# Sputter deposition system for controlled fabrication of multilayers*

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## Abstract

A detailed description of a sputter deposition system constructed specifically for the fabrication of x-ray and neutron multilayer monochromators and supermirrors is given. One of the principal design criteria is to maintain precise control of film thickness and uniformity over large substrate areas. Regulation of critical system parameters is fully automated so that response to feedback control information is rapid and complicated layer thickness sequences can be deposited accurately and efficiently. The use of either DC or RF magnetron sources makes it possible to satisfy the diverse material requirements of both x-ray and neutron optics.

## I. Introduction

There is, at present, a growing demand for multilayers as monochromators in both x-ray and neutron scattering studies of condensed matter. This is particularly so at Brookhaven National Laboratory where two major sources exist, the National Synchrotron Light Source (NSLS) and the High Flux Beam Reactor (HFBR).

In order to utilize multilayers as monochromators, accurate and uniform deposition of particular sequences of thin film layers over large surface areas is required. In the x-ray case, a particular layer thickness sequence must be used in order to minimize the absorption which occurs predominantly in the high-Z layer.[1] For neutrons, absorption is usually not a problem, but hundreds or even thousands of layers must often be deposited according to a gradually varying thickness algorithm in order to increase the angular acceptance for a given wavelength.[2] In both cases, random fluctuations in film thickness can result in destruction of coherence and consequent decrease in reflectivity. Control over layer thickness variations is also crucial for a second type of multiple thin film device for neutrons, known as a supermirror, which is composed of a sequence of bilayer thicknesses that in effect extends the region of total mirror reflection beyond the ordinary critical angle.[3] Another requirement for high reflectivity is that the two bilayer materials chosen have a large difference in refractive indices. High- and low-Z materials such as tungsten and carbon give a good match for x-rays. For neutrons, nickel and titanium, where the scattering is predominantly nuclear and/or magnetic in origin, are suitable.

In the following Section, a versatile deposition system will be described which has been built to satisfy as closely as possible the above criteria for fabricating efficient multilayer monochromators. Finally, in Section III, future planned capabilities of the system are discussed, in particular regarding the sputtering of ferromagnetic materials such as iron which are of importance in making polarizing multilayers for neutrons.

## II. Description of deposition system

A photograph of the deposition system is shown in Fig. 1 and a schematic diagram of the principal sections is shown in Fig. 2. The deposition system consists of a central stainless steel box chamber 75 cm wide by 75 cm high by 90 cm deep with cylindrical extensions 30 cm in diameter by 75 cm long on both sides. The sputtering components are contained within the central chamber. The side extensions are required to allow translation of long mirror substrates past both sputtering targets. The cylindrical extension on the right side also contains a roughing port for pumping the chamber down from atmospheric pressure. At the front and back of the central chamber are large doors for access to the sputtering components and the mirror substrate. The top of the central chamber has connections for a water vapor cryopump and gas inlet valves. The bottom of the chamber connects to the main vacuum pump system which includes a turbomolecular pump and foreline pump. The gas and pressure monitoring sensors are also located on the bottom of the chamber.

### Pumping system

The chamber volume is roughed from atmospheric pressure to 50 milliTorr in about 5 minutes with a Welch 1375 (1000 liter/min) rotary mechanical pump. Upon attaining the base roughing pressure, the roughing pump is isolated and the high vacuum line opened. High vacuum is achieved with a Welch 3133 (1500 liter/sec) vertical turbomolecular pump backed by a Welch 8851 (510 liter/min) direct drive mechanical pump. The time required to reach the $10^{-5}$ Torr range depends on the humidity conditions during the preceding open chamber condition. In order to minimize pumpdown time, argon gas is used to backfill the chamber to atmospheric pressure before opening the door. During the pumpdown cycle, a Polycold PFC-500 (43,000 liter/sec) fast cycle water-vapor cryopump is activated at a pressure of 2 x $10^{-5}$ Torr. The chamber pressure subsequently drops to approximately 3 x $10^{-6}$ Torr. Near the end of the pumpdown cycle, the high vacuum valve is closed and the chamber interior is scrubbed with a glow discharge. The ultimate high vacuum base pressure reached in the system is about 1 x $10^{-6}$ Torr. During the sputtering operation, argon gas is continuously leaked into the system and the chamber pressure dynamically maintained at 2 milliTorr. In order to avoid overheating of the turbomolecular pump rotor due to gas friction, the high vacuum valve is positioned partially closed and the turbomolecular pump is operated in the half speed rotor condition.

Gas supply and pressure monitoring

Prepurified argon gas (99.998%) is used for sputtering. The gas flow and chamber pressure is controlled by a closed-loop MKS Baratron system. The control system makes use of a capacitance manometer for measuring the chamber gas pressure and a Granville-Phillips metal-sealed servo valve to regulate the argon gas flow. Additional pressure gauges (i.e., ionization, thermocouple, Shultz-Phelps, and convectron) are used to monitor different sections of the vacuum system during different conditions of operation. The partial pressures of the gaseous components in the main chamber of the vacuum system are identified and monitored with a Varian (VGA-100) quadrupole residual gas analyzer. The analyzer is especially useful for locating real and virtual leaks.

Substrate translational system

The multilayer substrates are held in place on a stainless steel carriage. The carriage is connected via machineable ceramics (Macor), V-guide bearings and tracks from the Bishop Wisecarver Corp. The ceramic parts make it possible to isolate the carriage both thermally and electrically from the chamber. Thus, the carriage can be either heated or cooled and/or operated with an electrical bias during the coating process. The tracks, which are approximately 240 cm long, are held in place along the inside walls of the chamber and side extensions. The V-groove rollers and tracks are easy to assemble and allow motion in vacuum with minimal friction. Both ends of the 63 cm long carriage are attached to a closed loop, stainless steel, 6.35 mm pitch drive chain. The chain is driven at the far end of one chamber extension with a sprocket and drive shaft which is attached to a Ferrofluidic rotary feed-thru. The far end of the opposite extension has a free-rolling sprocket under tension which completes the chain drive system. The chain, tracks and V-groove rollers are shielded from the sputtering targets in the main chamber, thus preventing any material build-up. Translational carriage speeds between 5 cm/min and 150 cm/min are anticipated for the different material sputtering rate and bilayer thickness combinations that are contemplated. The drive for the substrate translation system is provided by a Slo-Syn DC stepping motor which is activated by a Superior Electric MITAS intelligent motion controller. The controller monitors and controls the carriage position and speed. End point sensors are placed at both ends of the track to define the physical limits of the carriage travel.

Sputter deposition

The configuration of the multilayer substrate and the material to be sputtered (sputtering source) is one of parallel vertical planes. In making bilayers, two different material sources are located in the same plane and spaced along the direction of motion of the substrate. The substrate and source planes are about 5 cm apart. The planes are vertical in order to prevent settling of particulate matter on the substrate and/or source during deposition. The sputtering sources used are MRC 8" diameter, circular planar magnetrons. A flat aluminum ground plane with apertures opposite the circular sources is located between the sources and the substrate. The apertures are shaped to provide a uniform coating thickness over the entire width of the mirror substrate. Each of the two magnetron sputtering sources is powered by a separate SPS-5000 DC power supply from RF Plasma Products. The power supply is capable of operating in either a constant voltage, constant current or constant power control mode. The power levels applied to each of the two sputtering sources are set so that the individual sputtering rates provide the necessary bilayer thicknesses for a given rate of substrate translation.

The regulation of substrate temperature is an important factor regarding interdiffusion, crystallinity and adhesion of the deposited multilayers. Here the reduced electron heating associated with magnetron sources is an advantage, allowing substrate temperatures to be maintained more independently. The use of DC and/or RF magnetron sources allows refractory, insulating and even alloy materials to be deposited at relatively high rates and degree of control.

Monitoring of coating thicknesses

The thickness of each coating of the multiple bilayer at any location along the length of the mirror substrate is determined by the instantaneous rate of deposition and rate of substrate translation as that point passes in front of the particular sputtering source. If both these rates are kept at predetermined levels, the desired uniform bilayer thickness sequence will be achieved. During the multilayer fabrication, the deposition rate is dependent mostly upon the material sputtering rate and to a lesser extent on the ambient argon pressure. In order to achieve a constant deposition rate throughout the duration of the multiple bilayer fabrication process, it is important to monitor and control both the power applied to the sputtering targets and the ambient argon pressure in the main chamber. The d.c. magnetron power supplies are operated in a constant power control mode. The gas flow/pressure control system is operated in a constant pressure mode. Located opposite each sputtering source, and just above the translating mirror substrate, is a stationary quartz crystal sensor to monitor the deposition rate of that sputtering target. Both sensors are equipped with a pneumatic shutter assembly in order to allow periodic sampling and prevent excessive coating buildup on the sensors. The output from each sensor is fed into a separate film monitor/controller.

Process control system

The process control system is shown schematically in Fig. 3. A Hewlett-Packard 217 minicomputer is the heart of the process control system. The minicomputer communicates with the two Inficon XTC thin film monitor/controllers over the IEEE 488 (HPIB) interface bus. The minicomputer communicates with the Superior Electric MITAS motion controller system over an RS232 link. The MITAS controller has built-in intelligence to allow the drive motor operation to be programmed independently of the computer for test purposes. The minicomputer also communicates with the two SPS-5000 D.C. power supplies via the HPIB interface.

During the deposition process, the computer monitors both target material deposition rates, both sputtering power levels, the argon plasma pressure, and the substrate carriage position and speed. From these inputs and the software program entered into the computer, adjustments in carriage speed and/or sputtering power levels are made.

A commercial thin-film design program, FILM*STAR,[4] is used to design the multilayer coatings and provide the capability to model the performance of the films under a variety of test conditions, such as at visible wavelengths and at variable angles of incidence.

## III. Future capabilities

As a diagnostic for x-ray multilayer fabrication, an in-situ soft x-ray reflectometer system is planned to be added in the future. The reflectometer assembly will be located between the two sputtering heads, in the center of the main chamber. The reflectance of the multilayer will be measured as a function of the substrate scan direction. This will permit the reflectivity of the multilayer to be monitored on a layer-by-layer basis, and the resulting values compared with calculated expectations.[5]

It is possible to polarize a neutron beam with multilayers by choosing one film material to be ferromagnetic and saturating it with a magnetic field applied perpendicular to the momentum transfer vector.[6-8] Polarizing multilayers are valuable since high quality, conventional polarizers, such as Heusler alloy single crystals are not widely available. The ferromagnetic material most often used for such polarizing multilayers is iron. Unfortunately, iron cannot be sputtered in an ordinary magnetron source. Although iron can be readily sputtered in a diode arrangement (and also at low pressures if an RF potential is applied), it is desirable to obtain the higher sputtering rates and lower substrate heating possible with a magnetron. A special design with a reconfigured magnetic field is now being developed to achieve this goal.

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## Biography

Robert P. Di Nardo received his Mechanical Engineering degree and M.S. and Ph.D. degrees in Physics from Stevens Institute of Technology. He worked at C.B.S. Laboratories/EPSCO Labs for five years where he developed thin film processes required to fabricate electrical, optical, and lubrication coatings. He spent two years with Electron Systems and Technology where he was involved with the materials and vacuum processing of electron multiplier tubes. For the past six years, he has been with the Instrumentation Division of Brookhaven National Laboratory where he provides special thin film coatings for particle, optical, and x-ray instrumentation. Dr. Di Nardo is a member of Sigma Xi.

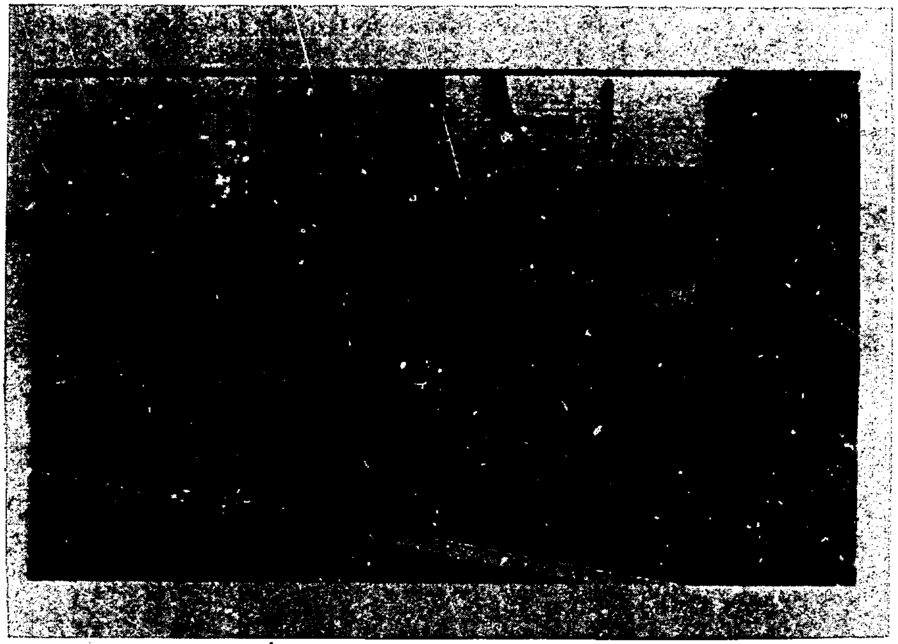

Fig. 1. Photograph of deposition system

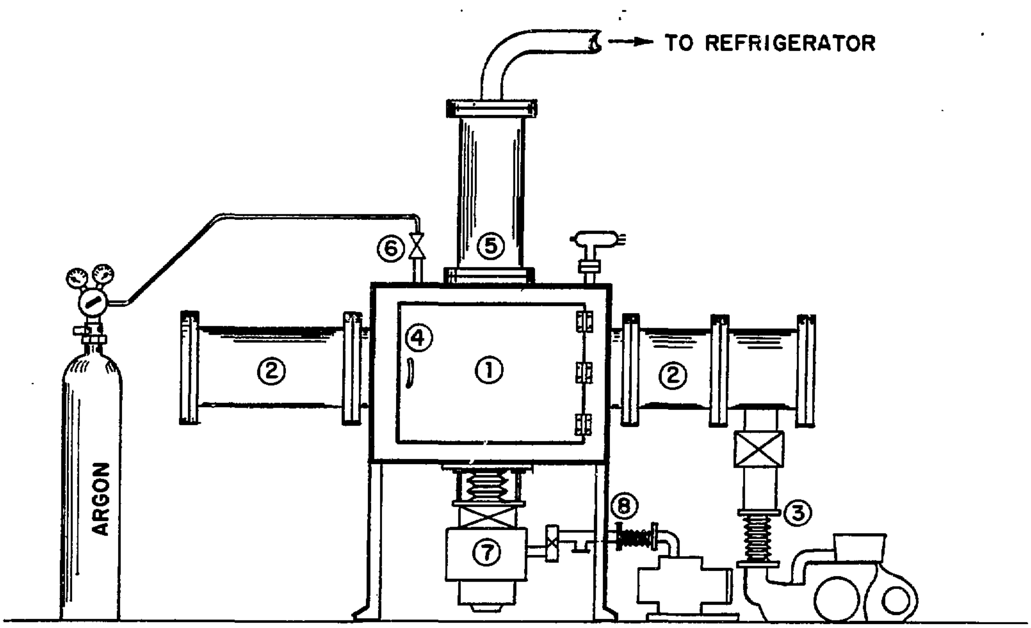


Fig. 2. Diagram of deposition system. 1. central chamber; 2. side extensions; 3. rough pumping section; 4. entrance door; 5. water vapor cryopump; 6. gas inlet; 7. turbomolecular pump; 8. foreline pumping section

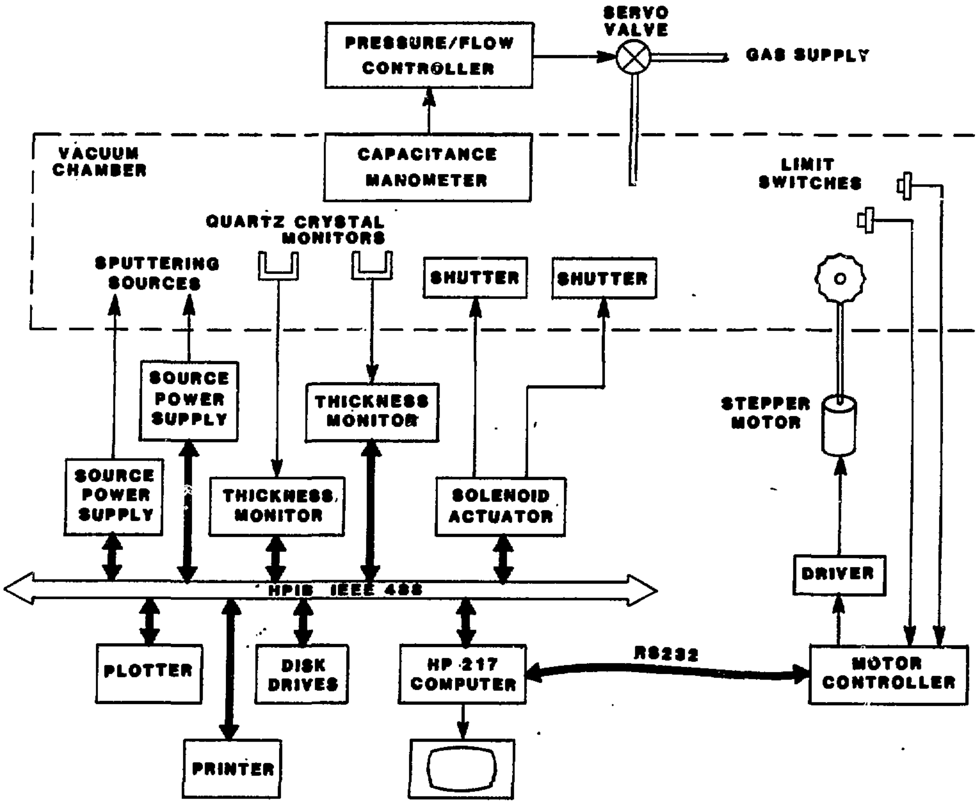


Fig. 3. Schematic diagram of process control system