Tungsten nanoparticle formation in plasma discharges

Kishor Kumar K [(*)1], L. Couedel[1], C. Arnas[1] and F. Onofri [2]

[1] Laboratore PIIM, CNRS-Université d'Aix-Marseille, Campus de S[t] Jérôme, 13397 Marseille, France
[2] Laboratoire IUSTI, Technopôle de Château-Gombert, 5 rue Enrico Fermi, 13453 Marseille, France
[(*)] Kishor-Kumar.Kalathiparambil@univ-amu.fr

The study aims to understand the conditions leading to the formation of nanometer sized particles in a DC discharge with parallel electrodes configuration in which Tungsten is used as the cathode material. The Tungsten nanoparticles are characterised using electron microscopy and light scattering. Results indicate that the size distribution evolution are correlated with the discharge parameter evolution.

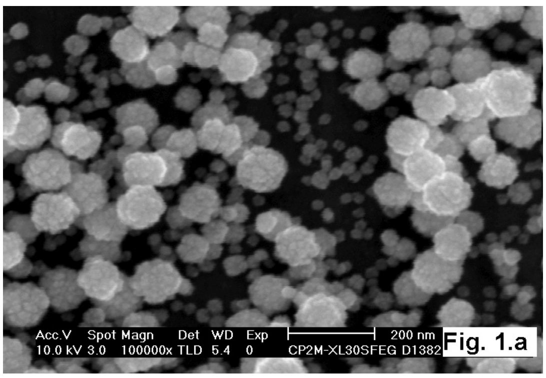
In a DC discharge, energetic ion bombardment of the cathode surface results in sputtering of the cathode material. Nanometer sized dust particles, which have a spherical geometry are produced from the sputtered atoms during the discharge by homogeneous nucleation process [1-2].

The experimental setup consisted of stainless steel vacuum chamber containing the parallel plate configuration of electrodes. A regulated DC power supply was used to create a discharge between tungsten disc which was used as the cathode and a grounded stainless steel disc placed horizontally below as the anode. During the discharge, Argon pressure in the chamber was 0.6 mbar. The discharge current density was kept at a constant value (0.5 mA cm^{-2}) and the discharge voltage was the free parameter whose time evolution was acquired during the discharge. In these conditions, the sputtered atoms leaving the cathode surface have ~ 12 eV mean energy. They thermalize with argon atoms at a distance of ~ 1 cm from the cathode. The anode disc had a hole at the center where the substrates (stainless steel foils) were placed to collect the tungsten dust. During the study, discharges of various durations (3s – 500s) were produced.

The collected dust particles were analysed using Scanning Electron Microscopy (SEM) and High Resolution - Transmission Electron Microscopy (HR-TEM). The SEM micrographs were used for measuring the size of the particles and to study their surface morphology. HR- TEM analysis gave information regarding the structure of the particles. SEM and TEM analysis showed nanometer sized spherical particles on the substrate. Figure 1.a shows the typical spherical nanoparticles of tungsten.

The size histograms fitted well to log-normal size distribution function. The parameters of the fitted function, namely the mean size and the geometrical standard deviation, were evaluated and a growth law was formuated relating the mean size and the duration of the discharge.

From the HR-TEM micrographs, it was observed that the smallest particle units consit of tungsten cristallites of ~ 2 nm.



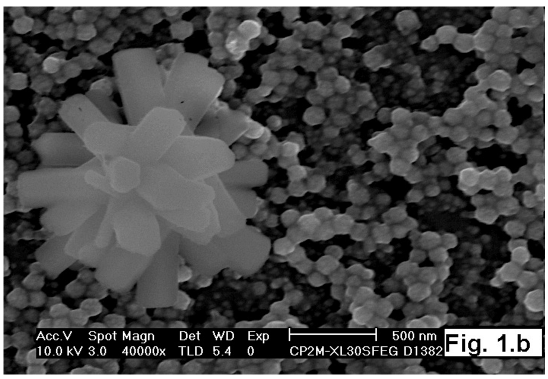


Fig. 1 .a: SEM micrograph showing the particles on the substrate after a discharge of 500 s duration. Particles are spherical in shape with rough surface. Fig. 1.b, SEM micrograph showing the particles on the substrate after a discharge of 500 s duration in which arcing was produced.

The analysis also revealed the presence of an other type of particle due to arcing during the plasma discharge such as the particle on the left side of Figure 1.b.

Light scattering method [3] was also used to evaluate the size parameters and shapes of the particles in the plasma discharge.

Optical Emission Spectroscopy (OES) study of the discharge plasma was carried out and the intensity of the emission lines of the prominent species – Ar I, Ar II, W I, WII were measured. Their time evoultion were correlated with the evolution of the discharge voltage and hence with the growth law of the dust particles.

Details of the characterisation experiments, the results from the size distribution measurements and light scattering experiments, the OES studies and the correlation between the various discharge parameters with the particle growth law will be presented.

Acknowledgments

This work is supported by ANR under the project number ANR-09-BLAN-0070-01 and by Euratom/CEA association contract in the framework of FR-FCM and EFDA.

References

[1] D. Samsonov, J. Goree, J. Vac. Sci. Technol. A **17** (1999) **2835-2840**
[2] A Michau et.al., Plas. Phys. Contr. Fus. **52** (2010) **124014**
[3] F. Onofri et. al., Appl. Optics, **34** (1995) **7113–7124**