

# Optical investigations of tungsten dust particles in a DC glow discharge

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## Abstract

The dynamics of a tungsten nanoparticle cloud grown from the sputtering of a tungsten cathode in DC argon glow discharges is reported. The dust particle cloud was imaged by light scattering of a vertical laser sheet going through the plasma. The dust particle size distributions were measured in-situ by light extinction spectrometry and compared to those obtained from electron microscopy measurements of the particles collected at the anode centre. It was observed that particles are pushed towards the anode and the plasma edges. Discrepancies in the particle size distributions were explained by the spatial inhomogeneities of the dust cloud.

## Introduction

Tungsten nanoparticles can be grown from the sputtering of a tungsten cathode in DC argon glow discharges [1]. In this article, particle size distributions (PSDs) and the dynamics of the nanoparticle cloud were investigated. The dust cloud was imaged by scattering of a vertical laser sheet passing through the plasma. PSDs were measured in-situ using light extinction spectrometry (LES) and compared to PSDs obtained from scanning electron microscopy (SEM) measurements of the particle collected at the anode centre. It was observed that during the discharge, particles moved towards the anode and the plasma periphery. Moreover, LES-PSDs and SEM-PSDs did not necessarily match especially for long discharge durations. These PSD discrepancies were explained by the spatial inhomogeneities of the dust cloud.

## Experimental set-up

The electrode assembly was contained in a cylindrical vacuum chamber of 30 cm diameter and 40 cm length. DC glow discharges in argon at a static pressure of 0.6 mbar were initiated between two parallel electrodes : a tungsten cathode of 9.9 cm diameter and a grounded stainless steel anode (see Fig.1). The electrodes were separated by a distance of 10 cm by two half glass cylinders. The anode disc had a hole at the centre through which the tungsten nanoparticles produced during the discharge were collected. A regulated power supply was used to bias the cathode. The discharge current density was kept at a constant value ( $0.53 \text{ mA} \cdot \text{cm}^2$ ). The cathode voltage was  $V_d \sim -600 \text{ V}$  giving a total flux of impinging particles  $\Phi_t \sim 7 \cdot 10^{15} \text{ cm}^2/\text{s}$ , a

sputtering yield was  $\Upsilon \sim 3.8\%$  and a flux of sputtered tungsten atom  $\Phi_W \sim 7 \cdot 10^{14} \text{ cm}^2/\text{s}$ . These parameters were favourable to nanoparticle formation by homogeneous nucleation from the sputtered atoms [1]. The discharge durations were up to 600 s.

In order to study the dynamics of the dust particle cloud, a 2 cm vertical laser sheet (wavelength  $\lambda = 532 \text{ nm}$ ) was going through the plasma. Its vertical position between the electrode could be adjust. The scattered light at  $90^\circ$  was recorded with CCD camera at 1 frame per second (integration time 0.5 s). The low frame rate was necessary to detect small particles and/or thin dust particle clouds.

PSDs and particle concentration were obtained in-situ by LES. A collimated and polychromatic beam (1 mm in diameter) with spectral intensity  $I_0(\lambda_i)$  and wavelengths  $\lambda_i$  was passing through the dust cloud and the transmitted spectral intensity  $I(\lambda_i)$  was collected and directed towards a spectrometer. Extinction coefficients at different wavelengths were derived. One could then extract the PSD and concentration of the particle assuming the particle had the complex refractive index of pure tungsten and a mass fraction of 0.94. One should note that PSDs and concentration measured by LES are integrated over the discharge diameter. A detailed description of this technique is given in Ref.[2]. LES-PSDs were then compared to the SEM-PSDs of dust particles collected at the anode centre during the discharge (samples were exposed to the plasma for a given duration; for example from 30 s to 120 s after plasma start).

### Laser light scattering

The laser sheet was positioned to visualise the dust cloud just above the anode. As it can be seen in Fig.2(a), dust particles could be detected from  $t \sim 180 \text{ s}$ . Dust particles were detected faster in the upper part (away from the anode). At first, the dust particle cloud looked more or less homogeneous (see Fig.2, snapshot at 200 s). From  $t \sim 250 \text{ s}$ , dust particle in the highest part of the laser sheet were pushed toward the side and the bottom of the discharge (see Fig.2(b) and, snapshot at 270 s) and no particles were further detected in the remaining time of the experiment. This phenomenon was also observed upon approaching the anode but with an increasing time delay. It should however be noted that at  $\sim 1 \text{ cm}$  above the anode, a thin particle cloud was detected and was separated from the primary dust particle cloud (see Fig.2, snapshot at 400 s). After  $\sim 500 \text{ s}$ , the primary dust cloud was no longer visible above the anode. Nevertheless a dim cloud was observed for  $\sim 50 \text{ s}$  which could correspond to a new generation of dust particles.

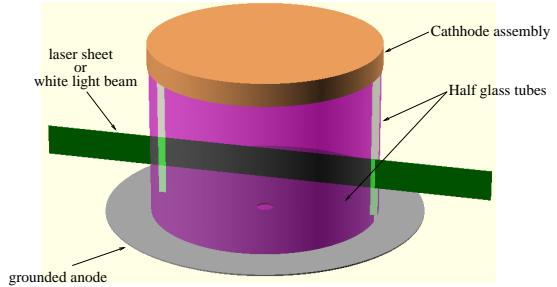


Figure 1: Sketch of the experimental set-up.

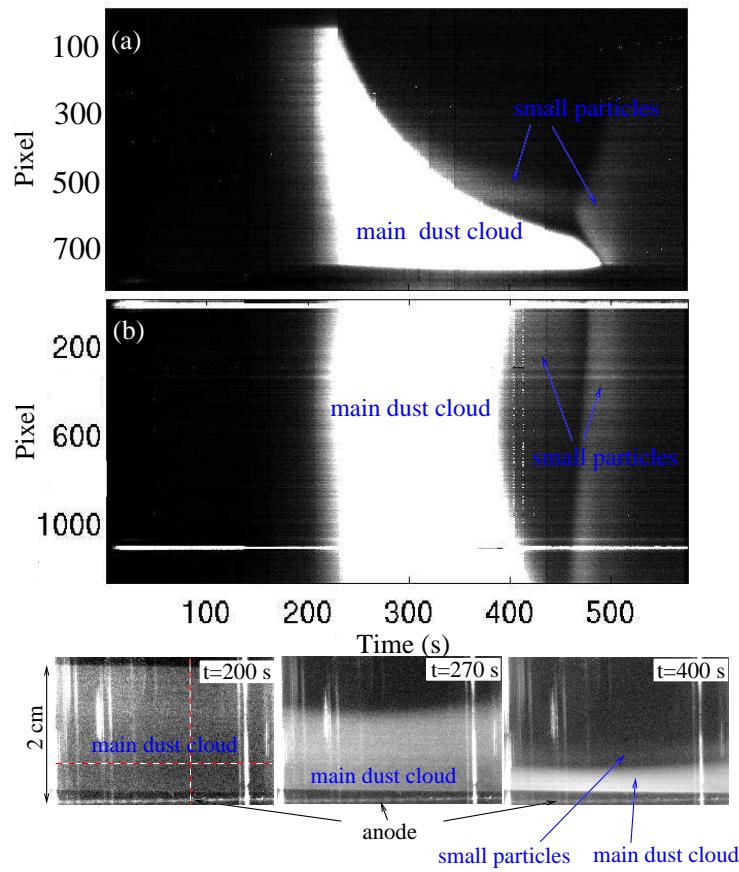


Figure 2: Video imaging of the dust cloud above the anode. Top: (a) Evolution of a column profile as a function of time. (b) Evolution of a line profile as a function of time. Bottom: snapshot of the video at different times. Vertical and horizontal red dashed lines indicate the line and column chosen for the above profiles. Vertical bright lines are reflections on the glass tube.

### Light extinction spectrometry

The white light beam was positioned 2.8 cm above the anode. In order to compare LES-PSDs with SEM-PSDs, LES-PSDs were time integrated over a time interval corresponding to the exposure time of the sample on which the particles were collected. In Fig.3(a), the SEM-PSD of the particle collected between 30 s and 120 s after plasma start along with the corresponding time integrated LES-PSD are presented. As can be seen they are in good agreement with each other. This results was not surprising because, as it was observed by laser light scattering, the dust particle cloud looked quite homogeneous in the early stage of the discharge. Consequently the SEM-PSD have no reason to deviate strongly from the LES-PSD.

In Fig.3(b), the SEM-PSD of dust particles collected between 120 s and 200 s after plasma start along with the corresponding time integrated LES-PSD are presented. As it can be seen, PSDs were very different and only the larger size LES-PSD tail covered the SEM-PSD. In the previous section, it was shown that the primary dust particle cloud moved toward the anode

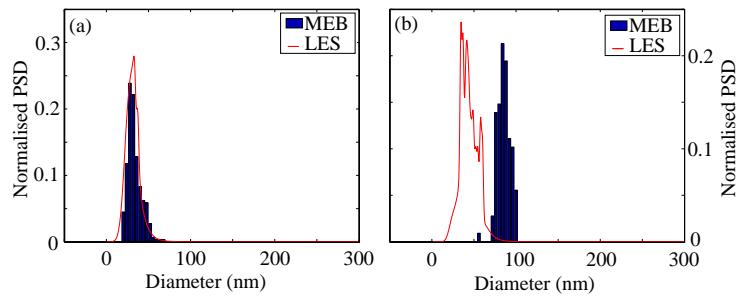


Figure 3: SEM-PSDs and time integrated LES-PSDs 2.8 cm above the anode. (a) Time interval: 30 s to 120 s. (b) Time interval: 120 s to 200 s.

and the plasma periphery for long discharge durations. Moreover, the upper part of the cloud (closer to the cathode) were evacuated sooner than the lower part. Consequently, the LES light beam passed through a inhomogeneous dust particle cloud and the LES-PSD reflected all the particles along the diameter of the discharge. On the contrary, SEM-PSD rendered only the particles falling at the anode centre and did not represent the full extent of the discharge PSD. By light scattering a dim cloud was seen above the anode centre for long discharge durations. In LES-PSDs large amount of small  $\sim 30$  nm particles were detected compared to the SEM-PSDs. AS LES-PSDs reflected the particle along a diameter, one can suppose that the small particles are most probably close to the axis of the discharge while the big particles are at the periphery.

### Discussion and conclusion

In this article, PSDs and dynamics of a tungsten nanoparticle cloud grown from the sputtering of a tungsten cathode in DC argon glow discharges were investigated. We have shown that the nanoparticle cloud moved towards the anode and plasma periphery. Secondary thin dust particle clouds were also observed and were well separated from the primary cloud. This results in a non uniform spatial particle distribution in the discharge. Consequently, LES-PSDs did not necessary match SEM-PSDs of dust particles collected at the anode centre especially for long discharge durations. Laser light scattering measurements confirmed strong spatial inhomogeneities along the LES white light beam path.

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### References

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