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Laser-Particulate Interactions in a Dusty RF Plasma

E. Stoffels, W. W. Stoffels, D. Vender, G. M. W. Kroesen, and F. J. de Hoog

Abstract—The interaction of particulates formed in an argon RF discharge containing 1–5% CCl_2F_2 admixture with a pulsed infrared laser (Nd:YAG, intensity $\sim 10^9 \text{ W} \cdot \text{m}^{-2}$, pulse duration $\sim 10^{-4} \text{ s}$) has been studied *in situ*. The white light emitted during this process has been monitored as a function of time and wavelength using a fast photo diode and an optical multichannel analyser. The spectra have been fitted with blackbody curves with a standard deviation of 5%. A spectral temperature of about 3500 K has been obtained for various plasma conditions and attributed to the decomposition temperature of the particulate material. A model based on laser heating, internal heat conduction and chemical decomposition is compared with the experimental results. This model predicts the time constants for heating and decomposition of the particulates and explains the dependence of the measured emission intensity on the laser intensity.

I. INTRODUCTION

SMALL dust particulates have been observed in a wide variety of semiconductor processing plasmas [1]–[3]. These grains, ranging in size from several nanometers to millimeters cause severe damage to devices fabricated by plasma processing. As advanced semiconductor technology aims at fabricating very small structures, it is necessary to avoid formation of even the smallest grains in the reactor. On the other hand, abundant particulate formation in several discharges suggests a new application of these plasmas as particulate sources, delivering grains with a desired size and physical properties. In both cases an *in situ* measurement of the particulate size and density is required. Commonly applied light scattering is an experimentally easy technique, but its efficiency decreases drastically for particulates much smaller than the wavelength of the scattered light. In the Rayleigh scattering regime the intensity is proportional to the sixth power of the radius. However, other kinds of laser interaction with the particulates can possibly provide a more sensitive detection technique.

A high power infrared laser (Nd:YAG) has been used for the destruction of particulates in an argon/ CCl_2F_2 RF plasma [4]. The laser-particulate interaction is followed by bright, white light emission. A comparison with light scattering has revealed that this emission is at least 10 times more intense than scattering at 90° and the intensity ratio of these signals increases with decreasing particulate size. There are some more advantages of detecting the broad band emission. The stray light, which disturbs the scattering experiments can be easily eliminated here. Moreover, a uniform angular

distribution of the radiation is experimentally more convenient for detection.

In this work we study the emission, following the laser destruction of particulates, by means of time resolved optical emission spectroscopy. Most physical properties of these particulates are unknown. However, considering the composition of the gas and the infrared absorption spectra we can assume, that they are teflon like polymers $(\text{CF}_2)_x$ [5]. In the estimates made below the physical properties of teflon are taken and particulates are assumed to be spherical.

II. THEORY

The initial particulate temperature can be slightly above the ambient plasma temperature ($\sim 300 \text{ K}$) as its surface is heated by bombarding ions accelerated in the sheath surrounding the particulates. When a laser beam strikes the cluster surface, laser energy is absorbed, causing the particulate to be heated. Heat conduction within the particulate will homogenize the temperature distribution. Energy is lost at the surface by radiation, evaporation or thermal decomposition and conductive losses.

The place and time dependent energy balance within a particulate can be written as:

$$\rho C \frac{\partial T}{\partial t} = \kappa I (1 - R_\theta) e^{-\kappa d} + \nabla \cdot (K \nabla T) - \delta(r - a)(\Phi_{\text{rad}} + \Phi_c + \Phi_v) \quad (1)$$

where ρ and C are the solid state mass density ($\text{kg} \cdot \text{m}^{-3}$) and heat capacity ($\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$). The first term on the right hand side describes the laser heating, with κ —absorption coefficient in the solid state (m^{-1}), I —laser intensity ($\text{W} \cdot \text{m}^{-2}$), $1 - R_\theta$ the angle dependent fraction of the laser intensity penetrating the particulate and d —the optical path length of a laser photon at a given point within the particulate. The second term represents the heat conduction within the particulate (K —thermal conductivity). The last three terms refer to surface processes (denoted by the Dirac delta function) at $r = a$, where a is the radius of a particulate: Φ_{rad} —radiation losses, Φ_c —conduction losses to ambient gas and Φ_v —losses due to evaporation or chemical decomposition (all in $\text{W} \cdot \text{m}^{-2}$).

Based on this formula several estimations can be made. First we have to note that if we expect the particulates to emit blackbody like radiation, the optical depth for a single particulate ($\kappa \cdot a$), must be at least 10. Together with a typical size of a particulate (0.1 – $1 \mu\text{m}$), estimated from the ratio of forward and perpendicular He-Ne laser scattering, this gives us a lower limit for the absorption coefficient of 10^7 – 10^8 m^{-1} , which is a realistic value for opaque materials. Therefore the total power deposited in a particulate by the laser is simply

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$\pi a^2 \cdot (1 - R)I$, where RI denotes the total scattered intensity. Note that in case of such a high absorption by a single particulate the laser intensity would be seriously attenuated in the plasma even for very small R . Since we observe an attenuation (A) of about 1% over the whole plasma length (L), this gives us an estimate of the particulate number density:

$$N = \frac{A}{\pi a^2 \cdot L} \quad (2)$$

Substituting $L = 0.2$ m it gives 10^{10} – 10^{12} m⁻³ for particulates between 0.1 and 1 μ m.

Further we note that for absorbing particulates with a in the order of the wavelength the reflection coefficient is about 1/2 [6]. For smaller particulates ($a < \lambda$) the scattered intensity (R) decreases as a^6 . The absorbed intensity on the other hand depends on a^2 when $a > \kappa^{-1}$ and on a^3 , when $a < \kappa^{-1}$ and homogeneous volume heating occurs. This feature makes laser absorption interesting as a detection technique for small opaque clusters. Even though for smaller particulates the emission spectrum deviates strongly from blackbody, other radiative processes (fluorescence, photoluminescence) can be useful for cluster detection.

Some other simplifications of the energy balance (1) can be made by estimating typical time constants for thermal conduction losses. This can be done by introducing appropriate gradient lengths in a heat conduction equation. A time constant for heat conduction within the particulate can be expressed by: $\tau_{in} = \rho C a^2 / 3K$, where the gradient length is estimated by the particulate size a . The characteristic time of the energy loss by thermal conduction to the ambient gas (Φ_c) is: $\tau_{out} = \rho C a \Lambda / 3 K_{amb}$. Here Λ is the gradient length, which we approximate by the mean free path for neutrals in the ambient gas and K_{amb} is the thermal conductivity of the gas. Substituting the typical values for teflon: $\rho = 2.2 \cdot 10^3$ m⁻³, $C = 10^3$ J · kg⁻¹ · K⁻¹ and $K = 0.3$ W · m⁻¹ · K⁻¹, and $\Lambda \sim 0.3$ mm, $K_{amb} = 1.6 \cdot 10^{-2}$ W · m⁻¹ · K⁻¹ for an argon plasma at 27 Pa and 300 K, we find $\tau_{in} \approx 2 \cdot 10^{-6}$ s and $\tau_{out} \approx 10$ ms for a 1 μ m particulate. This implies that at the time scale of the laser pulse (100 μ s), the internal heat conduction provides a uniform temperature profile within a particulate, while the heat losses to the ambient gas can be neglected.

The energy losses due to particulate evaporation or thermal decomposition can be related to the decrease rate of the particulate radius: $\Phi_v = H / V_m \cdot (da/dt)$, where H is the energy of the decomposition process (J · mol⁻¹) and V_m the molar volume (m³ · mol⁻¹). Using the above simplifications and substituting Wien's displacement law for the radiative losses the energy balance (1) integrated over the particulate volume can be written as:

$$4/3 a \rho C \frac{dT}{dt} = (1 - R)I - 4\epsilon \sigma_s T^4 - 4 \frac{H}{V_m} \frac{da}{dt} \quad (3)$$

where $\sigma_s = 5.67 \cdot 10^{-8}$ W · m⁻² · K⁻⁴ and ϵ is the integrated emissivity. In the initial phase of particulate heating the loss terms can be neglected. A simple expression for the heating time (τ_{heat}) is therefore:

$$\tau_{heat} = \frac{4a\rho C \Delta T}{3(1 - R)I} \quad (4)$$

Fig. 1. A schematic view of the parallel plate plasma configuration.

When a particulate is heated up to a sufficiently high temperature, evaporation or decomposition become important. Assuming a constant temperature during this process, the time dependent behavior of the particulate radius is:

$$\frac{da}{dt} = \frac{V_m}{4H} ((1 - R)I - 4\epsilon \sigma_s T^4) \quad (5)$$

The above formulas will be used to estimate the heating and destruction rates for our particulates.

III. EXPERIMENTAL SETUP

The capacitively coupled discharge has been operated at 13.56 MHz by an ENI ACG-3 radio frequency generator with an ENI 5 matching network optimizing the power dissipation in the plasma. The maximum power input is 120 W. The plasma is sustained between two parallel aluminum plates (diameter—12 cm, separation—5 cm). A 10 cm Si wafer is placed on the lower RF electrode. The argon/CCl₂F₂ mixture is introduced homogeneously through a slit around the RF electrode (see Fig. 1). Typical dusty plasma conditions are: 1.5 sccm (about 5%) CCl₂F₂ and 28.5 sccm Ar flow at a total pressure of 27 Pa and power input of 100 W. The measurements have been performed after 30 to 60 min of plasma operation, when only long term fluctuations in particulate density occur.

The particulate destruction has been performed using a Quanta Ray DCR-11 Nd:YAG laser. The laser has been operated mainly in the long pulse mode at 1064 nm. In this mode the laser pulse consists of a train of short (ca. 1 μ s) pulses, spread over about 150 μ s. Occasionally, the laser has also been operated in a Q-switched mode, which results in a single 20 ns pulse. The maximum available pulse energy in both cases is about 0.4 J. The donut shaped laser beam is collimated into a pencil-like beam of 2 mm diameter by means of two lenses and fired through the plasma at a variable distance from the electrodes. For the long pulse mode this results in a maximum laser intensity $I \approx 8.5 \cdot 10^8$ W ·

Fig. 2. A scheme of the optical emission setup. After a start pulse of the pc, the optical multichannel analyzer (OMA) triggers the Nd:YAG laser. The laser pulse, focused by two lenses in a parallel beam, heats the particulates in the plasma. The emitted radiation is fed to a monochromator, using a lens-fibersystem. The wavelength selected light is detected by the gated diode array of the OMA, resulting in a time and wavelength resolved signal.

m^{-2} . Typically, the experimental data have been obtained by averaging the emission over 25 laser shots. In order to avoid signal attenuation due to particulate destruction in the laser beam, the laser repetition frequency has been kept below 1 Hz. This is sufficiently low for a complete particulate redistribution and the removal of the evaporated material to the pumps. A scheme of the setup for optical emission is given in Fig. 2. The emission is collected at 90° through a quartz window. The time resolved wavelength integrated emission intensity has been measured using a fast photo diode in combination with a digital oscilloscope. A high pass filter (KG 3) has been used to discard scattering and stray light at 1064 nm. In order to collect time resolved spectra the emission has been imaged using a quartz lens on a quartz fiber guiding it to a 25 cm Jobin-Yvon monochromator supplied with a 150 grooves/mm grating. Finally the light signal has been detected by a gated intensified diode array which is part of an optical multichannel analyzer (OMA, EG&G model 1461). The transmission of the optical system and the sensitivity of the diode array have been determined using a tungsten ribbon lamp. This correction has been incorporated in all data. Since the gain of the diode array decreases rapidly in the near infrared region, only visible emission (up to 800 nm) could be collected. The background plasma radiation has been subtracted from the measured spectra.

IV. RESULTS AND DISCUSSION

The radiation caused by the particulate interaction with a strong infrared laser beam consists practically only of continuum. No atomic or molecular line (band) emission has been found, even in the extinction phase. The shape of this continuum is best approximated by a Planck blackbody curve with a temperature around 3000 K. A typical spectrum and its fit are shown in Fig. 3. The standard deviation of the

Fig. 3. A typical emission spectrum from the particulates (solid line). The dashed line is a blackbody fit to the data, corresponding to a temperature of 3600 K. The spectrum has been collected 1 cm above the powered electrode during laser destruction of particulates in a 27 Pa, 100 W plasma, containing 5% CCl_2F_2 and 95% argon at the total gas flow of 30 sccm (standard conditions). The laser energy is 0.25 J/pulse, the gate delay of the detector (OMA) with respect to the onset of the laser pulse is 30 μs and the gate width is 20 μs .

Planck curve fit for the majority of spectra does not exceed 5%. Only for weak signals in the late extinction phase the standard deviation increases up to 10%. Similar blackbody like spectra have been found by Geoghean for particulates produced by laser ablation of a solid [7].

The wavelength integrated emission intensity as a function of time is shown in Fig. 4. Laser stray light collected on the window, displaying the actual laser pulse shape is given for comparison. Fig. 5 shows the detailed time resolved structure of these two signals. It can be seen that though the laser-induced visible emission roughly follows the infrared laser signal, it does not display the short pulse time structure of the stray light (Fig. 5). This is in agreement with the blackbody character of the emission and it is essentially different from the light normally observed from a laser-induced plasma above a solid surface. In the latter case the emission, containing line (band) radiation as well as free-free continuum (Bremsstrahlung), decays rapidly within the first microsecond after the laser pulse [8], [9]. In our pressure range the plasma itself can by no means emit blackbody like radiation. The vapor cloud, generated by the laser destruction of the particulates is also unlikely to do so, as it quickly becomes optically thin while expanding. The absorption coefficient of an expanding cloud decreases with increasing cloud volume ($\kappa \sim a^{-3}$), which implies that the optical depth decreases as a^{-2} with the cloud radius. Since the expansion speed, corresponding to the surface temperature of the particulate is approximately 1000 m/s in our conditions, the optical depth of a cloud is 100 times lower than this of a solid ($a \sim 1 \mu\text{m}$) after only 10 ns. From this cloud free free, free bound or bound bound emission can be expected but not a blackbody like continuum. Therefore we can state that the observed blackbody emission originates from the surface of the heated particulates and consequently it delivers information about their heating and destruction kinetics.

An interesting aspect of the time dependent behavior of the continuum radiation (Fig. 4) is a 30 μs delay between the fast

Fig. 4. Wavelength integrated laser-induced emission ($\sim 300\text{--}1000$ nm, with a KG 3 filter) from the particulates under standard plasma conditions (solid line) and normalized laser stray light (dashed line), measured using the fast diode (averaged over 25 laser shots). The laser energy is 0.18 J/pulse.

Fig. 5. Wavelength integrated laser-induced emission (solid line) and laser stray light (dashed line), measured with the fast diode. The conditions are the same as in Fig. 3. These data correspond to single shot measurements. The position of individual laser peaks varies from shot to shot.

rising laser pulse and the maximum visible emission. A first guess is that it is caused by a slow laser heating. Indeed, (4) predicts a heating time of about $40\text{ }\mu\text{s}$ for a $1\text{ }\mu\text{m}$ cluster heated up to 3000 K at the laser energy of 0.2 J ($4 \cdot 10^8\text{ W}\cdot\text{m}^{-2}$). However, the temperature, obtained by fitting the time resolved spectra (Fig. 6), is high already at the onset of the laser pulse. This effect can be attributed to a broad particle size distribution. From (4) follows that small particulates are heated faster and as the emission intensity of a blackbody is inversely proportional to $\exp(h\nu/kT) - 1$ only the hottest particulates have a significant contribution to the observed spectrum, even if their emitting surface is small. Therefore the emission just at the onset of the laser pulse originates from small particulates, which are heated very fast to the decomposition temperature. The maximum emission intensity corresponds to particulates with a larger emitting surface and it is reached after the larger clusters have been heated.

If we assume the integrated emissivity (ϵ) to be 1 (perfect blackbody), the radiative losses ($4\pi a^2 \sigma T^4$) at the highest measured temperature of 3700 K are 10^{-4} W , which is a small fraction of the laser power absorbed by the $1\text{ }\mu\text{m}$ particulate ($\pi a^2 \cdot (1 - R)I \sim 10^{-3}\text{ W}$ for 0.3 J/pulse). Thus after the particulates are heated the major part of the laser energy is

Fig. 6. The time resolved spectral temperature (circles), obtained by fitting blackbody curves to the laser-induced emission and the laser-induced emission intensities (triangles), obtained by integrating the OMA spectra over wavelength ($\sim 400\text{--}700$ nm). The laser energy is 0.25 J/pulse , the gate width of the OMA is $20\text{ }\mu\text{s}$. The deviation in temperature has been estimated by fitting several parts of the spectra (e.g. $300\text{--}700$ nm, $400\text{--}800$ nm).

Fig. 7. The laser-induced emission intensity, obtained from the OMA spectra as a function of the laser energy for several CCl_2F_2 percentages (at standard pressure, power and flow): $5\%\text{ CCl}_2\text{F}_2$, collected 1 cm above the RF electrode (5 times enlarged, circles), $1\%\text{ CCl}_2\text{F}_2$, collected 1 cm above the RF electrode (triangles) and $1\%\text{ CCl}_2\text{F}_2$, collected 2.5 cm above the RF electrode (100 times enlarged, diamonds). At $1\%\text{ CCl}_2\text{F}_2$ in the plasma large structures ($\geq 10\text{ }\mu\text{m}$) have been observed 1 cm above the RF electrode, while 2.5 cm above the RF electrode only very small clusters ($\leq 100\text{ nm}$) are present, as estimated from the forward and perpendicular scattering intensities using a He-Ne laser. The laser energy is 0.3 J/pulse , the gate width of the OMA is $200\text{ }\mu\text{s}$, the gate is coincident with the onset of the laser pulse.

consumed in the destruction process. In case of a teflon-like structure this process is thermal decomposition (presumably into CF_2 fragments) rather than evaporation. We estimate the decomposition energy by the energy needed to break a C-C bond (about 3.5 eV). Substituting this into (5) we obtain a decomposition rate of $da/dt \sim 10^{-2}\text{ m/s}$ at $10^9\text{ W}\cdot\text{m}^{-2}$. This implies that a particulate of $1\text{ }\mu\text{m}$ is decomposed within about $100\text{ }\mu\text{s}$. Consequently, most particulates within the laser volume are destroyed during the laser pulse, especially at higher laser energies.

The total emission intensity has been determined as a function of the laser energy for three different plasma conditions (see Fig. 7). A threshold energy of about 0.03 J/pulse ($6 \cdot 10^7\text{ W}\cdot\text{m}^{-2}$) is needed for the initiation of this laser-induced process. This energy corresponds with heating a $1\text{ }\mu\text{m}$ particulate to about $2000\text{--}3000\text{ K}$ (see (4)) during the whole laser pulse. Indeed, Fig. 8 shows that at the threshold laser intensity

Fig. 8. The spectral temperature as a function of the laser energy for several CCl_2F_2 percentages (as in Fig. 6): 5% CCl_2F_2 , collected 1 cm above the RF electrode (circles), 1% CCl_2F_2 , collected 1 cm above the RF electrode (triangles) and 1% CCl_2F_2 , collected 2.5 cm above the RF electrode (diamonds).

the particulate temperature is significantly lower (2700 K) than the "normal" decomposition temperature (3500 K). Both the laser-induced emission intensity and the temperature saturate at high laser intensities. The saturation temperatures are always close to 3500 K and independent of the conditions in which the particulates were grown. Obviously, this is the decomposition temperature of the cluster material.

From the last observation it follows that a laser with an extremely high intensity is not necessarily efficient in inducing an intense blackbody like radiation from particulates. Contrarywise, too high an intensity causes a fast particulate destruction, which limits the duration of the emission, without increasing its intensity ($T = \text{const}$). This is in agreement with the experiments with a Q -switched laser (pulse duration ~ 20 ns). Typically we have observed a 100–1000 times lower emission at the same laser energy. In this case the surface heating and destruction are extremely fast, but the irradiated intensity is limited due to the fact that the destruction process occurs at a constant temperature. Small particulates ($a \sim \kappa^{-1}$) are destroyed immediately, while for larger particulates ($a \gg \kappa^{-1}$) only the outer layer is heated by the laser, as internal thermal conduction is not fast enough to provide a homogeneous temperature profile within a particulate. After the laser pulse the remaining particulates will cool down by internal heat conduction within less than 1 μs . Therefore the upper limit for the duration of this event is 1 μs . In comparison with the emission from a 100 μs long pulse, it is clear that the total emission is much lower. Thus in order to obtain the optimal detection limit for the technique described in this work the laser pulse should be long and the absorbed intensity should, after heating the particulates, only balance the radiative losses at a temperature just below 3500 K to prevent the particulate destruction.

V. CONCLUSION

Laser interaction with particulates formed in an $\text{Ar}/\text{CCl}_2\text{F}_2$ RF plasma results in intense white light emission. The time resolved spectra show blackbody like curves with temperatures varying between 3500 K at the start of the laser pulse and 2500 K in the extinction phase. The time evolution of the total

intensity shows a maximum at about 30 μs after the onset of the laser pulse which agrees with the calculated heating time for a 1 μm particulate. A corresponding delay for the temperature cannot be observed due to an inhomogeneous particulate size distribution. The typical particulate temperature of about 3500 K has been found in all dusty plasmas studied. The temperature and the emission intensity saturate with the laser intensity, which suggest particulate decomposition at this saturation temperature. As the absorption of laser light by small particulates depends less strongly on the size ($\sim a^2 - a^3$) than scattering ($\sim a^6$), various techniques involving laser-induced radiative processes can be more sensitive for the detection of small particulates than the commonly used light scattering.

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