STUDY OF THE INFLUENCE OF THE GEOMETRY OF CASTELLATION AND THE INSIDE DEPOSITED MATERIAL (CARBON FILMS, MIX OF CARBON FILMS WITH METALLIC IMPURITIES) ON THE EFFICIENCY OF CLEANING BY A PLASMA TORCH

George Dinescu et al.

Low Temperature Plasma Physics Laboratory

National Institute of Laser, Plasma and Radiation Physics, Magurele

1. Overview

The project proposes to test the efficiency of plasma torch for removal in open atmosphere of hydrogenated carbon material previously deposited by PECVD technique on flat and castellated surfaces of aluminum and/or wall like materials. The nature of the deposit is important and also the castellation geometry. The deposits of composite materials are of interest. The ultimate goal of the project is the assessment of cleaning efficiency on castellated surfaces of various geometries inside gaps of various widths, including sub-milimetric ones.

Summary of the obtained results: The model surfaces designed in the frame of the project are described by two aspects: i) the geometrical aspect related to the design and realization of surfaces with gaps of defined depth and variable distances, ii) material aspects, related to the fact that the surfaces of the gaps should be coated on the internal faces with layers of carbon and carbon metal mix. Moreover, after the cleaning experiments, in which the plasma jet coming from the torch is directed inside the gaps, the rate of layer removal should be measured, so access to the inside surfaces should be provided for a measurement technique.

The chosen approach was to build up surfaces from separated parts, by assembling them after deposition of the layers. Such approach allows also measurement of the removed material after the cleaning process, by disassembling and examination separately the pieces. Aluminium cubes have been machined as separate elements of castellated surface. By assembling the cubes gaps are created in between the lateral faces. The width of the gaps can be varied by displacing the cubes in the range 0.5-2mm, with a deepness of 23 mm.

The test layers consisting of hydrogenated carbon were deposited by Plasma Enhanced Chemical Vapor Deposition (PECVD). The test layers consisting of carbon mixed with metal (in the present case aluminium) were realized by combining PECVD with Magnetron Sputtering (MS) by alternatively exposing, under computer control, the surface to be deposited to PECVD or MS source, for predefined periods of time.

Profilometry and photography were utilized as techniques of measurement of the cleaning effect. Plasma torch was placed at 2 mm from the top surface either on the gap middle, either on the gap margin. The decrease of layer thickness due to material removal was measured, along the deepness of the gap, over a sharp transition zone, between a deposited and a non-deposited region on the cube face (the sharp transition zone was realized by partially masking the cube face during deposition).

Additional cleaning experiments were performed on flat surfaces coated with other materials, diamond-like layers (DLC) and real layers deposited on the back sides of IR mirrors previously used in JET.

2. Detailed results

2.1. Model surfaces. Geometry and material aspects

The model surfaces designed in the frame of the project are described by two aspects: i) the geometrical aspect related to the design and realization of surfaces with gaps of defined depth and variable distances, ii) material aspects, related to the fact that the surfaces of the gaps should be coated on the internal faces with layers of material similar to codeposited layers in Tokamaks.

Moreover, after the cleaning experiments, in wich the plasma jet coming from the torch is directed inside the gaps, the rate of layer removal should be measured, so access to the inside surfaces should be provided for a measurement technique. The chosen approach was to build up surfaces from separated pieces, by assembling them after deposition of the layers. Such approach allows also measurement of the removed material after the cleaning process, by dissasembling and examination separately the pieces.

Aluminium cubes have been machined as separate elements of castelated surface. The schematic with the dimensions and an image of such a cube is shown in Figure 1. By assembling two cubes a gap is created in between the lateral faces (Figure 2) . The width of the gap can be modified by displacing the cubes.

2.2. Results and discussions.

Deposition process. Parametric study

During the deposition there were used the following parameters: Ar mass flow rate Φ_{Ar} = 70sccm, C_2H_2 mass flow rate Φ_{C2H2} = 5sccm, magnetron RF power P_{RFMG}=100W, PECVD plasma source RF power P_{RFC}=160W. The optimum values for sequential cycle times: T_{CYCLE} = T_{Al} + T_{ADM} + T_C + $T_{PUMP,}$ were T_{CYCLE} value was chosen to to be 23s, were determined. Such us it was measured that T_{ADM} = T_{PUMP} = 5s assure stationary flow of C_2H_2 during the a-CH deposition step. The most important times during one cycle are deposition times, T_{Al} and T_C (T_{Al} + T_C being 13s); these times control the thickness of Al and C layers deposited during one deposition step and in such a way the concentration and distribution of Al and C inside the deposited Al/a-C:H film (for example, a small T_{Al} will conduce to a small Al concentration).

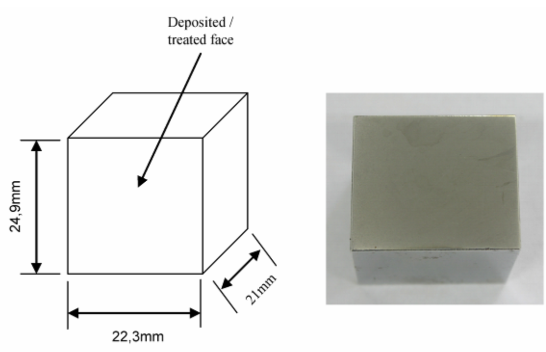


Figure 1: Schematic view and image of a cube used for delimitation of a gap

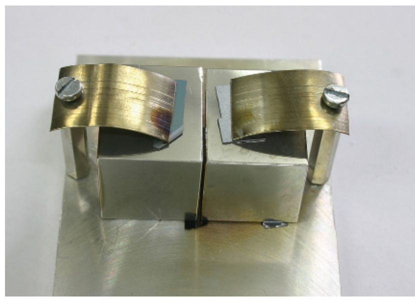


Figure 2: The assembling a two cubes creates a gap. The internal surfaces of the gap are coated with the laver to be removed

Deposition of test layers consisting of hydrogenated cabon

The test layers consisting of hydrogenated carbon deposited on the inner faces of the gap was realized by Plasma Assisted Chemical Vapor Deposition (PACVD) in a vacuum deposition setup starting from acetylene diluted in argon. The films are carbonic in their dominant composition with hydrogen bonded to complete the dangling bonds. Such hydrogenated carbon is also specific to tokamak codeposited layers, with the difference that tritium is there bonded instead of hydrogen. Neverthelees for the cleaning process is not expected to be a difference between tritium and hydrogen.

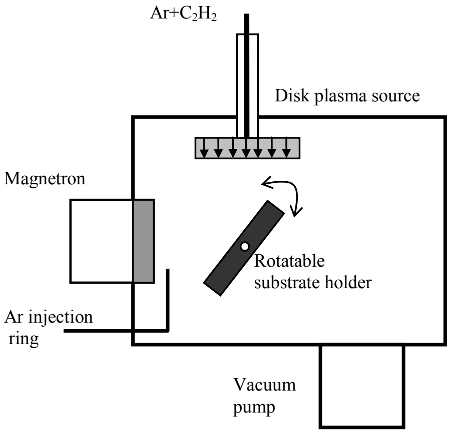


Figure3: In this experimental configuration, the substrate is successively exposed to magnetron sputtering and PECVD plasma sources

Deposition of test layers consisting of hydrogenated carbon mixed with aluminium

The test layers consisting of hydrogenated carbon mixed with aluminium were deposited on the inner faces of the gap by Plasma Assisted Chemical Vapor Deposition (PACVD) combined with Magnetron Sputtering (MS). In this case on the same deposition chamber have been placed two plasma sources, mounted at 90 degrees, like it is presented in Figure 3. The substrate is placed on a rotatable holder. The mixed layer is obtained by succesively exposing (under a computer control) the substrate to each of the two sources. The mixed film composition (metal in amorphous carbon) is varied by changing the exposure time to each source.

Experiments of cleaning

Profilometry was chosen as technique of measurement of the cleaning effect. With profilometry the decrease of layer thickness due to material removal can be measured, over a sharp transition zone, between a deposited and a non-deposited region on the cube face. The

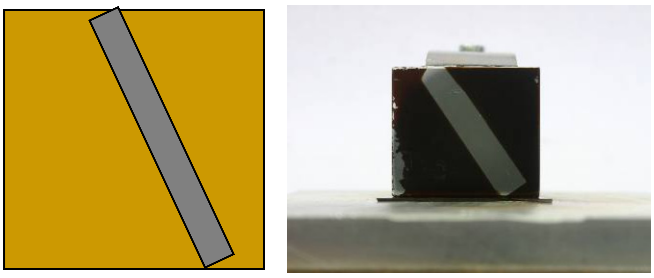


Figure 4: a) the schematic of the face of the cube with the deposited and the non-deposited zones , b) image of a cube face with deposited and non-deposited zones

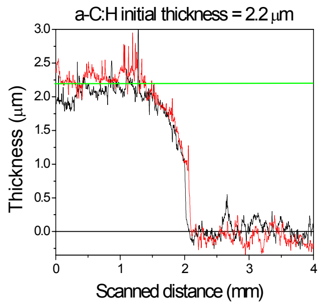


Figure 5: The profile of the transition zone (y axis is layer thickness and x axis is the distance scanned by the profilometer tip)

sharp transition zone was realized by partially masking the cube face during deposition as shown in Figure 4. A schematic view and an image of the surface limiting the gap, after deposition through the mask is shown in Figure 4a and b. A profile of the transition zone, as given by profilometry is shown in Figure 5

From the profile the thickness of the deposited layer was maesured, it is about 2.2 μm

Cleaning experiments were realized by positioning the plasma torch above the gap, as shown in Figure 6a. In Figure 5 b is shown an image recorded during the cleaning procedure.

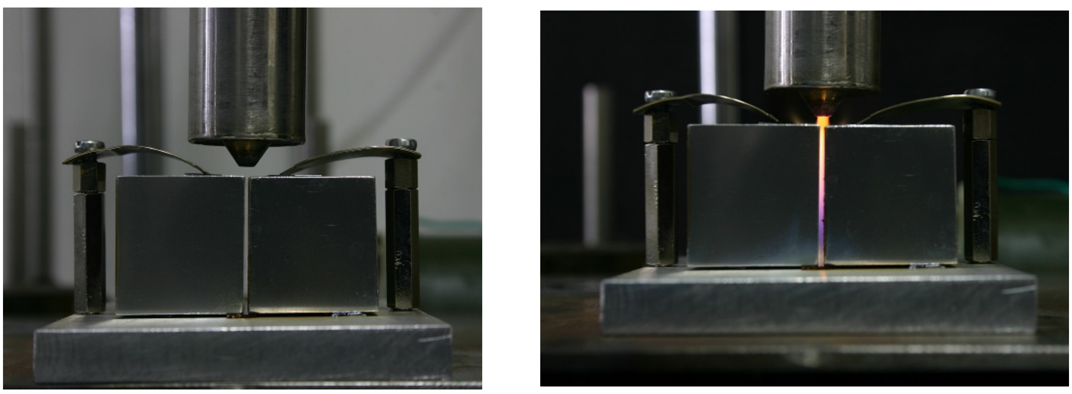


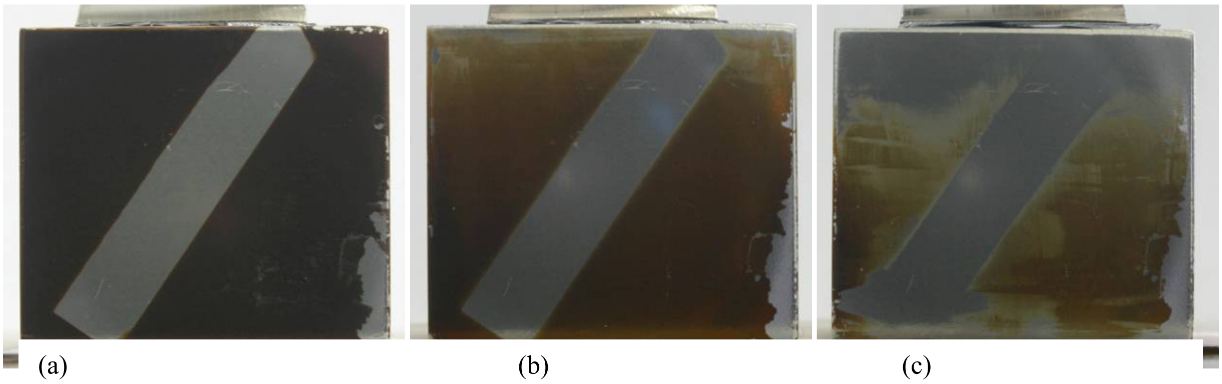
Figure 6: a) The positioning of the plasma torch above the gap, b) an image recorded during the cleaning procedure

The cleaning parameters were: Nitrogen flow = 8200 sccm,RF power = 350 W, Distance from top face of the built castellation = 2mm, Scanning speed = 5mm/s, Gap width = 0.5 – 1.5 mm The distance from the plasma torch tip to the castellated surface was 2 mm.

Investigation of material removal (hydrogenated carbon, mixed layers, DLC and real Tokamak layers))

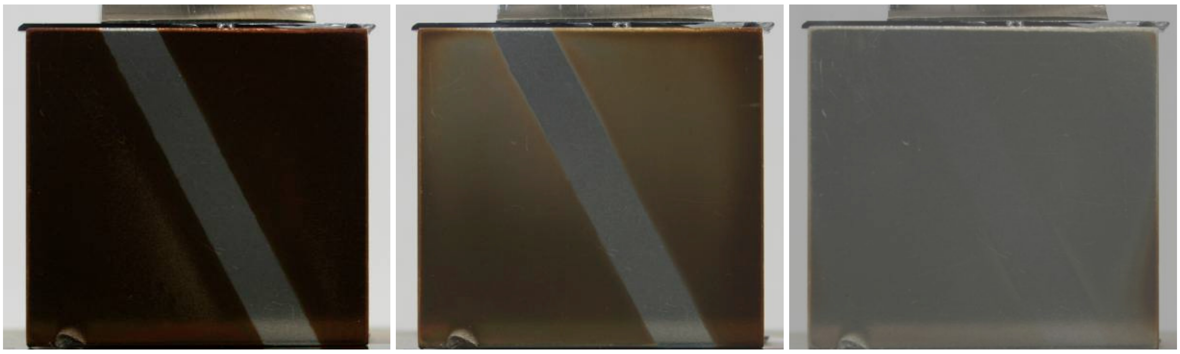
Illustration of the removal process by imaging (hydrogenated carbon)

The material removal was investigated by imaging and profilometry. The removal was realized using repetive scanning movements along the gap. The cubes were dismantled and the material



(a) (b) (c)

Figure7 : Images of the cube face after cleaning with the plasma torch positioned on the middle of the gap after a) 1 scan , b) 46 scans and c) 101 scans



(a) (b) (c)

Figure 8: Images of the cube face after cleaning with the plasma torch positioned on the side of the gap after a) 1 scan , b) 20 scans and c) 46 scans

removal was investigated after increasing numbers of scans. In Figure 7 are shown images of the inner surface limiting the gap, at the begining of the procedure, after 46 and 101 scans, respectively for a gap of 1 mm width , with the plasma torch positioned on the center of the gap. The cleaning is more efficient when the plasma torch is positioned on the side of the cube. This is proved by the images in Figure 8, showing that almost complet cleaning is possible after 46 scans in this case.

Evaluation of the cleaning rates from profilometry measurements

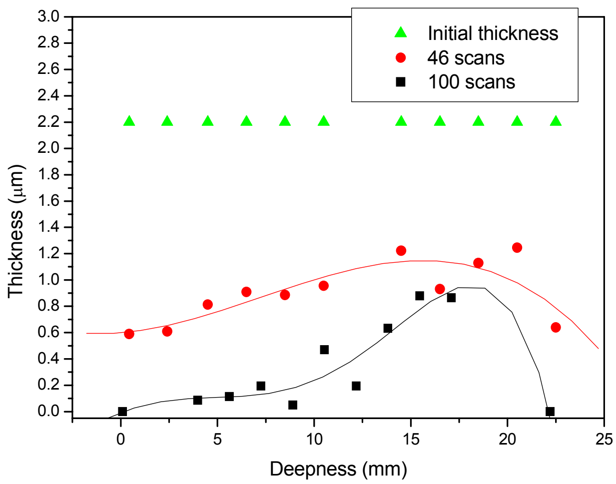


Figure 9: the thickness profiles of the initial layer and of the remained layers after the 46 and 100 scans inside the castellation.

In Figure 9 are presented the thickness profiles of the initial layer and of the remained layers after the 46 and 100 scans inside the castellation.

From the figure it is observed that the cleaning is more efficient at the top side of the gap, from where the material is removed first. This si convenient for cleaning Tokamak tiles, because it was reported that the co-deposited layers are thicker at the upper side of the gaps. Another aspect is that the cleaning is again very efficient at bottom, were the plasma reflexion occurs. This was proved also by placing a silicon piece coated with 1.5 μm layer at the bottom of the

The mass removal rate will be defined as the amount of mass removed by the plasma torch in the time unit. It can be calculated from the removed volume considering a constant density. The volume is obtained by measuring the thickness of the layer before and after the removal procedure.

Let us consider a surface with the length *l* and the height *h* (which defines the castellation) covered with an uniform layer having an initial thickness *d* and the density *ρ*. It is submitted to scanning by the plasma torch which is moving along the length *l* of the castellation with a speed *v*. The removed material in each point of the castellation is described by the difference between the initial thickness and the remained thickness.

The local (meaning at a given value of height) removal rate will be:

$$R_{local} = \frac{\Delta m}{\Delta t} = \frac{\rho \Delta V}{\Delta t} = \rho \frac{\Delta d * l * \Delta h}{\frac{l}{v} * N_{scans}} = \frac{\rho * v * \Delta h * \Delta d}{N_{scans}},$$

where the total time was obtained from the scanning speed and the total number of scans after which the removal is evaluated, Δd is the removed thickness of the layer at a given height, and Δh is the position where the removal was evaluated.

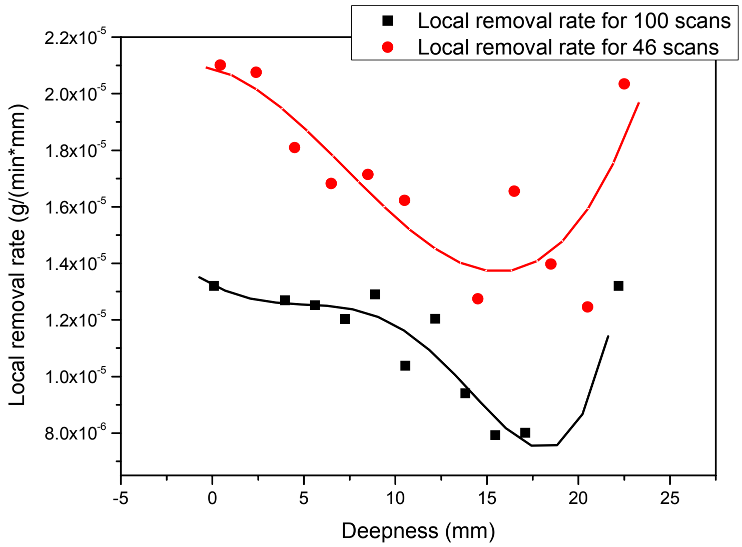


Figure 10: The local removal rate dependence upon position (cleaning parameters: Nitrogen flow 8200 sccm, RF power 350 W, Distance from top face of the built castellation 2mm, Scanning speed 5mm/s, gap width 0.5 mm

The profile of the local removal rate was evaluated considering Δh = 1 mm around the measured height position. The results are presented in Figure 10.

One can see that that the removal rate is higher for all the heights during the first scans. Moreover, the local removal rate is decreasing with the castellation deepness and presents an increase near the bottom of the castellation, due to the plasma reflection on the bottom part.

The integral removal rate can be evaluated by integrating the local removal rate along the whole castellation deepness. The corresponding values are $R_{integral(46)} = 3.5 \times 10^{-4}$ g/min and $R_{integral(100)} = 2.4 \times 10^{-4}$ g/min, indicating a slight dependence of the rate upon time (faster cleaning in the early times of the procedure).

Cleaning of mix layers consisting of hydrogenated carbon and aluminum

Layers of hydrogenated carbon and aluminum mix, realized by the two sources technique (PECVD and MS) as described before were deposited on flat silicon substrates. The layer thickness was 1μm. The layers were submitted to plasma torch cleaning in nitrogen using a power of 350 W, at 200sccm nitrogen, and torch tip to sample distance 3 mm. In Figure 11 are presented the cleaning traces obtained for an increased number of scans. The complete removal is apparently obtained after 10 scans. Nevertheless, from the images appears that the most probably the surface remain contaminated, probably with aluminum compounds (it is expected that aluminium oxide is formed).

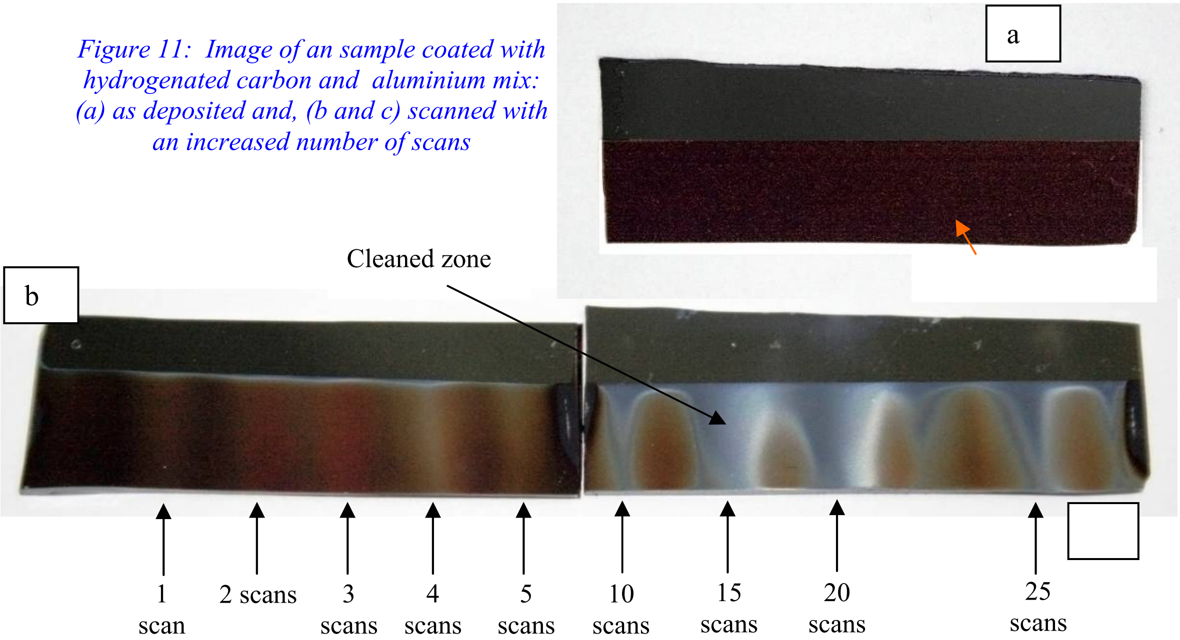


Figure 11: Image of an sample coated with hydrogenated carbon and aluminium mix: (a) as deposited and, (b and c) scanned with an increased number of scans

Cleaning of flat surfaces coated with Diamond Like Carbon layers.

The samples were supplied by Max-Planck-Institute for Plasma Physics, Reactive Plasma Processes Material Science, Garching, Germany (Dr. Thomas Schwarz-Selinger, Prof. Wolfgang Jacob). The samples characteristics were: dense film of DLC, deposited on Si substrate, thickness ~500 nm. The plasma torch removal conditions were: Nitrogen flow = 5700 sccm, RF power = 350W, distance plasma source to sample = 3 mm and 6 mm.

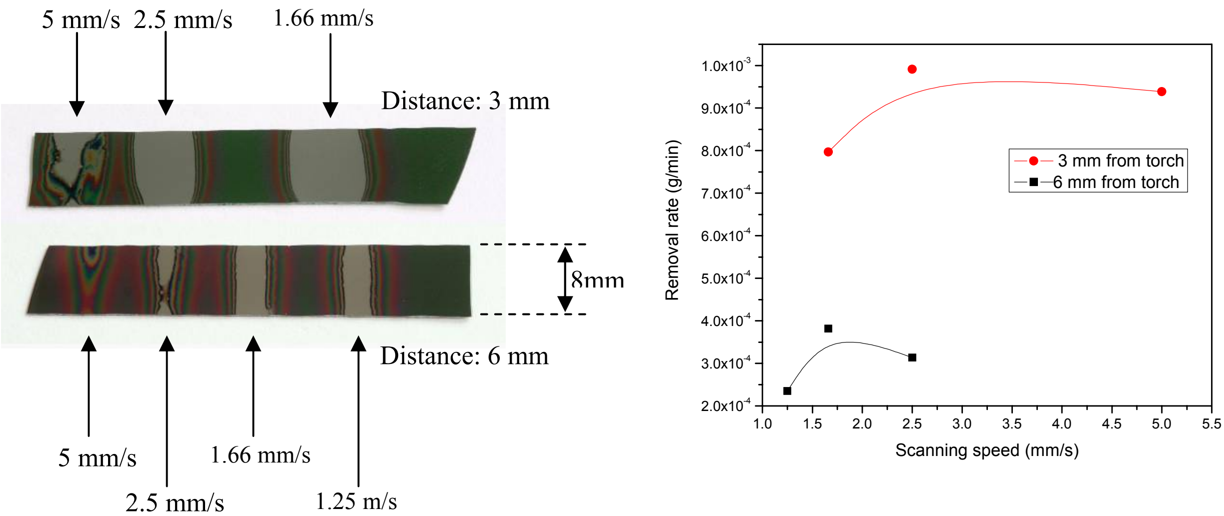


Figure 12: Cleaning tests on DLC layers: image of cleaning traces for various scanning speeds at 3 and 6 mm distance (left) , and the minimal removal rates estimated from the images (right)

In these experiments the plasma torch was directed perpendicular onto sample surface and have been used various torch speeds. The image of cleaned traces for different scanning speeds and the two distances is presented in Figure 12. The removal rates were estimated from the image (density considered 2 g/cm^3), but they are only limited minimal values, because the substrate was reached in all the traces. The removal rates are higher than 10^{-3}g/min for 3 mm distance and 3 x10^{-4} g/min for 6 mm distance.

Cleaning tests realized on back sides of the mirrors

The samples were supplied by JET, consisting of Ni made mirrors used in JET (prior the Be evaporators, several years till 1989). The most probable the mirrors supported carbon or carbon composites deposition.

Removal conditions:

Nitrogen flow = 6400 sccm
RF power = 350W
Distance plasma source – mirror = 7 mm
Scanning speed = 5 mm/s

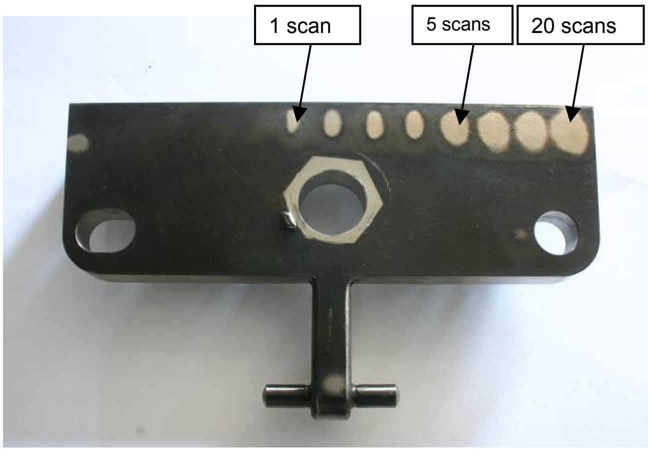


Figure 13: Illustration of the cleaning effect on the back side of mirrors exposed to scanning with the plasma torch; a) initial, b) coated with a mask, c) without mask, after cleaning at different number of scans

In Figure 13 are presented comparatively the images of the back side of mirrors in different stages of the cleaning procedure. The cleaning effect is visible on the image in Figure 13 c.

2.3. Conclusions.

Model surfaces have been designed and realized with deep gap and variable width geometries. The inner surfaces of the gaps have been coated with composite materials carbon/hydrogen or carbon hydrogen aluminum mix, with a thickness of up to 2.4 microns by Plasma Assisted Chemical Vapor Deposition and Plasma Assisted Chemical Vapor Deposition combined with Magnetron Sputtering. The coating was performed through a dedicated mask, creating a transition region between deposited and non-deposited zones distributed along the deepness of the gap, from top to the bottom. The gaps were scanned along their length by a plasma torch working in nitrogen aiming to clean the inner limiting surfaces. The cleaning effect after repetitive scans was investigated at variable deepness values from the top by imaging and by profilometry, applied to the transition region between the deposited and non-deposited zones. Removal of layers from inside gaps was demonstrated for gap widths 0.5-1.5 mm, deepness down to 20 mm. The removal rates are in the range 10^{-4} g/min. The cleaning effect is stronger near the top of gap, where the plasma is stronger and at the gap bottom where the plasma is back-reflected by the gap end. In relation with the plasma position the cleaning effect is stronger when the torch is positioned on the gap side, comparing with the positioning on the gap middle. The removal of mix layers was demonstrated, as well; the degree of surface contamination with remaining metallic traces has to be investigated. The cleaning with plasma torch was demonstrated also for DLC layers and real co-deposited layers from the back sides on IR mirrors exposed in JET.

Future work

An upgraded plasma torch device, able to work with more active (air, oxygen) gases is necessary for investigation of removal of mix layers with plasma generated at various conditions. The problem of the metalic compounds which can remain as residuals on the cleaned surface has to be studied.

References

Conferences

17. G. Dinescu, E.R. Ionita, M. Teodorescu, C. Stancu. T. Acsente, M. Bazavan, Radiofrequency expanding plasma sources at atmospheric pressure. Applications, invited lecture, Proceedings of 4th International Congress on Cold Atmospheric Pressure Plasmas: Sources and Applications (CAPPSA 2009) June 22-24, 2009, Ghent, pages 17-23. V. V. Yankov, JETP Lett. **60**, 171 (1994);
18. E.R. Ionita, C. Stancu, G. Dinescu, C. Grisolia Radiofrequency plasma jet cleaning inside TOKAMAK deep gaps at atmospheric pressure”, presented at International Colloquium on Plasma Processes (CIP 2009), June 22-26, 2009, Marseille, France (Book of Abstracts, page 254)
19. G. Dinescu, Surface modification and film deposition with expanding RF plasmas at atmospheric and low pressure, invited lecture, 16th International Summer School on Vacuum, Electron and Ion Technologies (VEIT 2009), 27.09-2.10. 2009, Sunny Beach, Bulgaria (Abstracts IL 17, page 34)
20. C. Stancu, E.R. Ionita, G. Dinescu, Carbon removal from deep gaps with atmospheric pressure plasma, Sixteenth International Summer School on Vacuum, Electron, and Ion Technologies, 28 September–2 October 2009, Sunny Beach, Bulgaria (Abstracts PA-21, page 59)

Presentations at meetings:

1. G. Dinescu, B.Mitu, E.R. Ionita, C.E. Stancu, M. Teodorescu, C. Grisolia, Carbon cleaning by a plasma torch from inside gaps- influence of the geometric aspect ratio of castellated surfaces - Fuel Removal EU PWI SEWG Summer Meeting, 15-17 June 2009, Cadarache
2. C. Stancu, E.R. Rosini, V. Satulu, T. Acsente, B. Mitu, G. Dinescu, C. Grisolia, Cleaning of co-deposited layers by movable devices based on radiofrequency discharges , MEDC Association Days, 26-27 November, 2009 , Magurele Bucharest