Prevention of biological surface contamination by deposition of silver nanoparticle-containing plasma $SiC_xO_yH_z$ thin films.

B. Despax[1], C. Saulou[2], M. Mercier-Bonin [2], P. Raynaud[1], S. Zanna[3], P. Marcus[3]

[1] LAPLACE Université Paul Sabatier, 118 route de Narbonne 31062 Toulouse Cedex 09, France
[2]LISPB INSA (UMR CNRS 5504) 135 Avenue de Rangueil 31077 Toulouse Cedex 04, France
[3]LPCS, UMR CNRS/ENSCP 7045, 11 rue P. et M. Curie, 75005 Paris, France

The growing interest in the dispersion of metallic particles in polymers or ceramic matrices is motivated by the prospect of developing new materials with technological applications. Recently, a large field of applications has been opened to prevent microbial colonization and biofilm formation. One possible strategy for the prevention of biofilm formation on surfaces is to coat the material with an antibacterial agent. The surface treatment can prevent bacterial adhesion to the surface or can kill bacteria as they come in contact with the surface.

The combination of silver particles as an inhibitor agent of biological colonization and silicon-based materials as an antifouling matrix seems to be of particular interest for biological and medical applications. In order to synthesize this kind of deposit, we used a process combining the silver sputtering and the plasma polymerization of hexamethyldisiloxane (HMDSO).

In gas mixtures such as argon and HMDSO, it is difficult to adjust the metal sputtering because of the large amount of organic fragments which contribute to a rapid polymer covering on the metal target. In order to control the target poisoning, we utilized a pulsed injection of HMDSO. In fact, at the same rf power input, the time duration choice of pulsed HMDSO injection adjusts the covering rate on the target and consequently the Ag sputtering[1]. The competition between Ag sputtering and plasma polymerization was monitored by optical emission spectroscopy. The silver lines (546.55 and 520.91 nm), the H_β transition (486.00 nm) and the Ar transition (549.59 nm) were easily detected and identified. The Ag lines and Hβ transition were assumed to be related respectively to the silver atom presence and the HMDSO dissociation in the plasma phase. Physicochemical analyses of deposits revealed the presence of silver-containing SiCO films. Infrared, Raman and XPS studies showed that the SiCO network was mainly constituted of Si-O bonds, Si-C bonds and C=C bonds. Moreover XPS and Energy dispersive X-ray analysis with electron probe (EDX) measured the film silver content.

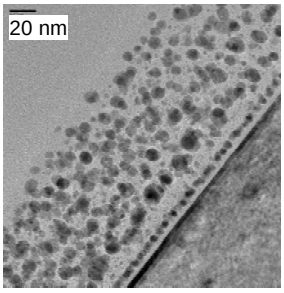

Figure 1: TEM cross section of Ag-SiCO films

Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) (Fig.1) revealed the presence of well distributed silver nano particles from the surface into the material bulk. Their shape and size distribution depend on the competition between the sputtering and the plasma polymerisation. In Fig.1, the mean particle size is around 7 nm. By decreasing the duration of HMDSO pulsed injection, the mean size of Ag grains can be varied between 4nm and 100 nm. This is mainly a consequence of the increase in silver sputtering due to the decrease in target poisoning.

Ageing tests on Ag-SiCO films in saline solution (NaCl 150 mM) during 2 weeks showed a partial oxidation of matrix and a partial release of silver from the surface and the subsurface into aqueous solution. Besides, TEM coupled with EDX revealed that the whole layer was affected by the saline solution. Indeed, the presence of chlorine was detected at the interface between the matrix and the nanoparticles in the deposit bulk.

Finally, some Ag-SiCO layers deposited on stainless steel were tested on the bacteria *Escherichia coli* ATCC 10536 as gram-negative microbial model and on the yeast *Saccharomyces cerevisiae*. The viable bacterial survival number was determined after 24h-exposure to different silver-coated surfaces. A significant drop in viable bacterial count (>5log reduction) was observed, whatever the Ag content, thus showing the high antimicrobial activity. Besides, the silver penetration were studied on *Saccharomyces cerevisiae* by TEM and EDX

1-B. Despax, P. Raynaud , Plasma Process. Polym., 4, 127, (2007)