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Comparative study of 3.4 micron band features from carbon dust analogues obtained in pulsed plasmas

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Abstract. Syntheses of carbon dust analogues are key experiments in laboratory astrophysics, as an approach to study some chemical and topological features of interplanetary and interstellar carbon dust. We report a comparative experimental study for carbon dust analogues obtained in (1) an atmospheric pressure dielectric barrier discharge (DBD), fed with helium – saturated hydrocarbons gas mixtures, (2) a low pressure radio frequency (RF) discharge and (3) a pulsed laser deposition (PLD) experiment with a Nd:YAG laser and a graphite target. The aliphatic –C–H stretching band, known as the 3.4 micron feature, as well as the CH_2/CH_3 ratio, the H/C ratio value and the physical appearance at microscopic scale, show a variability that is influenced by the synthesis method and the experimental parameters of each specific technique.

Keywords. infrared: ISM

1. Introduction

Dust is ubiquitous in many astrophysical environments, such as interstellar medium, star and planet forming regions, near planetary atmospheres or the coma of comets. It plays many different roles in these environments and its formation, as well as the evolution in high energy fields of radiation or particles, still needs to be understood. Thus using Earth based experiments, various synthesis methods or post processing techniques are explored in order to match the astrophysical observations or to obtain information on dust processing in space (Maté *et al.* 2016). Many experiments proved their potential to grow aliphatic, aromatic or mixed aliphatic/aromatic carbon structures, which exhibits IR absorption features similar to infrared absorption spectroscopy data from space (Pino *et al.* 2008). Some of the experimental approaches are: condensation, physical vapour deposition, plasma deposition, combustion and pyrolysis, pulsed laser deposition. Depending on the operational parameters, of which gas mixture and temperature have critical significance, various allotropic forms of carbon can be deposited, with variable hydrogen content and different physico-chemical properties.

Recently, our group reported a new experiment for low temperature deposition of carbon dust analogs, in form of the both non-aromatic thin films and 'fluffy' aggregates (Hodoroaba *et al.* 2018). We discuss in this paper the results on a comparative analysis of carbon dust analogues, exploring various pulsing regimes of the plasma, from Hz range to MHz range, in order to select optimal deposition parameters. Morphological and IR spectral features are emphasised and the astrophysical implications are examined.

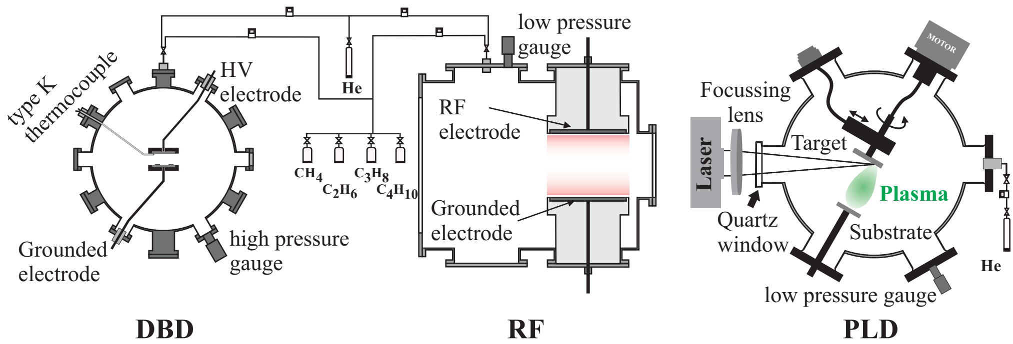

Figure 1. Schematic representation of the plasma sources used for laboratory astrophysics experiments on the synthesis of carbon dust.

2. Experimental set-up and physico-chemical investigations

Carbon dust analogues were deposited using three types of pulsed plasmas (Fig. 1). Dielectric Barrier Discharge (DBD) works at near atmospheric pressure (600 Torr) in He - Hydrocarbons (15%) and is driven by high voltage pulses (6 kV, 500 ns, 1 kHz). Radio Frequency Discharge (RF) works at low pressure (0.5 Torr) in He - Hydrocarbons (15%) gas mixture, at constant power of 35 W. Pulsed Laser Deposition (PLD) works at low pressure (0.75 Torr) in He quenching gas, using a carbon target and a Nd:YAG laser (third harmonic, 355 nm, 5 ns, 10 Hz). Deposition time was 6 hours for DBD, 30 min for RF and 20 min for PLD.

The DBD experimental setup consists of a stainless steel chamber housing two rectangular aluminium electrodes (7 cm length, 5 cm width) attached to glass dielectric plates (1 mm thickness) placed in plane-parallel geometry. In the 5 mm discharge gap the flexible graphite substrates are placed on the ground electrode. Gas temperature is monitored using electrical and spectral methods, as well as the growth yield of carbon dust.

The RF experimental setup consists of two circular stainless steel electrodes (7 cm diameter), at 3 cm discharge gap, enclosed in a stainless steel chamber. The flexible graphite substrates are placed on the grounded electrode.

The PLD experimental setup consists of a stainless steel chamber containing a pyrolytic carbon rotating target and the substrate (i.e. flexible graphite) holder, located at 5 cm distance from the target and ensuring a 1 cm in diameter deposition area.

3. Scanning electron microscopy

Scanning electron microscopy helped reveal significant differences of microscopic scale morphology between the dust analogues, without any sample metallization (Fig. 2). The product obtained in DBD has a hierarchical ‘fluffy’ structure, the individual flakes exhibiting micrometer-scale dimensions. The ‘fluffy’ clusters formed by these flakes through aggregation can exceed 100 μm and are unevenly distributed on the substrate. Larger dimensions of these clusters are achieved towards the edge of the substrate, where the likelihood of streamer formation increases, favored by high local electric field.

The RF product however, exhibits a film–like morphology. This is coherent with visual observations made during deposition: a thin layer of film formed on the power electrode is fragmented into small pieces that are kept levitating by the electric field until they are heavy enough to fall onto the substrate. These thin film fragments have varying dimensions, the length reaching values from tens of micrometers to almost one millimeter; the width is hard to asses do to their tendency to twist and entangle.

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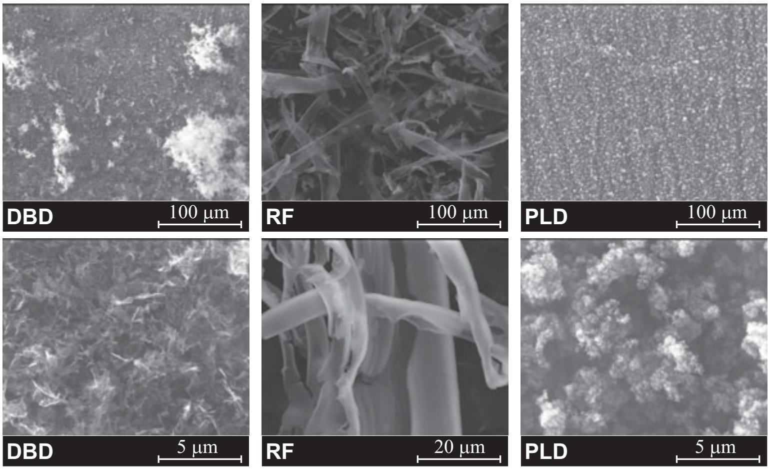

Figure 2. Typical Scanning Electron Microscopy (SEM) pictures of carbon dust products.

The product obtained via PLD method shows an even distribution on the substrate surface. A hierarchical organisation is again observed, with a cauliflower–like growth pattern. Micrometric aggregates are formed by sub–micron spherical grains.

4. Fourier transform infrared spectroscopy

The 3.4 μm band (between 3000 and 2800 cm^{-1}), is present in the spectra of all products and is assigned to aliphatic -CH vibration modes (Figure 3). From the detailed study of this band we approximated the CH_2/CH_3 ratio to 2.2 in DBD and PLD products and 1.4 in RF, using the method detailed by Chiar *et al.* (2013) & Hodoroaba *et al.* (2018). None of the aromatic carbon signatures are present in any of the three infrared spectra (i.e. 3050 cm^{-1} and 1590 cm^{-1}).

The product obtained in DBD also presents signatures for sp^2 -C=C stretching (1640 cm^{-1}), sp^2 -C=O stretching (1700 cm^{-1}), sp^2 R-O-CH_3 stretching (1500 cm^{-1}), sp^3 -CH_2 scissor mode (1460 cm^{-1}), sp^3 -CH_3 symmetric bending (1375 cm^{-1}) and out of plane sp^2 -C=C-H signatures (between 1000 and 800 cm^{-1}) as shown in Figure 3.

The thin film fragments obtained in RF present the signatures characteristic to out of plane sp^2 -C=C-H signatures (between 1000 and 800 cm^{-1}), sp^3 -CH_2 scissor mode (1460 cm^{-1}) and sp^3 -CH_3 symmetric bending (1375 cm^{-1}). The noise in the 1900 to 1500 cm^{-1} region, makes difficult to identify supplementary carbon signatures (see Fig. 3).

The PLD product has two specific signatures for hydroxyl (1720 cm^{-1}) and secondary alcohols -C-OH (1110 cm^{-1}) as illustrated in Figure 3. These signatures correlate strongly with the X-ray photoelectron spectroscopy measurements that indicates a high oxygen percentage (around 13%) as compared to the other two methods that have oxygen levels under 5%. Additionally, the noise from 1900 to 1300 cm^{-1}, make difficult the identification of the other carbon signatures in this range.

5. Astrophysical implications

Morphological analysis of dust using optical, SEM and atomic force microscopy was restricted so far to interplanetary, cometary and asteroidal dust. The results share many common features, the most important being the micron and sub-micron hierarchical organisation and the 'fluffy' appearance, with many pores and irregular surfaces. It can be observed from Fig. 2 that the DBD and PLD dust analogues share these features.

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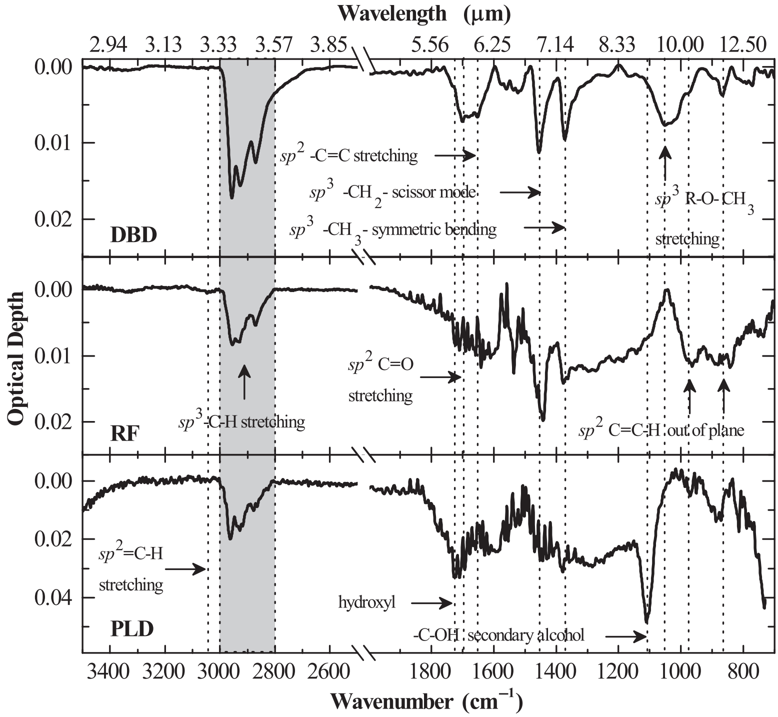

Figure 3. FTIR spectra of carbon dust carbon dust analogues, with emphasis on the 3.4 μm band.

On the other hand, spectroscopic features of interstellar dust are measured using infrared observatories for a few decades now, in a limited range of IR spectrum. Thus, it is possible to compare those observations with IR spectra of carbon dust analogues. Indeed, the spectroscopic features of dust analogues obtained by pulsed plasmas show similarities with astronomical sources presenting high column density of sp^3 hybridized carbon atoms (Sgr A*, IRAS 08572+3915, IRAS 19254âĂŞ7245, NGC 1068 and NGC 5506). Moreover, the accurate analysis of the 3.4 μm bands features allows the calculation of the CH_2/CH_3 ratio (about 2.2 maximum for our analogues) and the H/C value (close to 1), these data being susceptible to modification by post-synthesis processing using radicals and energetic ions or UV radiation. The 'fluffy' carbon aggregates deposited in DBD plasma combines both, spectroscopic and morphological features of carbon cosmic dust.

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