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On the diagnostics of growing powder particles in a process plasma

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Introduction

Powders which are produced or modifed by using plasma technology have interesting and useful properties, e.g. very small sizes (nanometer to micrometer range), uniform size distribution, and chemical activity. Size, structure and composition can be tailored to the specific requirements, dependent on the desired application (Bouchoule 1999, Stoffels et.al. 2001, Kersten et.al. 2001).

An approach for coating of externally injected particles has been demonstrated in (Kersten et.al. 1998), where an argon rf-plasma was employed to charge and confine particles, while a metal coating has been performed by means of a separate dc-magneteron sputter source. In the present study we perform the synthesis of small carbon particles as well as the deposition of thin amorphous carbon (a-C:H) films onto SiO_2 grains (~1µm) in a methane or acetylene process plasma, respectively. Already after a very short process duration, the laser scattering increases remarkably due to the simultaneous formation of dust in the course of a-C:H deposition. After examination of the collected particles by electron microscopy (SEM) one can observe a rather small amount of large coated SiO_2 grains and a huge amount of small carbon dust particles (~100nm). Surprisingly, the carbon dust particles show almost the same size. In order to quantify the consumption of the precursor gas (CH_4 or C_2H_2, respectively) and to determine the dominant species for film deposition on SiO_2 grains and C-dust formation the precursor molecules have been monitored by IR laser diode absorption spectroscopy (Röpcke et.al. 2000). Furthermore, the particle growth has been observed by recording the intensity of the laser light signal through the particle cloud.

Experimental

In comparison to the particle coating process by magnetron sputtering [Kersten et.a. 1998], we perform the deposition of thin amorphous carbon (a-C:H) films onto SiO_2 grains (~1µm) in a acetylene process plasma. The experiments have been carried out in a reactor PULVA1 which is schematically drawn in Fig.1. The rf-electrode as well as different diagnostics (video, TDLAS) are mounted in a spherically shaped vessel. Typical discharge conditions are as follows: power 5 … 100W, pressure 1 … 10Pa, gas composition Ar : C_2H_4 0 … 5.

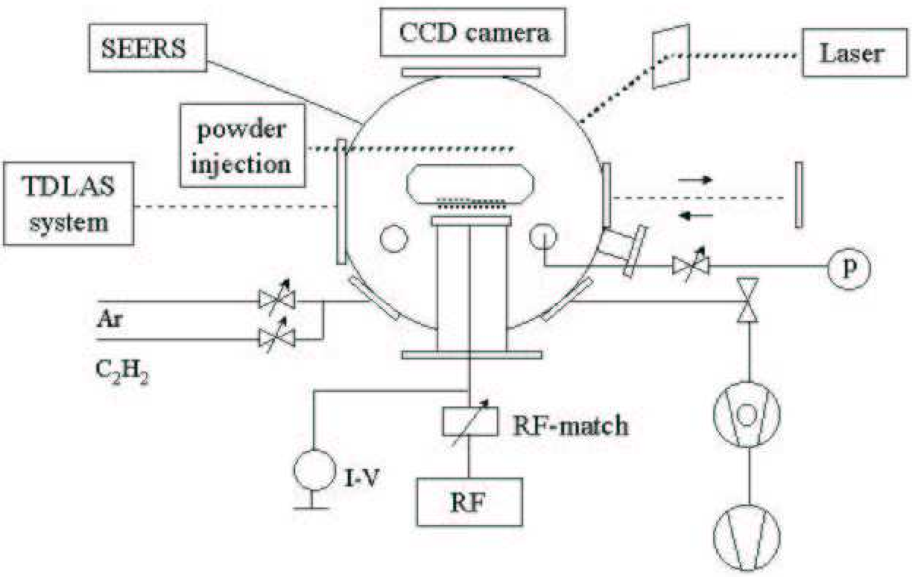

Fig.1: Scheme of the experimental set-up PULVA1.

Results and Discussion

Under relevant experimental conditions not only a- C:H deposition onto the externally injected SiO_2 particles but also generation of small carbon dust particles occur. After examination of the collected particles by electron microscopy (SEM) one can observe a rather small amount of large coated SiO_2 grains and a huge amount of small carbon dust particles (~100nm). Surprisingly, the carbon dust particles show almost the same size and form clusters, see Fig.2.

In order to quantify the consumption of the precursor gas for film deposition onto SiO_2 grains and C-dust formation the acetylene molecules have been monitored by IR laser diode absorption spectroscopy (TDLAS) (Röpcke et.al. 2000). Already after a very short process duration (about 20s) the TDLAS absorption decreases which indicates a fast and efficient decomposition of the C_2H_2 molecules (Fig.3). The dissociated radicals, mainly C_2H, are the precursors for the particle formation which starts spontaneously in an Ar/C_2H_2 plasma. Similar observations have been made by (Hong et.al. 2002). The small particles are already visible in the laser light after a short time.

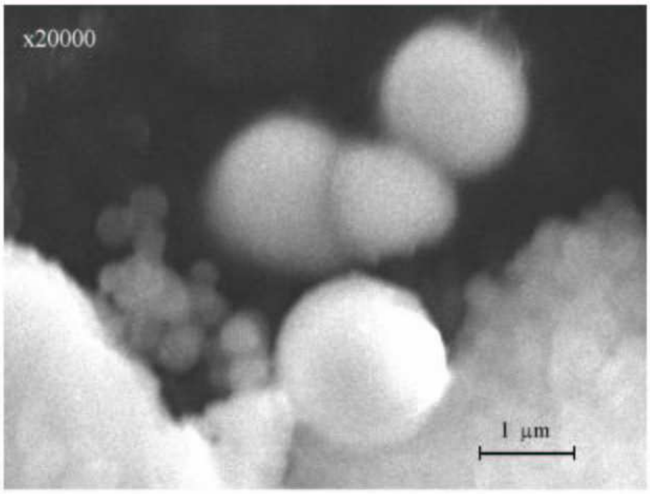

Fig.2: a-C:H coated SiO_2 particles (middle) and synthesized small carbon particles which form large clusters (left and right).

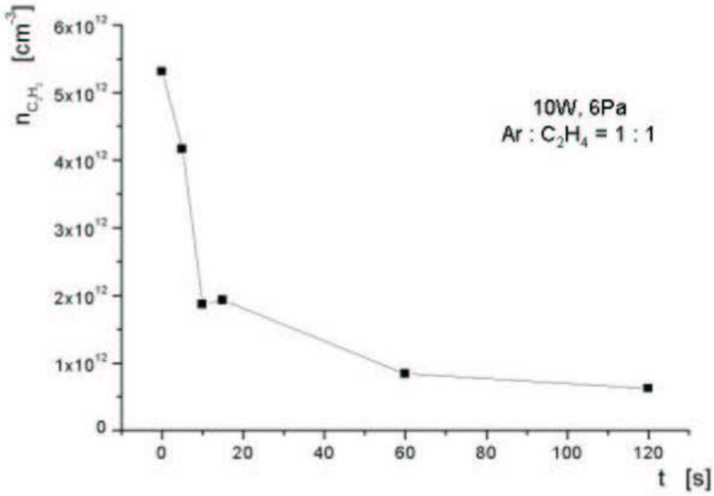

Fig.3: Decrease of the C_2H_2 absorption by decomposition of the precursor gas.

Simultaneosly, the laser light intensity through the plasma drops remarkably due to the formation of carbon dust in the course of a-C:H deposition (Fig.4). After about 100s an oscillation of the laser light intensity can be observed due to the dynamics of carbn dust formation. The growth mechanism is accompanied by the action of the various forces (particle drop due to gravitation, particle flow due to ion drag etc.) and the development of voids. This behaviour results in alternating dust-free zones and dust zones. After switchingoff the acethylene supply, the light intensity increases and becomes stable again due to vanishing particle density – as it can be seen in Fig.5.

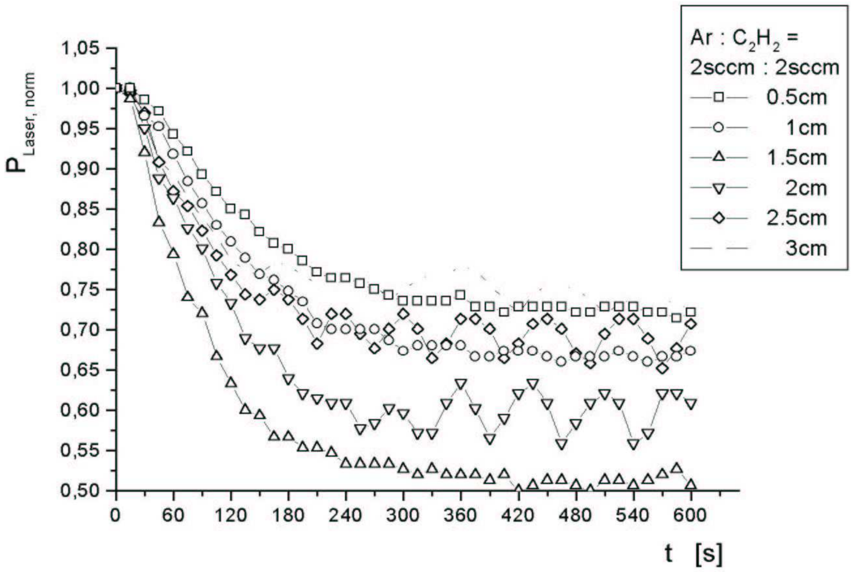

Fig.4: Decrease and oscillation of the laser light due to the growth and thedynamics of the particles at different hights above the rf-electrode.

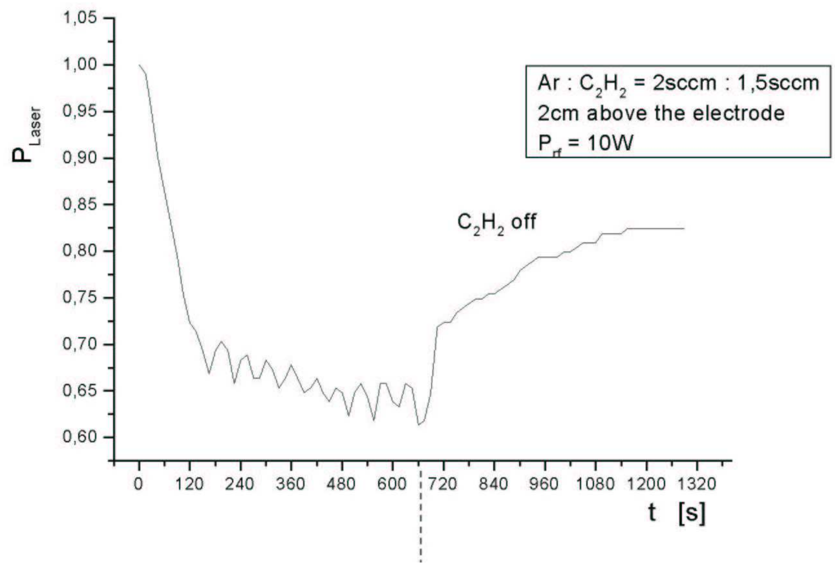

Fig.5: Vanishing particle density after stop of C_2H_2 supply.

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