

AUTOMATION OF A DUAL ION BEAM SPUTTERING SYSTEM FOR OPTICAL APPLICATIONS

I. DI SARCINA, L. DOMINICI, D. FLORI, F. MENCHINI, F. MICHELOTTI, S. SCAGLIONE

Abstract

Ion beam sputtering is one of the high-vacuum processes employed for the production of thin films starting from solid targets. It consists in the ejection of surface atoms from a target by momentum transfer from bombarding ions to surface atoms. The deposition conditions strongly influence the characteristics of the deposited films, such as their thickness, structure, refractive index, absorption and mechanical stability.

Advances in optical design techniques have led to such refined designs that even small variations of the material properties are intolerable. For this reason, it is fundamental to precisely control the deposition conditions and ensure their repeatability.

In the ENEA Casaccia Optical Coatings Laboratory, a Dual Ion Beam System (DIBS) facility is constantly operating. It comprises two Kaufman-type ion sources for film growth and assistance, a three-target rotating stage, and several configurations for the sample holder. Last year we decided to upgrade the plant with an automation system which could improve its reliability and repeatability standards.

Key words: Dual Ion Beam Sputtering, deposition parameters, automation, LabVIEW

AUTOMATIZZAZIONE DI UN SISTEMA DI DEPOSIZIONE CON DOPPIO CANNONE IONICO PER APPLICAZIONI OTTICHE

Riassunto

Lo sputtering con cannone ionico è una delle tecniche ad alto vuoto utilizzate per la produzione di film sottili partendo da target solidi. Consiste nell'espulsione degli atomi di superficie di un target grazie al trasferimento di momento da parte degli ioni incidenti.

Le condizioni di deposizione influenzano le caratteristiche dei film depositati in particolare lo spessore, la struttura, l'indice di rifrazione, l'assorbimento e la stabilità meccanica.

Le moderne tecniche di design hanno portato alla realizzazione di progetti così raffinati che anche minime variazioni delle proprietà dei materiali sono intollerabili. Per questa ragione, è importante controllare con estrema precisione le condizioni di deposizione e garantire la loro ripetibilità.

Nel Laboratorio di Trattamenti Ottici dell'ENEA Casaccia è in funzione un impianto a doppio fascio ionico (DIBS). Esso comprende due sorgenti ioniche di tipo Kaufman per la crescita dei film e l'assistenza, un porta target rotante con tre alloggi e numerose configurazioni per porta campioni. Nello scorso anno è stato effettuato un upgrade dell'impianto con un sistema di automazione che ne ha migliorato l'affidabilità e la ripetibilità.

Parole chiave: Sputtering a doppio cannone ionico, parametri di deposizione, automatizzazione, LabVIEW

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AUTOMATION OF A DUAL ION BEAM SPUTTERING SYSTEM FOR OPTICAL APPLICATIONS

1. INTRODUCTION TO SPUTTERING TECHNOLOGIES

There are many deposition technologies for material coating manufacturing, which can either purely physical or purely chemical methods, or a combination of the two (1).

Evaporation and sputtering are the two basic kinds of physical vapor deposition (PVD) processes. Basically, both processes consist in the “vaporization” of the material to be deposited and its subsequent “condensation” on a substrate. In the first technique, the atomization of the material is obtained by thermal processes (evaporation), while in the second it is obtained by collision process.

Sputtering consists in the ejection of surface atoms from an electrode by momentum transfer from bombarding ions to electrode surface atoms (see Fig. 1). With respect to evaporation, the sputtered particles arrive at the substrate with higher energies, and this circumstance determines many advantages to the deposited films, in terms of

- better adherence, density and stability of the films
- better thickness control
- better exploitation of the target material
- great versatility in the choice of target and substrate materials.

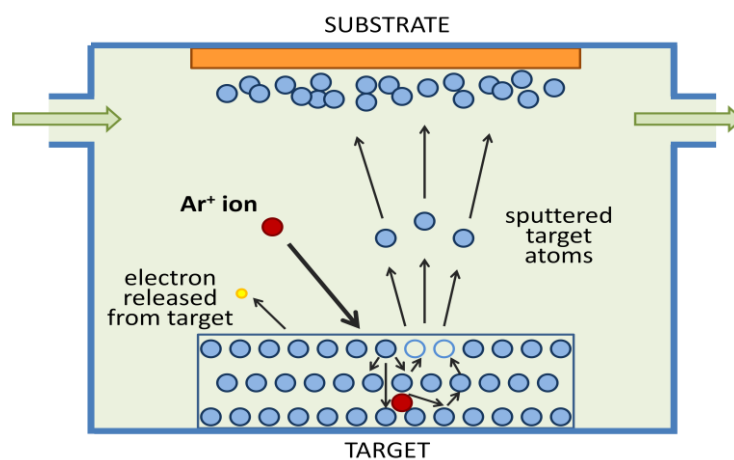


Figure 1 – The sputtering process.

Sputtering is an elemental process, in which the ions collide with the atoms of the target and in some conditions are ejected from the target itself. The ratio between the number of ejected atoms and the number of ions is named *sputtering yield*. It depends on the ion beam characteristics (ion mass, ion energy etc.) and the properties of the target. Generally, the different atoms of a multi-component target have different sputtering yields and the initial stoichiometry of the sputtered material is modified during the ion beam sputtering process (preferential sputtering). In general, the oxygen concentration of sputtered oxide thin films results lower than the correct stoichiometric value and the optical absorption results increased.

Sputter-deposition has become a generic name for a variety of processes, which are briefly described in the following.

Diode sputtering: uses a plate of the material to be deposited as the cathode (or rf-powered) electrode (target) in a glow discharge. Material can be thus transported from the target to a substrate to form a film. Films of pure metals or alloys can be deposited when using noble gas discharges (typically Ar) with metal targets.

Reactive sputtering: compounds can be synthesized by sputtering elemental, alloy or compound targets in reactive gases.

Bias sputtering or ion plating: is a variant of diode sputtering in which the substrates are ion-bombarded during deposition to produce several desired effects, such as re-sputtering of deposited material, low-energy ion implantation, desorption of gases or modification of a large number of the film characteristics.

Magnetron sputtering: is another variant which uses transverse magnetic fields to produce important modifications to the basic process, such as decreasing the deposition temperature and increasing the deposition rates.

Ion-beam sputtering: the sputtering ions come from ion beams of glow discharges.

Each technology has its limitations and its advantages over the others, which have to be understood in order to obtain the desired film characteristics.

1.1 Ion-beam sputtering

In ion-beam sputtering, the ion beam is produced in and extracted from glow discharges in a differentially pumped system. The target and substrate are situated in a high-vacuum environment rather than in a high-pressure glow discharge: glow discharge artifacts can therefore be avoided and higher purity films usually result.

Ion bombardment is particularly indicated in the case of dielectrics, where the conventionally deposited film is amorphous or polycrystalline due to the low mobility of the depositing atoms.

There is a wide variety of ways in which ion beams are used in the processing of thin films. Depending on the application, the beam may be composed of atoms of a simple inert or reactive gas, molecules of a more complex nature or even clusters of molecules. Different source configurations are used to generate these beams, and are designed to cover a wide range of energy and current combinations. The three sources which have been most widely used in the production of thin films are briefly presented in the following.

Hollow cathode (2): the desired gas (typically oxygen) is flowed into the discharge region, and a high voltage is applied between the cathode and the anode. A small carbon aperture in one end of the chamber allows some of the energetic species to escape, but unfortunately with little control over their energies and a strong angular dependence of the flux. Typical required powers are of 0.5 A at 0.5 to 1 kV. The particles have a spread of 10 to 50 eV, peaked at less than a hundred eV.

Duoplasmatron (3): a heated filament, and a combination of electric and magnetic confining fields, create a small, high-density plasma. It produces a focused beam which may be rastered

electronically, and the gun may be operated with chamber pressure in the 10^{-7} to 10^{-8} torr range. Typical energies are of 1 to 10 keV and total currents of tens to hundreds of microamperes.

Kaufman source (4) (5): it consists of a plasma chamber, combined with thermionic emission and magnetic confinement to sustain the discharge. Two grids are used to contain the plasma and extract ions from the discharge region. It can operate with beam energies from about 10 to 2000 eV, with typical maximum beam currents of 1-2 mA/cm². The improved ionization efficiency of the electrons permits the use of relatively low system pressures (about 10^{-5} torr). The energy spread of the ions in the beam is typically only a few eV, and the beam energy and current may be varied independently.

1.2 The Dual Ion-Beam Sputtering

The layout of the Dual Ion Beam Sputtering facility (DIBS) operating in the Laboratories of ENEA, Optical Coatings Laboratory is reported in Fig. 2.

The stainless-steel vacuum chamber has a cylindrical shape and comprises two Kaufman-type ion sources (Ion Source, controller model IT2500). The beam of the first ion source is directed towards the target (three different targets can be mounted on the holder), whereas the beam of the second ion source is devoted to the energetic assistance of the growing film.

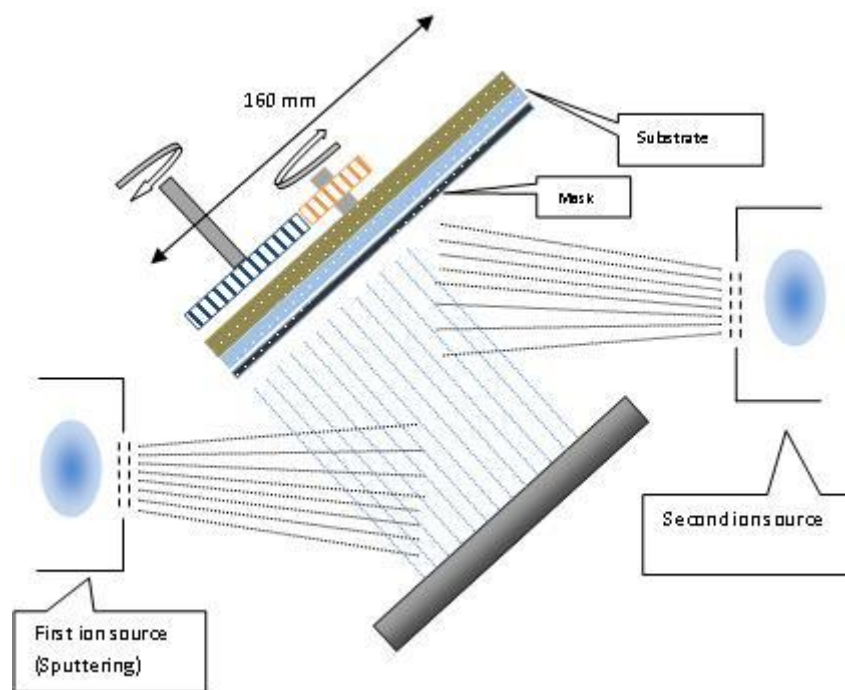


Figure 2 - DIBS layout.

The two-grids Kaufman ion source produce a mono-energetic beam with a Gaussian-like shape. The beam voltage (V_b) and grid accelerator voltage (V_a) are the two parameters governing the ion beam current density and the shape of the beam. In particular, the divergence of the beam varies with the ratio $R = V_b / (V_b + V_a)$: a collimated beam (low divergence) can be obtained with R close to 1, which is the ideal value, whereas for lower values of R the beam diverges. During the ion beam sputtering process, the distribution of the material ejected from the target depends on the beam shape and non-uniformity of the thickness on large area substrates can be induced.

The chamber can be evacuated down to 4×10^{-5} Pa by a rotary stage followed by a turbomolecular pump. The chamber pressure is measured by a Penning Cold-cathode Full-range Gauge read by a Pfeiffer TPG 262 Dual Gauge Meter. The pumping cycle is automatically run by a controller.

Different gases are necessary for the deposition: a noble gas (Ar, Xe, etc.) as the ionizing gas, Oxygen for reactive sputtering and in case other gases to obtain compound films. The gases are flown through four MKS Mass Flow Controllers connected to an MKS 647B Mass Flow Meter.

Up to three targets can be accommodated in the DIBS on a carousel mount which can be rotated by a motor. The exact repositioning of the targets is guaranteed by three position sensors and an Ionvac PLC Controller.

There are several possible sample holders which can be mounted in the chamber and can be rotated. Their maximum size is 160 mm.

In brief, the main characteristics of the plant are:

- Backpressure $4-5 \times 10^{-5}$ Pa
- Working pressure $1-2 \times 10^{-2}$ Pa
- Ion beam energy $E_b=1500$ eV max
- Ion beam current $I_b=125$ mA max

The most important advantages of the DIBS technique are:

- ✓ high energy of the depositing atoms, which produce high-density thin films with refractive index similar to the bulk value;
- ✓ clean process due to the low deposition pressure;
- ✓ linear dependence of the growth rate from the number of ions bombarding the target also for very low growth rate;
- ✓ low deposition temperature, which permits the use of many substrates types (e.g. plastic).

On the other hand, the DIBS technique has some disadvantages, among which:

- ◆ stoichiometry alteration, due to preferential sputtering;
- ◆ intrinsic stress of the coatings, which tends to contract it (or expand it) exerting a force counterbalanced by the substrate. If the adhesion strength at the interface is sufficiently high to compensate the tangential stress, the film is mechanically stable. Otherwise, if the coating is weakly bonded to the substrate, delamination occurs. Ion beam sputtered thin films are generally affected by compressive stress;
- ◆ ion beam divergence, which causes film thickness inhomogeneity.

2. AUTOMATION OF THE DIBS PLANT

The properties of a vacuum-deposited thin film of a material are rarely those of the bulk, and significant differences are observed even in films of the same material when prepared under different conditions. Advances in optical design techniques have led to specifications which may consist of hundreds of layers working in interference conditions to provide the desired spectral profile. These refined designs have made small variations of the material properties intolerable. Reproducible and stable optical properties, low absorbance and mechanical durability are required for a variety of applications, and the control of the deposition process has become a key element for a successful exploitation of the deposition technique.

The main points to address are: the desired film thickness and its density, which are closely related to the deposition rates; the stability and repeatability of the process, which is of basic importance

for device manufacturing; the uniformity of the deposited films, which impose severe restrictions on the design of the deposition plant.

For all these reasons, it is fundamental to precisely control the deposition parameter and to ensure their stability and reproducibility. Until now, this was left to the experience and the skill of the operator, who should follow the deposition process from the beginning to the end. However, in many cases when the number and deposition time of layers was very high, the task could become very demanding. Hence the decision to implement the automation of the plant and of the whole deposition process was taken last year.

For the development of the automation system we decided to rely on National Instruments Modules and LabVIEW Software, with which we already had some experience. All the work was undertaken in collaboration with Prof. Michelotti and Dr. Dominici from Base and Applied Sciences for Engineering Department (SBAI) of the Sapienza University of Rome. The operating program runs in LabVIEW 8.0 and is contained in a LabVIEW Project which is executed on a Compact FieldPoint (cFP) Real Time system connected to the DIBS apparatus.

Only part of the instrumentation used during the deposition with the DIBS was connected to the automating apparatus, and it is summarized in the following:

- ION TECH Ion Source Power Supply (for both GUN 1 and GUN 2)
- MKS 647 B Multi Gas Mass Flow Controller
- Pfeiffer TPG 262 Dual Gauge Meter
- Motor for substrate rotation
- Motor for target positioning

The Compact Field Point is composed by the following modules:

- Two 8-channels Analog Input modules (cFP-AI-100)
- One 8-channels Analog Output module (cFP-AO-210)
- One 4-channels Relay module (cFP-RLY-423)
- One 8-channels Relay module (cFP-RLY-421)
- One 8-channels Digital Input and 8-channels Digital Output module (cFP-DIO-550)

2.1 Hardware connections

A service PC is connected to the Field Point via a mini-hub as depicted in Fig. 3.

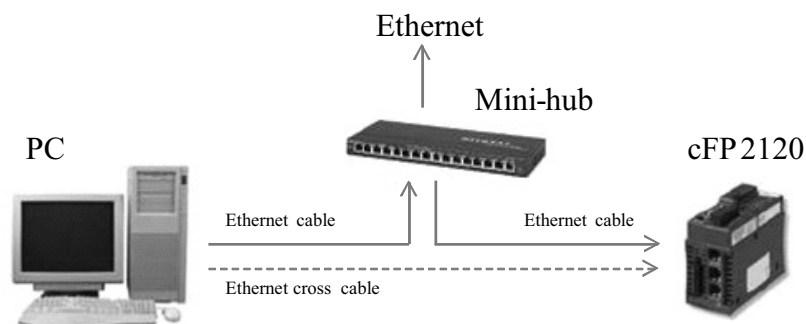


Figure 3 – Ethernet connections

The cFP is connected to the DIBS via 5 cables as shown in Fig. 4.

The control system of the Gas Flow Meter and the Pressure Controller are directly connected to the cFP central processing unit serial ports; the controllers of the two ion sources are connected to the analog-to-digital, digital to analog and relay modules; the motors for substrate and sample rotation are connected to the digital input-output module.

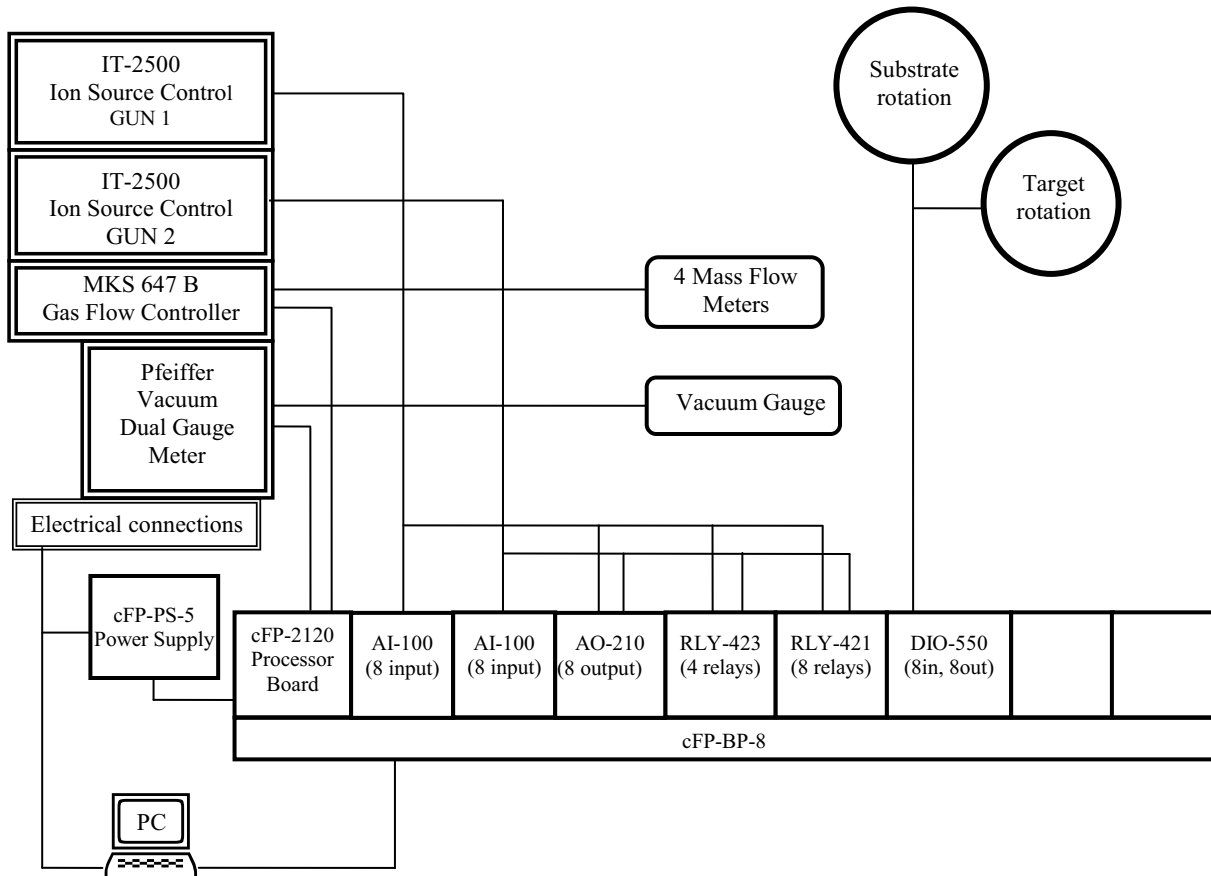



Figure 4 – Automation Connections

2.2 The control program

The program runs on LabVIEW Ver. 8.0 and following. The cFP must be equipped with the following software modules:

- Field Point Drivers 6.0.0
- Field Point VI Manager 6.0.1
- LabVIEW Real-Time 8.0
- Network Variable Engine 1.0.0
- NI-Serial RT 3.3.0 NI-VISA 4.3
- Variable Client Support 1.0.0

By double-clicking on the *DIBSmainprogram.vi* contained in the *DIBS.lvproj* project the Front Panel of the main program will be opened, ready to be run on the cFP and controlled via the Remote Panel on the Host PC. By clicking on the  icon the program is deployed on the cFP and is automatically run.

2.3 The Front Panel

The front Panel consists of 6 windows, each one is described in details in the following.

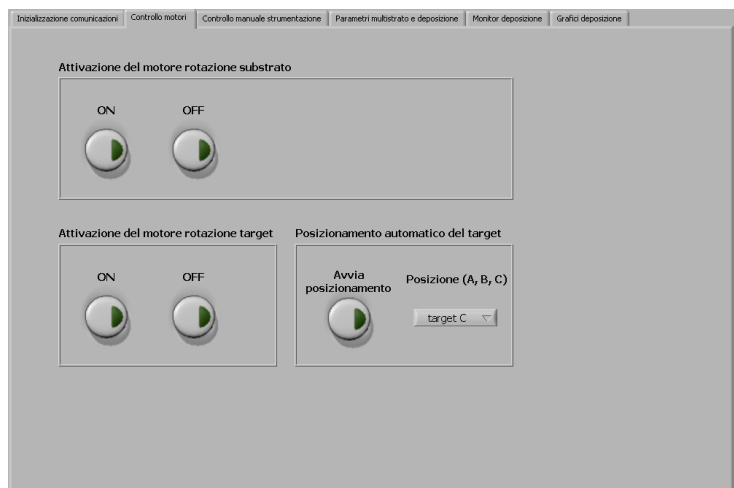
Communication Initialization

This window shows the opening parameters of the two serial ports of the cFP. Port COM2 is used to communicate with the Flux Meter, while Port COM3 to communicate with the pressure controller. The settings are normally fixed.



Motor control

In this window it is possible to manually start and/or stop the substrate and target rotation. It is also possible to set the target carousel in one of the fixed A, B or C positions by the position sensors. This window is automatically set inactive during an automatic deposition.

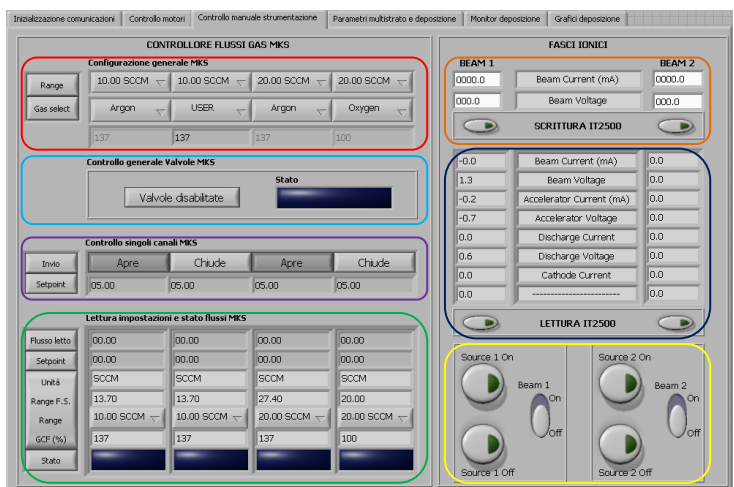


Manual control of instrumentation

The left part of this window is dedicated to the settings of the MKS 647B Flux Meter. Each of the four columns is relative to one of the four gas flux channels.

The upper block (**MKS General configuration**) allows to set the general parameters of the four flux channels, in particular their range and the gas conversion factor, which must be in accordance to the regulator settings.

The second block (**MKS general valve control**) allows enabling/disabling the opening of the flux valves.



The third block ([MKS single channels control](#)) allows opening each of the four valves of the flux regulator independently.

In the lower block ([MKS fluxes settings and state reader](#)) the state of the four flux regulators is shown.

The right part of the window controls the two IT-2500 ion sources controllers, respectively in the left and right column.

The first block ([Input IT2500](#)) allows setting the beam current (maximum 125 mA) and beam voltage (maximum 1500 V) for each gun.

The second block ([Output IT2500](#)) allows updating the reading of the state of the ion sources.

The third block ([Sources](#)) allows switching on/off the two ion sources and switching on/off the two ion beams.

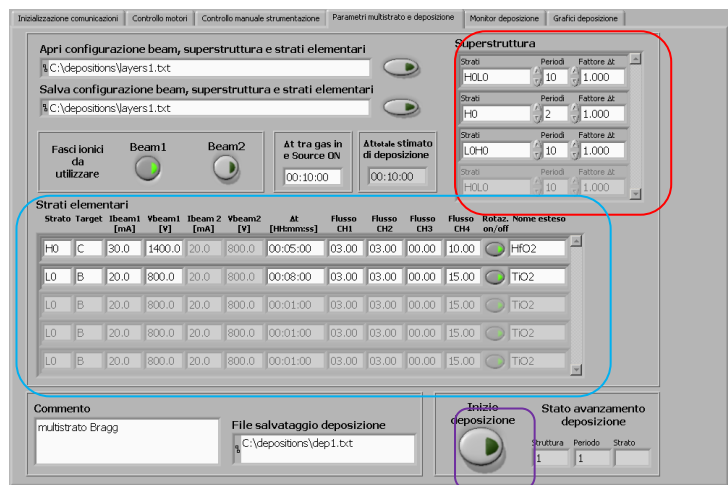
N.B. The ion source console must be set to Auto and Remote to be controlled via this program.

Deposition and multilayer parameters

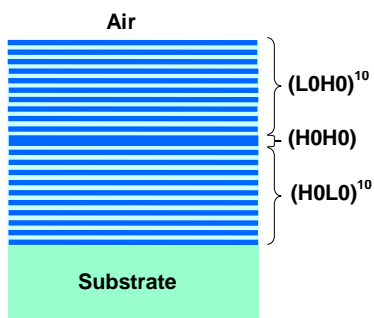
In this window it is possible to set all parameters relative to an automatic deposition, which can also be saved on a file for later use.

In the following, the automatic deposition procedure is described referring to a practical example.

Let us suppose we want to deposit a multilayer made of HfO_2 (mounted in position C of the target carousel) as the low-index material (L0) and TiO_2 (position B) as the high-index material (H0).



Let us suppose that the structure design is: Substrate/ $(\text{H0L0})^{10}\text{H0H0}(\text{L0H0})^{10}/\text{Air}$ as shown in the figure.



In the example, the multilayer is composed by three groups of layers:

$(\text{H0L0})^{10}$ where the couple HL is repeated 10 times

H0H0 where the H layer is repeated twice

$(\text{L0H0})^{10}$ where the couple LH is repeated 10 times

These data can be set in the block [Superstructure](#).

The factor Δt is a multiplicative factor of the ion source switching on time.

The single layers characteristics need to be set in the block [Elemental layers](#), one for each line. Such characteristics are: the initials of the layer, the position of the corresponding target, current and voltages of the two ion beams, the switching on time of the ion beams, the desired gas fluxes, the rotation of the substrate, an extended name for the layer.

The files containing the multilayer structure have the extension **.dibs**. The first line reports the number of active ion beams (0=no ion beam; 1=ion beam 1; 2= ion beam 2; 3=both ion beams). Then the superstructure is reported. Finally, the number and characteristics of the elemental layers

(the A, B and C target positions correspond to numbers 0, 1 and 2 respectively, while the switching on time appears in seconds).

```

esempio multistrato.dibs - Blocco note
File Modifica Formato Visualizza ?
beamnumber: 1
structures: 3
H0L0 1 1.000
H0 2 1.000
L0H0 10 1.000
elemlayers: 2
H0 2 030.0 1400.0 020.0 0800.0 0300 03.00 03.00 00.00 10.00 1 HfO2
L0 1 020.0 0800.0 020.0 0800.0 0480 03.00 03.00 00.00 15.00 1 TiO2
  
```

The deposition starts when the Start Deposition button is clicked, and, after a check of the structure, the window becomes inactive, and the Front Panel will switch to the next window.

The deposition data are saved in a file with extension **.depo** with the structure showed below.

```

esempio deposizione.depo - Blocco note
File Modifica Formato Visualizza ?
DIBS.l\proj Ver.1.0
nome file: C:\Users\ECAPD9\Desktop\2009 ENEA - DIBS\project\DIBS-LV86\esempio deposizione.depo
time 2010/01/18 21:08
Commento: multistrato Bragg

MKSrangeFS: 13.70 13.70 20.00 27.40
MKSunitsRA: SCCM SCCM SCCM SCCM
MKSchangCF: 137 137 100 137

Dt tra gas e source [s]: 600

beamnumber: 1
structures: 3
H0L0 1 1.000
H0 2 1.000
L0H0 10 1.000
elemlayers: 2
H0 2 030.0 1400.0 020.0 0800.0 0300 03.00 03.00 00.00 10.00 1 HfO2
L0 1 020.0 0800.0 020.0 0800.0 0480 03.00 03.00 00.00 15.00 1 TiO2

inizio dati: (tempo-pressione-Ch1-Ch2-Ch3-Ch4-Ib1-vb1-Ib2-vb2)
  
```

The first four lines contain: the program version, the file name, the date and a comment, if any. The following group of lines reports information about the gases: range, measuring units and correction factor. The following line reports the time interval between the first aperture of the gas lines and the ion beam switching on. The last block of lines reports the parameters which are monitored during the deposition and are saved every 5 seconds: time, chamber pressure, fluxes of the four gas channels, beam current and beam voltage of the two ion sources.

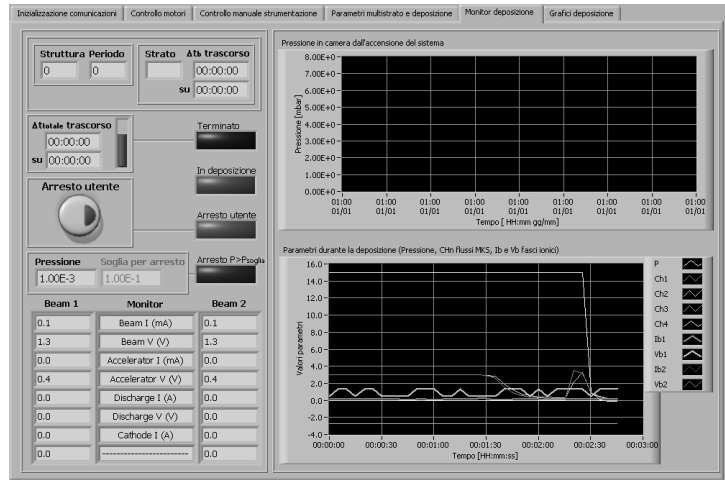
Both **.dibs** and **.depo** types of file can be read by the program to set the parameters of the multilayer.

Deposition monitor

When the deposition starts, the Front Panel automatically switches to this window.

Before going through this window, let us summarize all the phases in which the deposition process consists.

- Pre-multilayer phase. The gas regulators are opened to the maximum value set for the layers. After the interval Δt gas/source the ion sources are switched on, and 30 more seconds are waited.
- Multilayer phase. The iterative deposition cycle is started. For each layer, the corresponding target is set into position, then the gas fluxes are set to the desired values, the beam current and voltage are set and the ion sources are switched on for the desired time.
- Post-multilayer phase. The ion sources are switched off, while the gases are left open for 60 minutes. Finally, the gas channels are closed and the deposition process is completed.



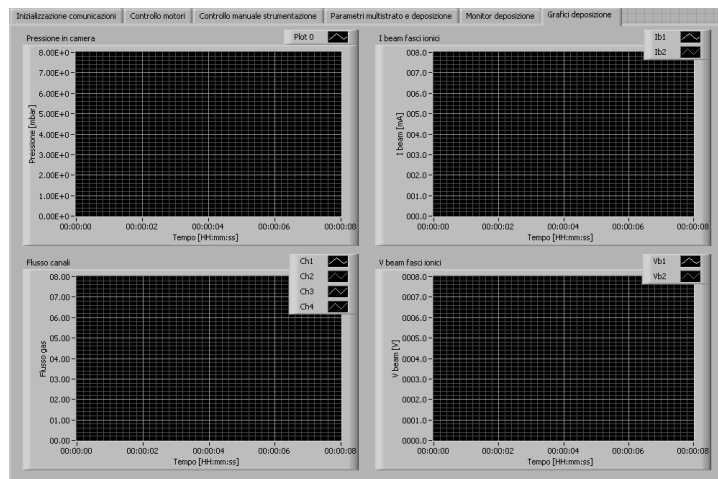
In the Deposition monitor window the state of the deposition can be followed.

The upper graph represents the chamber pressure. The lower graph reports some deposition parameters: pressure, flux Ch1, flux Ch2, flux Ch3, flux Ch4, beam current 1, beam voltage 1, beam current 2, beam voltage 2.

There are also indications about the layer which is being deposited and the elapsed time.

Deposition graphs

In this window the parameters during deposition are plotted separately for clarity.



3. CONCLUSIONS

The automation of the DIBS deposition plant present in our Laboratories was successfully carried out.

During last year the plant was employed for the production of samples for an ESA project, and needed to be continuously run. During this period the automatic deposition procedure was carefully tested. The employment of the automation system permitted to improve the reliability on the whole deposition process, and most of all it allowed reducing the production times.

REFERENCES

1. H.K. Pulker, *Coatings on Glass*, Elsevier, New York (1984).
2. J. Ebert, *Proc. SPIE Int. Soc. Opt. Eng.* Vol. 325, p. 29 (1982).
3. M. von Ardenne, *Tabellen der Elektronenphysik, Lonophysik und Ubermicroskopie*, p. 554, Dtsch. Verlag Wiss, Berlin (1956).
4. H.R. Kaufman, *Adv. Electron. Electron Phys.* Vol. 36, p. 265 (1974).
5. H.R. Kaufman et al. *J. Vac. Sci. Technol.* Vol. 21, p. 725 (1982).

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