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POWDER DYNAMICS IN VHF SILANE PLASMAS

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ABSTRACT

Particulate contamination produced during plasma-assisted deposition of amorphous silicon devices can be responsible for reduced quality and yield. The threshold for powder formation imposes an upper limit on the rf power and hence the deposition rate. In this work, the parallel-plate capacitor discharge volume is illuminated and global, spatio-temporal powder dynamics are recorded by CCD camera for analysis. The onset of powder production is determined visually and from observed modifications to the discharge electrical properties such as the matching condition, the dc self bias and the rf power transfer efficiency. A systematic study has been made of the powder-free operational space as a function of rf power, rf frequency (13.56 -70 MHz) and substrate temperature.

I. INTRODUCTION

Powder formation during plasma-enhanced chemical vapour deposition (PECVD) is a source of contamination to be eliminated. On the other hand, the plasma synthesis of ultra-fine, high-purity microparticles has potential in the fields of ceramics and catalysors.

Since the work of Spears [1,2,3], powder formation in silane plasmas used for amorphous silicon (a-Si:H) deposition has been intensively studied [4-7]. In most cases, the particles are detected by laser scattering, and spatial resolution obtained by displacing the optical system or the reactor. A global visualisation of particles in etching plasmas has been obtained by CCD camera and laser rastering [8]. In all cases, negatively-charged particles suspended in the plasma accumulate near the plasma sheaths. Besides electrostatic forces, the particles are subject to thermophoresis forces [9] in the presence of any gas temperature gradient as well as viscous forces in the gas flow [4]. In addition, recent theoretical work has shown that charged particles can modify the electron energy distribution by Coulomb collisions and electron attachment [10], and introduce recombination centres [11].

In this paper, powder is observed by means of white light illumination of the reactor volume for pure silane plasmas [5]. The global, spatio-temporal evolution of the powder is recorded and digitised by CCD camera for qualitative analysis. Modifications to the electrode voltage also serve as a powder diagnostic. After a description of the experiment, the results are presented in the following order : Section III discusses the powder influence on electrical measurements such as plasma conductance, power transfer to the plasma and electrode self-bias; Section IV describes the dependence of powder profiles on electrode temperature and excitation

frequency (from 13.56 to 70 MHz). Section V is concerned with the temperature and frequency parameter space for powder-free operation.

II. EXPERIMENTAL ARRANGEMENT

The reactor (Figure 1) consists of two, symmetric stainless steel cylindrical electrodes of diameter 130 mm positioned 20 mm apart. The gas influx of 10 to 50 sccm is in the side wall of the cubic vacuum chamber of side 400 mm. The silane pressure of 0.1 - 0.3 mbar is feedback-controlled by means of a capacitance barometer acting upon the turbopump speed.

The 8 x 8 cm glass substrate is held by a steel template to the underside of the upper, grounded electrode which can be heated to 250 ^{o}C. The lower, rf electrode can be water-cooled and has a grounded guard screen. The rf power from a wideband (10 kHz - 200 MHz) amplifier is capacitatively-coupled to the rf electrode via a π-matching network at the input of which the forward and reflected rf power is measured with a directional coupler. The reactor walls are grounded and maintained at 100°C.

A passive, rf voltage probe ($\div$100) is positioned inside the lower cylinder and contacts the back surface of the rf electrode exposed to the plasma. The voltage waveform is monitored on an electrically-floating oscilloscope.

The reactor volume is illuminated with white light and observed at 90° by eye or CCD camera [5]. The windows are positioned at the end of 220 mm-long radial ports which suffices to eliminate any deposition which would otherwise falsify intensity measurements.

III. THE INFLUENCE OF POWDER ON PLASMA ELECTRICAL PROPERTIES

The voltage measurement made directly on the rf electrode, in conjunction with the total input power, can be used to estimate the effective power dissipated in the plasma by the subtractive method [12,13,14]. By this method, the plasma power is given by the difference between the input power without plasma and the input power with plasma, on condition that the measurements are made at constant electrode voltage by adjustment of the amplifier output level.

Figure 2a shows the square of the rms electrode voltage as a function of input power, measured with plasma (P_{tot}) and without plasma (P_o in vacuum). The reciprocal slope of the vacuum line can be equated with the circuit equivalent conductance G_o (real part of the admittance) as measured at the voltage probe position [14] using the relation :

$$P_o = G_o \, . \, V^2_{rms} \, .$$

G_o is observed to be a constant as expected for a linear circuit. The conductance with plasma, G_{tot}, is approximately independent of input power for a powder-free plasma (denoted by the unshaded region in Figure 2a). This can be understood by a simple calculation which shows that plasma conductance is approximately independent of electron density. Further evidence of constant plasma impedance is that the matching condition for zero reflected power, as determined by the value of the variable capacitors in the π-network, is independent of input power in the absence of powder formation.

As the input power is increased, a threshold level (P_{thres} in Figure 2a) is reached at which the discharge parameters change spontaneously. This threshold power and the duration of the change are reproducible for given

discharge initial conditions. During this transition, the rf voltage falls steadily and the matching condition is perturbed, ie the reflected power increases. Following the onset of these electrical changes, where no powder is as yet visible, powder is seen to accumulate near the lower, rf electrode edge where the electric fringing fields to the grounded guard screen are most concentrated. The powder formation then spreads towards the axis, eventually forming a suspended disc at the plasma/sheath boundary of the rf electrode. At all times, powder streams continuously radially outwards to form an orange/yellow deposit on the side walls and the chamber floor. Once the powder evolution has reached steady state, the rf voltage and reflected power levels are also stabilised. The break in the plasma line of Figure 2a corresponds to this transition which lasts from 0.5 to 20 seconds or more, depending on the pressure, inter-electrode spacing, rf frequency and electrode temperatures.

These electrical modifications correspond to an alteration in discharge impedance due to the powder formation. From the reduction in slope, the total conductance has substantially increased for the case in Figure 2a. An increase in conductance, the real part of the admittance, can be explained by a diminution of the phase angle between rf current and voltage; such a phase reduction has been reported for silane/argon plasmas [4] and diverse 'dusty' plasmas [9].

If we define the rf power transfer efficiency for our device to be the ratio of plasma power to total input power, it can be seen from Figure 2a that this fraction is higher for the plasma-powder ensemble than for the powder-free plasma - this is shown explicitly in Figure 2b. Numerical calculations [10] show that an effect of charged particles in plasmas is to shift the electron energy distribution towards lower energies by Coulomb

scattering and attachment. The increase in power transfer fraction from the electric field to the plasma-powder ensemble is thus explained by the higher cross-section for electron energy loss due to trapped particles in the discharge. However, although the power transfer fraction may be higher, this is to the detriment of the required plasma processes such as dissociation and ionisation because the content of high energy electrons in the tail of the distribution is reduced.

When the power is raised still further above the threshold value, the large quantity of powder produced eventually absorbs all the high energy electron content, thus stopping ionisation and the plasma is extinguished.

Trapped particles also influence the dc self-bias (V_{sb}) of the rf electrode. Self-bias depends on the ratio of the grounded and rf electrode effective areas for plasma current flow and is negative if the rf electrode area is less than the grounded electrode surface [15]. In the absence of powder, the self-bias for silane plasmas in our reactor is zero or slightly negative, as expected for symmetric electrodes in a grounded vacuum chamber : the mean-free-path of electrons is small enough to prevent significant leakage of plasma current outside of the electrode gap to the grounded chamber walls. With powder, however, the self-bias becomes positive; this observation has previously been attributed to the presence of negative ions [7]. In our case, we attribute the positive self-bias to the influence of powder which generally accumulates at the sheath in front of the cold rf electrode (see Section IV) : the negatively-charged powder particles have the effect of reducing the local free electron density and energy, and consequently the self-bias must rise in order to maintain equilibrium between electron and ion currents across the rf electrode sheath.

IV. POWDER PROFILES AS A FUNCTION OF TEMPERATURE AND RF FREQUENCY

Spatial profiles of powder formed and trapped in silane plasmas were monitored and digitised with a CCD camera by illumination of the reactor volume with white light [5]. The scattered intensity is a convolution of the particle size and number density, but nevertheless is a useful guide to the volume of particulates in the plasma. For the experiments described in this section, the substrate temperature was varied between 30 ^oC and 220 ^oC by heating the upper grounded electrode, while the lower rf electrode was maintained at 15 ^oC. The dependence on rf excitation frequency in the Very High Frequency range was also studied from 30 to 70 MHz.

Figure 3 represents a vertical profile, near to the electrode axis, of scattered light from the powder for different substrate temperatures with the effective plasma power and rf frequency held constant. At low temperatures, two distinct powder layers were visible - it is proposed that the negatively-charged particles, surrounded by positive ions, experience a potential well which traps the powder particles at the plasma/sheath boundaries [4]. When the temperature is increased, the total scattered intensity is reduced and the profile is displaced towards the cold electrode. For substrate temperatures above 170 ^oC, there is no longer any powder at the substrate sheath.

This profile displacement can be explained in terms of the thermophoresis force [9], by which a temperature gradient in the gas causes particles to move towards the cold electrode. For a sufficiently high temperature gradient (80 ^oC/cm in our case), the thermophoresis force appears strong enough to overcome the electric forces acting to hold

particles at the substrate sheath/plasma boundary, and displace them to the rf electrode sheath/plasma boundary.

Figure 4 shows vertical profiles of scattered light for different rf excitation frequencies at constant plasma effective power. The electrode temperatures are fixed at values where all the powder accumulates above the cold rf electrode, as explained above. The total scattered intensity and the layer width both diminish as the frequency is increased. The sheath width, as judged by the gap between the electrode and the powder layer, is also reduced at high frequencies. A possible explanation for these frequency effects is that the sheath potentials are strongly reduced in the Very High Frequency (VHF) range, for constant plasma power, thereby proportionately reducing the electric forces trapping the powder in the plasma.

To summarise this section, the problem of particulate contamination of films during plasma-assisted deposition could be alleviated by using temperature gradients to evacuate powder away from the film surface by particle thermophoresis [9], and by operating in the Very High Frequency regime to reduce electrostatic particle trapping in the plasma.

V. OPERATION DIAGRAMS FOR POWDER-FREE PLASMAS

In this section the parameter space for powder-free plasma operation is defined, for our reactor geometry, by measuring the plasma power threshold as a function of electrode temperature and rf frequency. This threshold is the boundary between powder-free plasma and plasma in which powder forms.

Figure 5 shows the plasma power threshold as a function of substrate temperature with the rf electrode maintained at 50 °C. At high substrate temperatures, higher power operation is possible before visible powder

formation begins. The powder initially accumulates at the sheath edge of the lower electrode as decribed in Section IV. Particle thermophoresis alone cannot account for the rise in plasma power threshold at high substrate temperature. This can be seen even more clearly in isothermal plasma experiments (since thermal gradients and therefore thermophoresis are absent), where powder formation can be suppressed if the electrodes are sufficiently hot [4]. To explain powder suppression at high substrate temperature, it is necessary to consider the origin of powder formation in the plasma. The powder in silane plasmas is formed by agglomeration of certain reactive radicals (powder precursors) produced by dissociation of silane by electron impact, and so the powder source term has to be considered. We can assume that radical reaction rates with the growing film surface depend on the surface temperature, because the deposition rate varies with temperature [16], all else being constant. This is equivalent to saying that the radical sticking coefficients are temperature-dependent. The surface temperature, therefore, influences the radical composition in the discharge by determining which radicals are preferentially incorporated into the film, and which are less likely to stick, thereby remaining in the plasma. The rise in power threshold with substrate temperature can then be explained if we suppose that the powder precursor radicals have a greater probability of sticking to the film surface at higher surface temperature.

If the power is further increased beyond the threshold level in our reactor, a second power limit is reached for which powder also begins to accumulate at the substrate sheath edge (Figure 5). The power difference between these two levels is larger at higher substrate temperatures probably because the thermophoresis force, displacing particles to the lower electrode sheath, is stronger in the larger temperature gradient. At

this second power limit, powder formation always begins directly opposite the junction of the glass substrate and the steel template holding the substrate to the ground electrode. This is probably caused by fringing fields due to the topography of the electrode surface, as reported by Selwyn [8,17] for powder trapped in etching plasmas. Careful design of the substrate holder to reduce fringing fields would help to avoid film contamination from particulates forming at the substrate sheath.

Figure 6 shows the plasma power threshold diagram for powder-free operation as a function of rf excitation frequency. Here, the upper electrode temperature is fixed at 200 °C and the lower at 50 °C. This figure illustrates the fact that higher rf frequency operation permits increased plasma power density whilst remaining free from particulate contamination. As mentioned in Section IV, a plausible explanation is that sheath potentials are smaller at higher frequency, for a given plasma power, thus reducing electrostatic trapping of powder and their precursor radicals. However, it should not be discounted that Very High Frequency (VHF) plasma operation also alters the relative distribution of the various dissociation products (eg by changing the electron energy distribution function as compared to that at 13.56 MHz) which could inhibit the production of those radicals acting as powder precursors. Some evidence to show the effect of frequency on dissociation and EEDF is the increase in deposition rate and plasma-induced emission in the VHF regime [18].

The above measurements were all made with continuous, constant amplitude rf power. Another important technique for powder suppression is by using rf power modulation at kHz frequencies [5,6], or intermittent pulsed rf power [4]. This method, combined with high substrate temperature and frequency, could allow still larger plasma power densities with no

powder formation, with a corresponding increase in deposition rate of high-quality films.

VI. CONCLUSIONS

The problem of powder contamination in plasma deposition reactors has been investigated. We discuss the transition from powder-free to powder-producing plasmas, during which the discharge voltage, plasma conductance, power transfer fraction and rf electrode dc self-bias all change spontaneously. This transition defines a maximum power threshold for powder-free plasma.

High substrate temperature and high rf excitation frequency both have the effect of raising the power threshold, allowing a higher power density and thereby increased deposition rate without powder contamination. It is argued that this could be due to a change in radical composition of the plasma in both cases, inhibiting the development of powder precursors in the discharge.

If plasma conditions are such that powder is produced, profile measurements of the scattered light show that the risk of film surface contamination can be minimised in two ways :

i) By maintaining the substrate at higher temperature than the counter electrode, thermophoresis forces [9] displace the particles away from the film, and

ii) By operating at higher frequency, the discharge electric fields are diminished, thereby the electric trapping forces [4] and so the accumulation of powder in the plasma is reduced.

From the point of view of powder contamination, plasma operation at high substrate temperature and high frequency are both seen to be beneficial.

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FIGURE CAPTIONS

Figure 1 : Schematic of experimental arrangement showing rf circuit, electrode voltage probe and white light illumination.

Figure 2 : Influence of powder on electrical properties of the discharge such as : a) rf electrode rms voltage squared; b) power transfer efficiency and ratio of dc self-bias (V_{sb}) to rf voltage (0.3 mbar, 30 sccm, 20 mm electrode spacing, 30 MHz, upper electrode temperature 200 °C).

Figure 3 : Dependence of powder profiles on upper electrode temperature (0.3 mbar, 30 sccm, 20 mm electrode spacing, 30 MHz, 6.4 W_{eff}).

Figure 4 : Dependence of powder profiles on rf frequency (0.3 mbar, 30 sccm, 20 mm electrode spacing, 5 W_{eff}, heated electrode temperature 220 °C, bottom electrode water cooled).

Figure 5 : Heated electrode temperature dependence of plasma power threshold for powder onset ("POWDER") and for powder trapping near the heated electrode ("POWDER HOT SIDE").

Figure 6 : Rf frequency dependence of plasma power threshold for powder onset (0.3 mbar, 30 sccm, 20 mm electrode spacing, upper electrode temperature 200 °C, lower electrode temperature 50 °C).

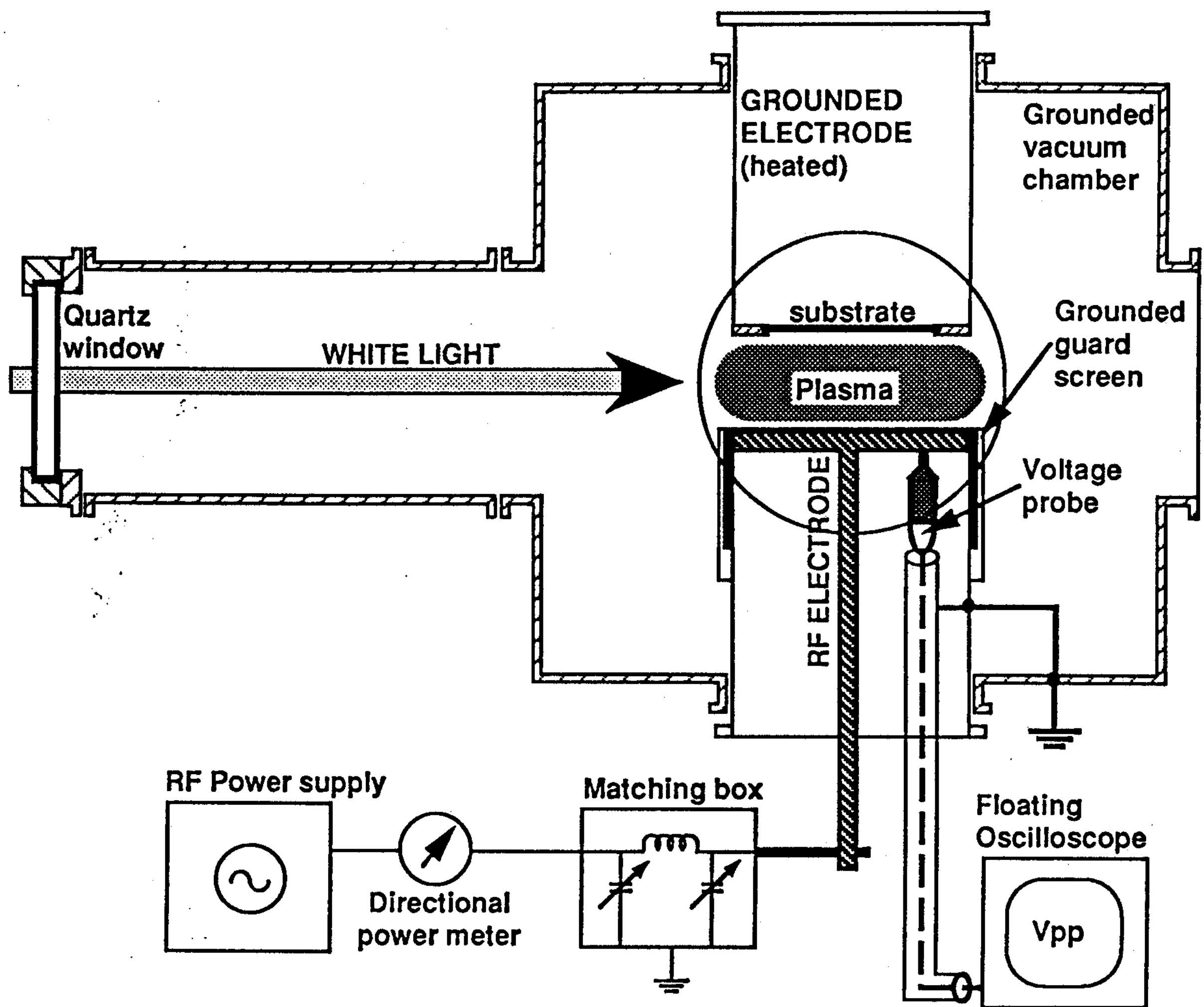
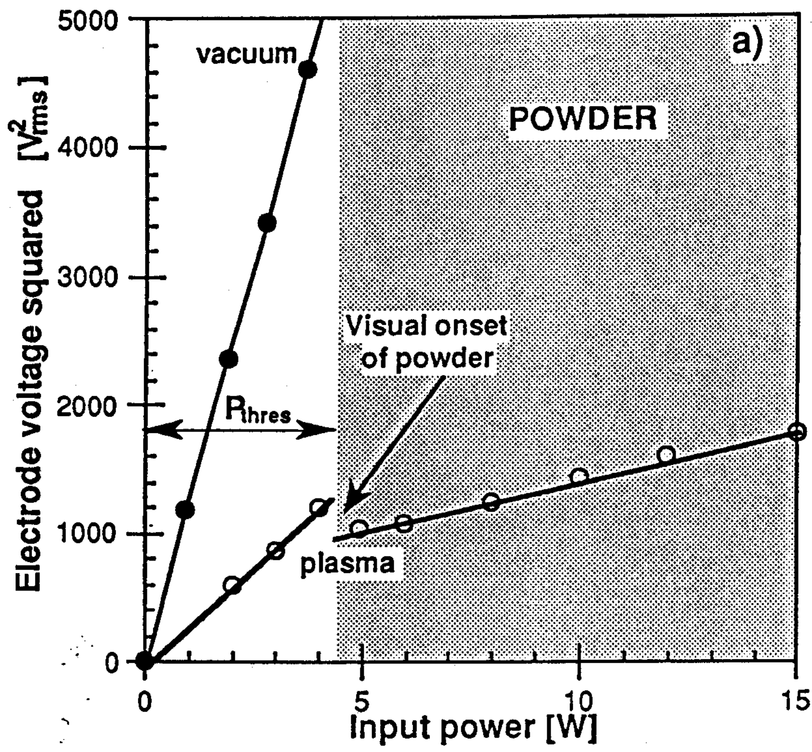


FIG 1



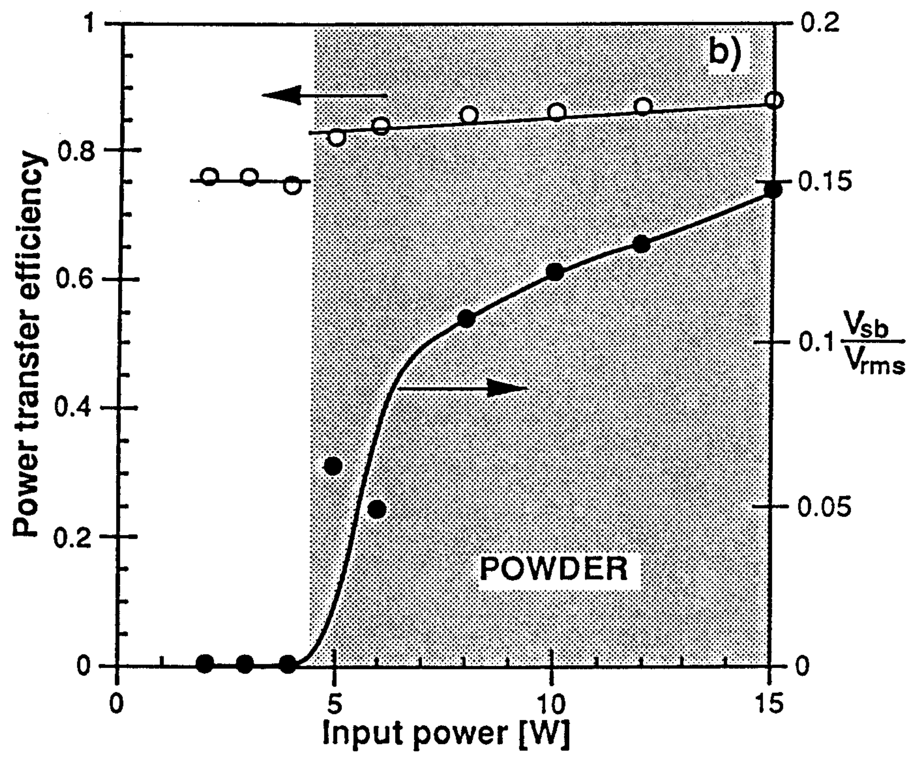


FIG 2

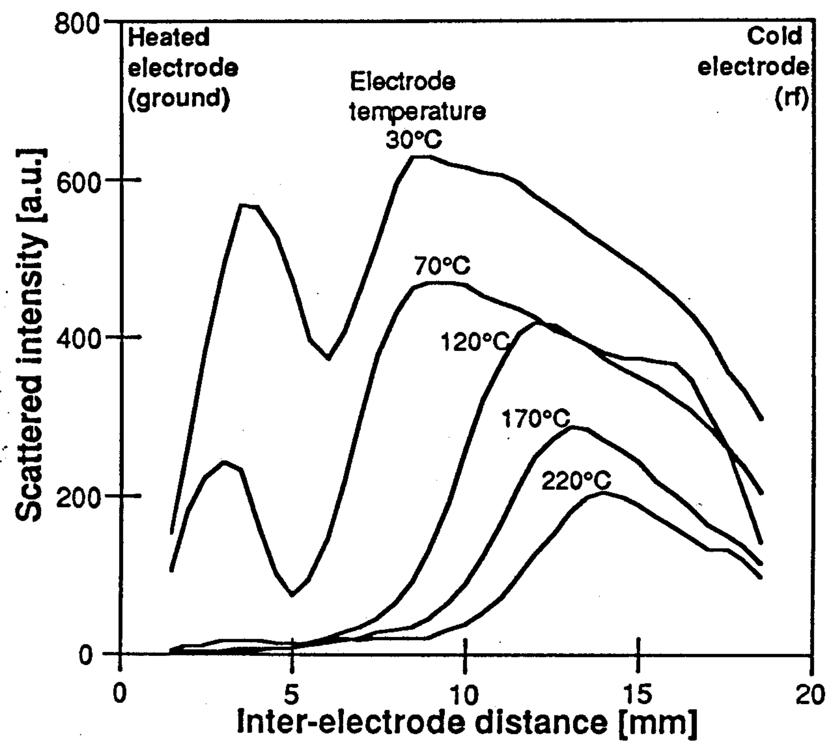


FIG 3

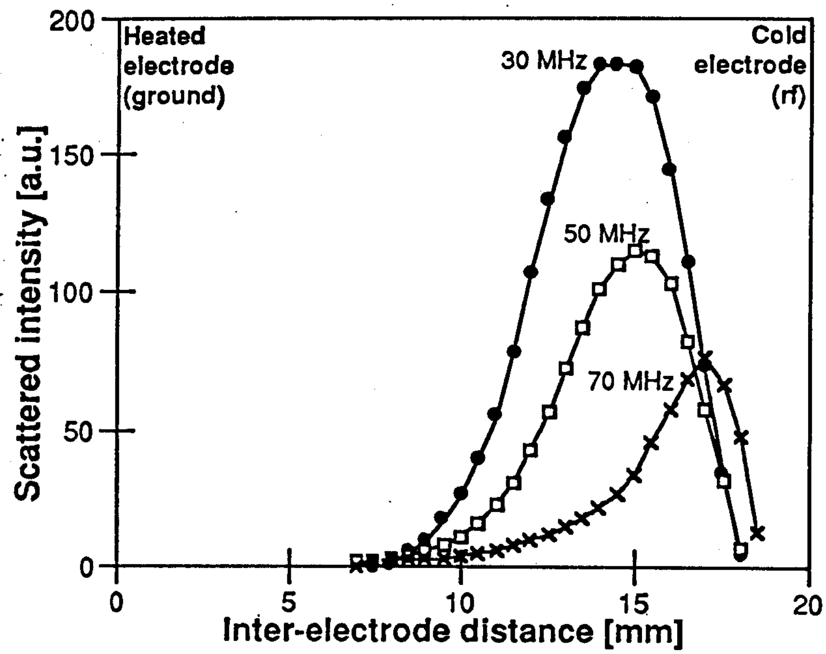


FIG 4

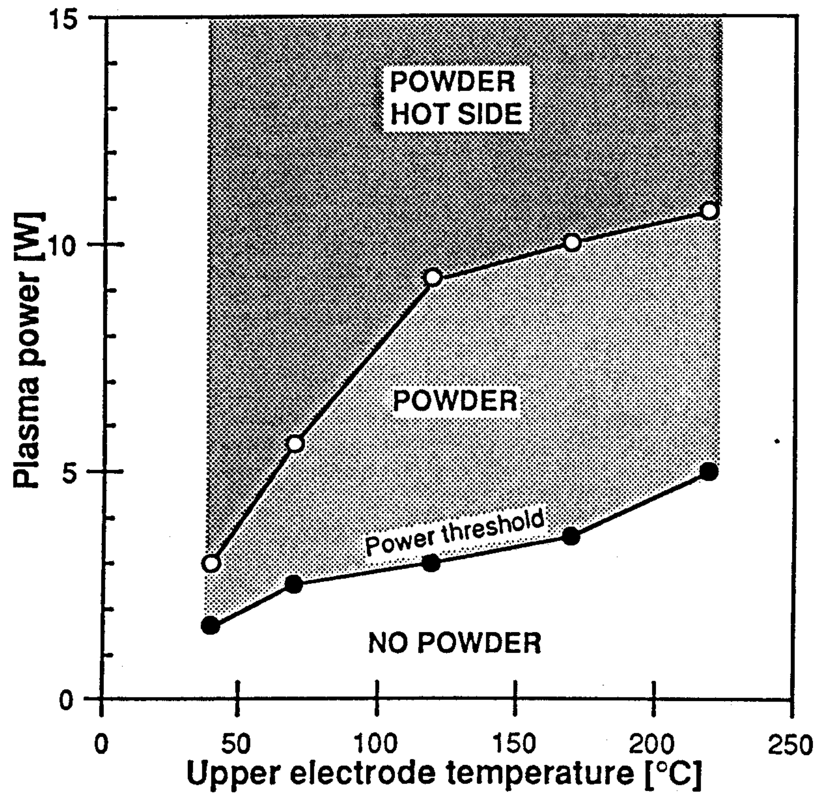


FIG 5

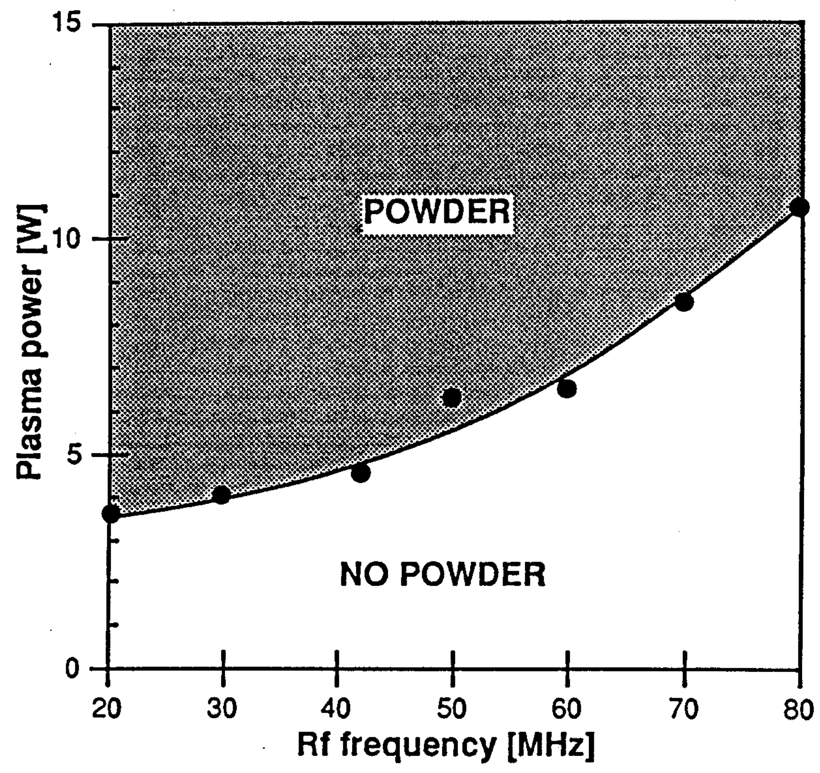


FIG 6