

II. Plasma Wall Interaction

ACTIVE IR THERMOGRAPHY BY PULSED PHOTO-THERMAL METHOD

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Overview

In the frame of the task it was proposed to develop an active infrared thermography method based on the pulsed photo-thermal effect, which will give an absolute measurement of the surface temperature independent on the emissivity values. Demonstration in the laboratory of this method requires the involvement of laser techniques but also preparation of adequate substrates and deposited layers. As substrates carbon and CFC, tungsten, beryllium are of interest and as deposited layers are of interest mixtures similar to that encountered in tokamaks: as example carbon with hydrogen, W/C +hydrogen, Al/C with hydrogen, (in these layers Al is chosen as substitute for beryllium). The samples for the laser experiments are to be realized and supplied by Finnish and Romanian groups.

In the frame of the project the Romanian team focused on the following particular objectives:

- 1 - Setting up of a technique and a setup based on a new approach for depositing layers of carbon mixed with metal impurities and hydrogen;
- 2 - Deposition of samples with thick layers (more than 10 microns) of mixed materials containing metals (Al as substitute for Be), carbon with hydrogen inclusions on various substrates.

Summary of the obtained results: A set-up for deposition of layers of mixed Al/C + hydrogen or W/C + hydrogen was realized. In the present approach (named sequential deposition) the deposition method consists in alternatively placing the substrate in front of two plasma sources: a magnetron for metal deposition and a Plasma Enhanced Chemical Vapor Deposition (PECVD) source for hydrogenated amorphous carbon (a-CH) deposition. Deposition experiments were performed. Graphite was used as substrate (polished samples dimensions 30x30x5 mm), but also silicon for material characterization purposes. The conditions in which composite layers can be obtained were identified, consisting in cycle times and plasma (RF powers and gas flow rates) parameters. The layer stability against the variation of such parameters was investigated. The deposition rates slightly vary between 6 nm/cycle (23s) to 8 nm/(cycle). The composition of the layers was examined by EDX (Energy Dispersive X-ray analysis) while the morphology using SEM (Scanning Electron Microscopy). Layers of the type a-C:H/Al with low Al content proven to be stable (limited oxidation, remain adherent on substrates), while a higher Al content a fast oxidation occurs leading to delamination

2. Detailed results

For preparing samples for Active IR Thermography investigations the Romanian group at NILPRP developed a new technique named *sequential* deposition. It consists of alternately placing the substrate in front of two plasma sources. The plasma sources are: i) a magnetron source for metal deposition, and ii) a Plasma Enhanced Chemical Vapor Deposition (PECVD) source for hydrogenated amorphous carbon (a-CH) deposition. In first attempts (utilized in the first part of the project, not reported here) of obtaining composite layers a-C:H/Al by continuous deposition with only one source (reactive magnetron sputtering with Al target) utilized with mixed gases (argon/acetylene) the control of deposition was difficult due to target poisoning with carbon. In the new approach the two mentioned plasma deposition processes are independent and sequential (every plasma source is ignited only when the substrate is in front of

it, while during the substrate moving between the plasma sources there are created specific conditions to each deposition process: gas composition, RF power, etc.). The substrate is transported between the two plasma sources by means of a stepper motor. During the PECVD process there is utilized as working gas a mix of Ar and C_2H_2 (as carbon precursor gas), while during the magnetron sputtering there is utilized only Ar. The magnetron metallic target is Al, while substrates consisted of polished graphite samples (30x30x5mm); also for material characterization purposes have been used silicon wafers and glass substrates. In addition, for a better control of the process an optical technique has been tested: the control of the deposition process is performed by means of optical emission spectroscopy, monitoring three plasma emission lines (the 396.1nm Al line, the 656,21nm H α line and Ar 738,18nm line).

2.1. Experimental setup and procedure

The sequential deposition setup was developed based on one deposition chamber assisted by gas supplying and pumping systems. Figure 1a presents a schematic view of this setup while in Figure 1b it is easy to remark the two plasma sources, the stepper motor, the computer interface and the optical spectrograph.

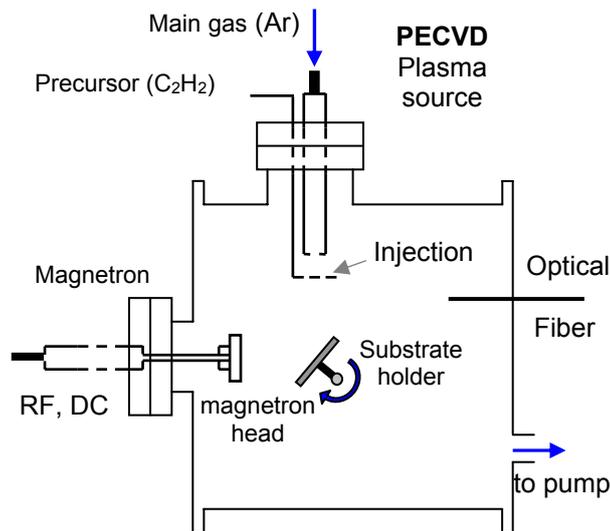


Figure 1.a: Schematic view of the deposition setup with computer controlled rotateable substrate.

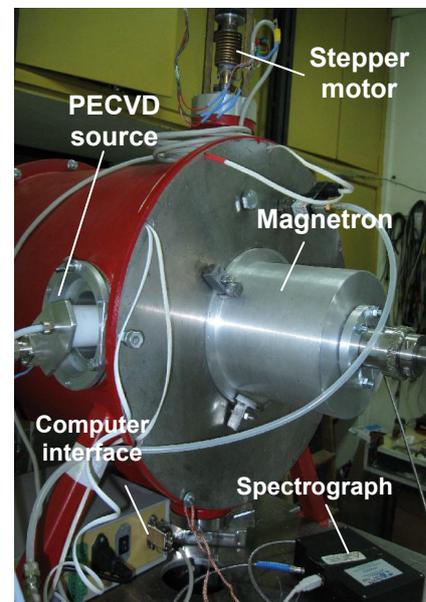


Figure 1.b. An image of the sequential deposition set-up, with its main components

The entire deposition process consists of a number N of deposition cycles, depending on the desired film thickness. We have to note that main gas (Ar) is flowing continuously through the reactor, while the precursor (C_2H_2) for deposition of the a-C:H material is present only during the PECVD process. A deposition cycle contains four steps, as presented in Figure 2:

- Magnetron sputtering deposition step, with a T_{Al} duration; during this process, only the magnetron head is energized via a matching box by an RF generator and the substrate is parallel to the magnetron;

- Transport and precursor admission step (T_{ADM} duration): the stepper motor move the substrate in a position parallel with the PECVD source. At the beginning of this step C_2H_2 is admitted in the reactor in; at the end of this step the flow of precursor gas attains a stabilized value and the pressure is suitable for the start of PECVD process;

- Deposition of a-C:H by PECVD, with a T_C duration; during this process, the substrate faces the PECVD plasma source which is energized by an RF generator;

-Transport and precursor pumping down step (T_{PUMP} duration): the stepper motor move backward the substrate, in the position parallel with magnetron head; at the beginning of this step the C_2H_2 flow is stopped.

For controlling of all presented processes (substrate movement, steps duration, gas admission, start and stop of RF generators, number of cycles) there were developed suitable hardware and software components based on a computer interface in association with a LabView virtual instrumentation program.

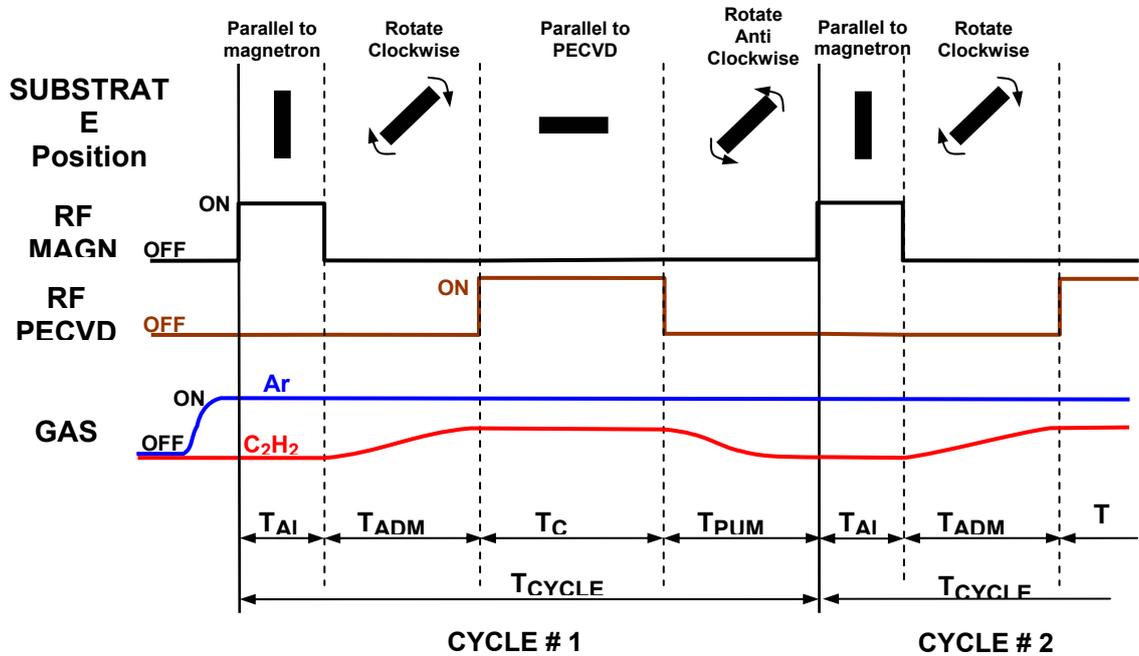


Figure 2: Temporal diagram of one cycle in the sequential deposition process.

The deposition process is monitored by means of optical emission spectroscopy, using an Ocean Optics CCD spectrograph (optical multichannel analyzer – OMA, accompanied by Spectra Suite software), allowing recording the time evolution of three plasma emission lines (the 396.1nm Al line, the 656,21nm $H\alpha$ line and Ar 738,18nm line). The presence of the hydrogen $H\alpha$ emission line proves the availability of H for incorporation in the deposited film. Figure 3 presents the evolution of these spectral lines during a sequential deposition with $T_{Al}=4s$, $T_C=9s$. We have to note that prior starting the effective sequential deposition there is necessary to clean the Al target (pre-sputtering phase).

In fig.4 is presented an image of a sample of mixed a-C:H/Al layer deposited by means of the above described sequential deposition technique.

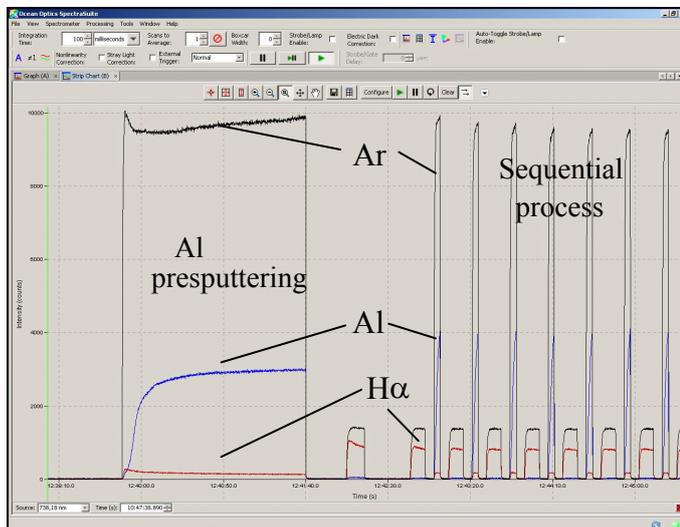


Figure 3: Time evolution of the Al, Ar and Ha lines during the Al target pre-sputtering (cleaning) and first seven deposition cycles; $T_{Al}=4s$, $T_C=9s$.

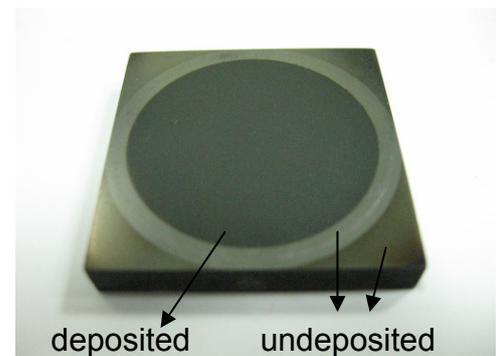


Figure 4: Samples Al/a-CH layer on graphite substrate (30mm x30mmx5mm) Deposition rate: $\approx 1 \mu m/hour$

2.2. Results and discussions

Deposition process. Parametric study

During the deposition there were used the following parameters: Ar mass flow rate $\Phi_{Ar} = 70\text{sccm}$, C_2H_2 mass flow rate $\Phi_{C_2H_2} = 5\text{sccm}$, magnetron RF power $P_{RFMG}=100\text{W}$, PECVD plasma source RF power $P_{RFC}=160\text{W}$. The optimum values for sequential cycle times: $T_{CYCLE} = T_{Al} + T_{ADM} + T_C + T_{PUMP}$, were T_{CYCLE} value was chosen to be 23s, were determined. Such as it was measured that $T_{ADM} = T_{PUMP} = 5\text{s}$ 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).

Deposition rates

For the sequential deposition method it is important to mention the deposition rate for a deposition cycle. The deposition rate was determined by AFM technique, for thin films deposited on Si using the mentioned RF powers and gas flows, for a number of $N=139$ cycles. During these depositions there were varied T_{Al} and T_C ; the resulting deposition rates are presented in Table 1:

Table 1:

Probe	$T_{Al}(s)$	$T_C(s)$	Rate (nm/cycle)	Duration for $10\mu\text{m}$ film (hh:mm:ss)
1	2	11	6	10:38:53
2	4	9	6	10:38:53
3	6	7	5.4	11:49:53
4	8	5	7.2	08:52:24

Film composition and morphology

Figure 5 presents an example of composition investigation (EDAX measurement) and morphology (SEM result) for a film deposited on polished graphite substrate using the previous mentioned deposition parameters, for $T_{Al}=2\text{s}$, $T_C=11\text{s}$. These investigations were performed on Si samples placed near the graphite probe during the deposition.

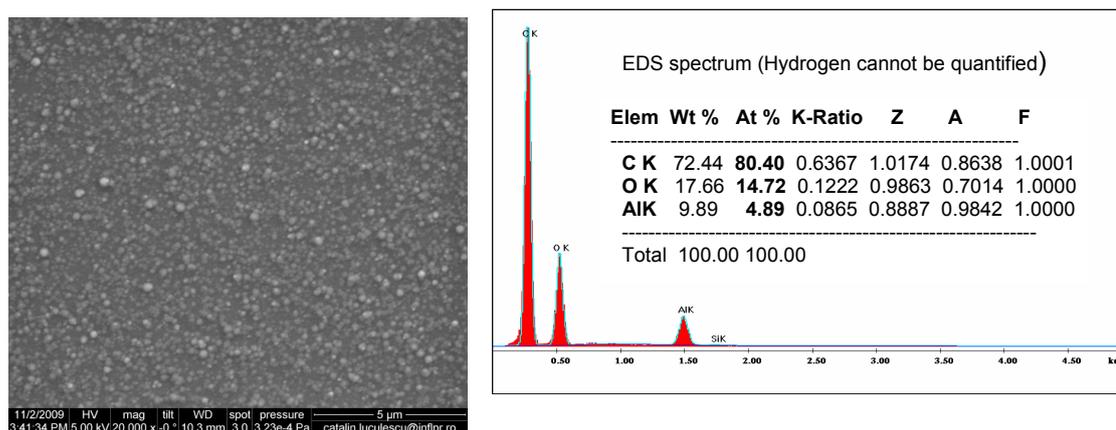


Figure 5. Morphology (left) and EDAX investigation (right) results for a a-C:H/ Al layer deposited with $T_{Al}=2\text{s}$, $T_C=11\text{s}$.

We observe the presence of Al and C in the deposited film. There are also present impurities, mainly oxygen. Presence of oxygen might be associated with the residual presence of this gas in the processing chamber during deposition, but mainly with oxidation during the exposure to atmosphere of the fresh deposited sample.

2.3. Conclusions.

Samples consisting of layers of mixed Al/a-C:H materials, (with Al as substitute of Be) deposited on polished graphite substrates were realized. For deposition we have explored reactive magnetron sputtering technique and a new deposition technique which makes use of two plasma sources mounted on the same deposition chamber, and separate injection points for the gases. The plasma sources are: a magnetron source for metal deposition and a PECVD source for hydrogenated amorphous carbon (a-CH) deposition. We proved the feasibility of this new deposition concept, which can be described as sequential deposition: it consists in alternately placing the substrate in front of each plasma source for predetermined periods of time.

The main advantage of the sequential deposition method is the possibility of fine controlling the film composition and morphology by adequate adjustment of T_{Al} and T_C deposition times. Nevertheless, it was observed that for obtaining thick layers (over 10 microns) it is necessary a longer time in the case of sequential deposition, in comparison to the reactive magnetron deposition case. Also the sequential deposition asks for important equipment utilization (two matching boxes, two RF generators, computer control of substrate rotation and exposure).

The deposited films are stable (in terms of color and adherence) in time only for small concentration of Al (for a maximum Al deposition time $T_{Al}=6s$). For larger Al concentration (Al deposition times up to $T_{Al}=11s$), the deposited layers change color very fast (from minutes in case of high Al contents to hours in case of lower Al content) and delaminate in air in maximum one day (most probably due to interaction between Al and atmospheric oxygen).