presents a particular challenge. Most of the research studying the behaviour of particles in plasmas is performed with lasers. However, a general problem with these methods, is the R_p^6/λ^4 -dependence of the Mie

scattering cross section in the Rayleigh limit (R_P is the particle radius and λ is the laser wavelength) that allows an high accurate detection of particle with radius greater than 10 nm (with an high power Ar ion laser at 488 nm). In other approaches, the generation of nano-particles is detected by the reduction of the electron density through electron attachment to particles using microwave interferometers or microwave resonant cavities. Unfortunately, all the methods described require expensive equipments or are unpractical to realize on commercial plasma reactors.

Recently, a number of study has pointed out as the particles growth in a plasma strongly affect the discharge impedance, leading to a more resistive behaviour. In addition, the non-linear character of plasma brings to a significant anharmonic waveform of the electrical parameters. In particular, higher harmonics seem to be very sensitive to the appearance of particles since the nucleation phase. So, the time analysis of these parameters (discharge current, voltage and phase angle) could become a simple and non-invasive technique to detect the particle occurrence which can be implemented in a simple way on a commercial plasma processing tools.

5.2 Time evolution of the electrical parameters

The evolution of the RF current, voltage, phase angle and their harmonic content are shown in figures 5.1, 5.2 and 5.3 for four different gas temperatures: 17 °C, 80 °C, 120 °C and 180 °C. Using these data to calculate the RF power behind the matching network, we estimate that maximally about 70% or 7 W of the forward power is delivered to the plasma chamber. The rest is absorbed in the cables and the matching network. Note however that during the experiments the impedance of the matching network is fixed. Thus, the net power input into the plasma

fluctuates as a result of changing conditions during the particle growth cycle. The appearance and the growth of the dust particles in the Ar-silane plasma changes the characteristics of the fundamental current as well as its harmonics (see figure 5.1).



Figure 5.1 - Evolution of the fundamental and harmonics up to fifth of the RF current at different gas temperatures.



Figure 5.2 - *Evolution of the fundamental and harmonics up to fifth of the RF voltage at different gas temperatures.*

Strong changes occur in all components and at each value of gas temperature. The strongest variations are recorded for the third and fourth harmonics (figures 5.1c and 5.1d), which drop to 25% and 10% of their initial value respectively. All other components, have this jump, but less pronounced.



Figure 5.3 - *Evolution of the phase angle between RF current and voltage of the fundamental and harmonics up to fifth at different gas temperatures.*

In particular, the second harmonic (figure 5.1b) shows only weak variations during the discharge time, which indicates less sensitivity to the occurrence of dust particles in the plasma.

Figure 5.2 shows the evolution of the RF voltage. Variations of 30% up to 70% from the initial values are measured for all components during the particle growth cycle. It should be noted that, in agreement with the data on

current, the strongest voltage drop is recorded for the third and fourth harmonics (see figures 5.2c and 5.2d).

The changes in phase angle between current and voltage during particle growth are shown in figure 5.3. The fundamental phase (figure 5.3a) only changes within few degrees during all the discharge time. However, it is obvious that the plasma changes from a nearly perfect capacitive mode, with a 90° phase angle, to a more resistive mode as the angle becomes smaller during particle formation. The higher harmonics, especially the third and fourth one, show big phase changes and are much more sensitive to the formation of the dust particles (figures 5.3c and 5.3d).

Generally, these results are in good agreement with the electrical parameters measured by Boufendi [59] as well as Shen and Kortshagen [61]. Analogies can be found in the evolution of the higher harmonics of current and voltage (especially for the third and the fourth ones) and in the strong variation of the higher harmonics of the phase angle. In our work we recorded bigger changes for all the electrical parameters, even at fundamental frequency, than in the results presented in a work of Shen and Kortshagen. This is probably due to the reduced volume of the discharge and the different gas mixture composition (here we use 5% Silane, compared to 0.5% in [61]). Nevertheless, the good agreement between the various experiments shows that the particle formation process and its impact on the electrical characteristics is independent of the particular reactor and plasma geometry. This proves that a general - device independent – model of particle formation and the particle plasma interaction is viable.

5.3 Effect of the gas temperature on the electrical parameters

Looking at the variations of the electrical parameters shown in the previous figures, when the electrode (and then gas) temperature is increased, a delay in time is observable. In figure 5.4 the time evolution of the fourth harmonic of current at 80 °C is shown. When the discharge is turned on the harmonic grows till it becomes stable. At the time t_1 it starts to decrease linearly up to the time t_2 when the previously mentioned strong drop occurs. The end of the drop is referred to as t_3 . It has been noticed that the time t_2 is a critical point for all the electrical components, as for t_1 and t_3 the fourth harmonic of current is the only one for which both of the times are clearly defined.



Figure 5.4 – Evolution of the fourth harmonic of current at gas temperature of 80 °C.

In order to determine if any relation sussists between the times t_1 , t_2 , t_3 previously defined and the gas temperature, several experiments are carried out. The results are presented in the figures 5.5, 5.6 and 5.7.



Figure 5.5 – Effect of gas temperature on the first time interval from figure 5.4: plasma ignition to t_1 .



Figure 5.6 – Effect of gas temperature on the second time interval from figure 5.4: time t_1 to t_2 .

Surprisingly, the first interval (ignition- t_1) shows a linear dependence on temperature. Due to the very fast discharge process at 17 °C, the time t_1 is not clearly defined. At the contrary, the second (t_1 - t_2 in figure 5.6) and third (t_2 - t_3 in figure 5.7) interval can be fitted by an exponential or asymptotic curve.



Figure 5.7 – Effect of gas temperature on the third time interval from figure 5.4: time t_2 to t_3 .

These relations confirm the results achieved by Boufendi *et al* for the amplitude of the third harmonic of current [60] and with laser diagnostics [59]. The same authors identify the time t_2 as the start of the so-called $\alpha \rightarrow \gamma'$ transition just after the beginning of the coalescence phase [59,60]. Consequently, t_1 and t_3 can be tentatively ascribed to the start of the nucleation phase and the end of the coalescence phase respectively. These results show that a detection technique for plasma control before coagulation phase can be available in a much easier way than by using sophisticated laser diagnostics.

5.4 Comparison with the optical measurements

A comparison between the electrical emission, and scattering measurements (see chapter 3) is presented in figure 5.8. In particular the fourth harmonic of current, the Si-H line and the He-Ne scattering are compared for two gas temperatures: 80 $^{\circ}$ C (figure 5.8a) and 120 $^{\circ}$ C (figure 5.8b).



Figure 5.8 – Comparison of the time evolution of the fourth harmonic of current (solid line), the Si-H line intensity (squares) and the He-Ne scattered light (crosses) for a gas temperatures of 80 °C (a) and 120 °C (b). The vertical markers indicate t_2 and t_3 (see figure 5.4).

At time t_2 , when the drop of the fourth harmonic of current occurs, the Si-H line intensity starts to increase. Moreover, the end of the drop t₃ corresponds to the instant in time when the Si-H line reaches the maximum value. At that time the intensity of the He scattered line starts to increase rapidly. As the particles at the end of the agglomeration phase are expected to be about 20 nm, this agrees very well with the estimated detection limit for the He-Ne scattering. During the agglomeration phase the electron density is drastically reduced, while the electron temperature increases. This typically results in an increased plasma emission, which is sustained in argon emission during the following homogeneous particle growth phase. In contrast, the Si-H emission has a maximum at the end of the agglomeration phase. The decrease of the Si-H emission during the homogeneous growth phase, suggests a reduction of the overall SiH_x density due to depletion. The overall correlation between electrical, emission and scattering data shows that particle growth affects various plasma properties. The various data give slightly different information, which taken together result in a complete picture of the particle growth process.