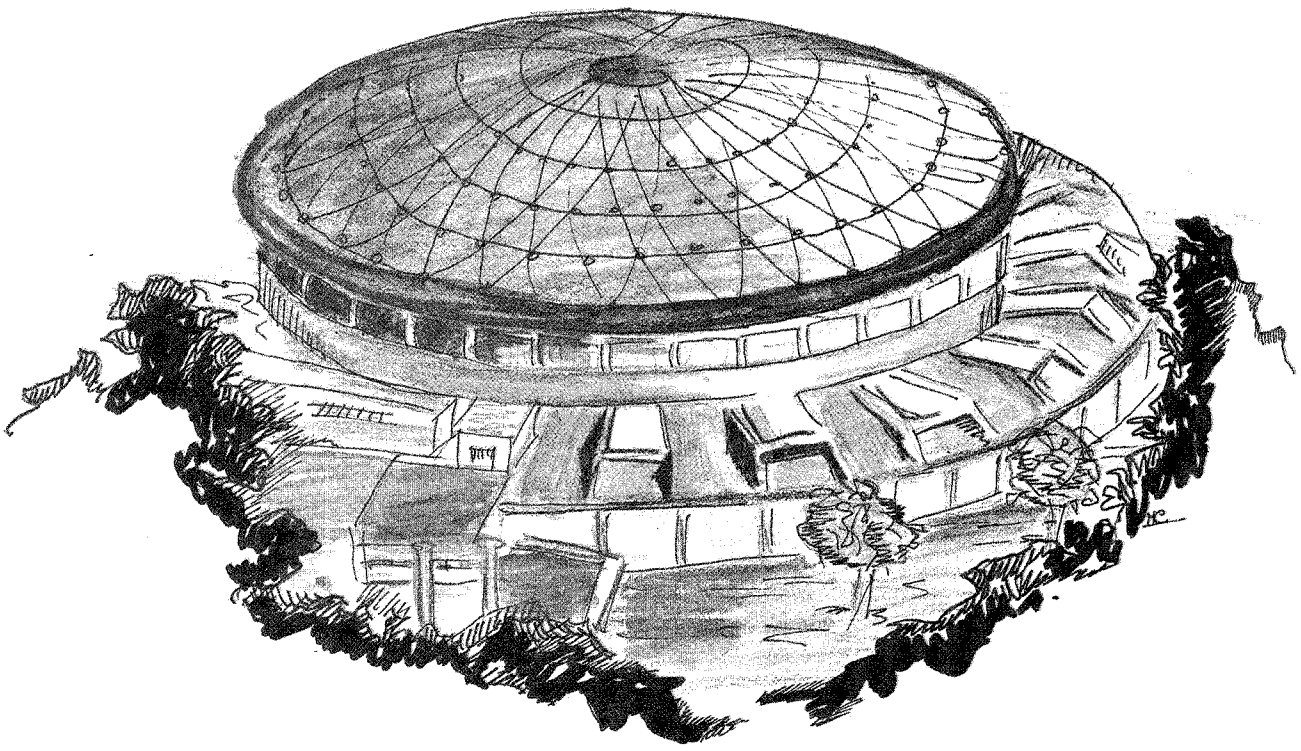
Laboratori Nazionali di Frascati

LNF-88/30(R)
1 Giugno 1988

I. Davoli, Zhu Zhi Ji, P. Chiaradia, S. Priori, M. Fanfoni:

A GLOW DISCHARGE PROCESS FOR OPTICAL ELEMENTS CLEANING



Servizio Documentazione
dei Laboratori Nazionali di Frascati
P.O. Box, 13 - 00044 Frascati (Italy)

ISTITUTO NAZIONALE DI FISICA NUCLEARE - ISTITUTO NAZIONALE DI FISICA NUCLEARE - ISTITUTO NAZIONALE DI FISICA NUCLEARE - ISTITUTO NAZIONALE DI

INFN - Laboratori Nazionali di Frascati
Servizio Documentazione

LNF-88/30(R)
1 Giugno 1988

A GLOW DISCHARGE PROCESS FOR OPTICAL ELEMENTS CLEANING

I. Davoli
INFN - Laboratori Nazionali di Frascati, P.O.Box 13, 00044 Frascati, also Dip. di Matematica e Fisica, Università degli Studi di Camerino - 62032 Camerino (Italy)

Zhu Zhi Ji
International Centre of Theoretical Physics, P.O. Box 586, Miramare 34100 Trieste, (Italy)

P. Chiaradia, S. Priori, M. Fanfoni
CNR - Istituto di Struttura della Materia, Via E. Fermi n. 38, 00044 Frascati, (Italy)

ABSTRACT

We report the results of a successful attempt made in the Laboratori Nazionali di Frascati for glow discharge cleaning of a glass artificially contaminated with carbon and of a monochromator grating exposed for long time to soft X-ray and VUV radiation. We also report the dependence of the cleaning rate with respect to the position along the stream of oxygen-water vapour mixture.

INTRODUCTION

People dealing with synchrotron radiation (S.R.) must sooner or later face the problem of the *in-situ* cleaning of optical components. Mirrors and gratings, especially the ones used in the low energy region of the S.R. spectrum, are located inside the pipes and work in the same vacuum conditions as the storage ring. The lack of a vacuum totally free of hydrocarbons and the cracking process induced by the UV and soft X-ray radiation produce a coating of graphitic carbon on the optical elements. This graphitic deposition appears as a brownish shade left on the optical elements which with the time reduce their reflectivity. Then a sort of mean life-time characterizes the optical elements. A special care in maintenance operations can extend the life-time of gratings and mirrors,

but does not avoid the contamination process. The situation is aggravated in the case of not fully dedicated machines because S.R. users, in order to utilize more beam time, work even when the vacuum in the Storage Ring is not at the best level and the contamination rate is increased.

Following the work made in the USA[1] and Japan[2], we have set up an apparatus for the cleaning process based on the idea that a radio frequency discharge, in an atmosphere formed by a mixture of oxygen and water vapour, is able to remove a consistent part of carbon contamination without any alteration of the optical surface.

EXPERIMENTAL

The experimental set-up consists mainly of two parts: a low-vacuum receptacle filled with humid oxygen atmosphere and the radio frequency apparatus which provide the required power for the glow discharge.

The vacuum chamber scheme is reported in Fig. 1. It is formed by a 6" flanged nipple modified with a $2"^{3}/_{4}$ glass window inserted orthogonally to the axis of the chamber for inspection. On the end flanges there are two adaptors. The first one connects the chamber with the pumping system which consists of a service cross holding a low-vacuum gauge (Granville-Phillips) and another view port, of a cold trap and finally of a rotary pump. The second adaptor connects the sample chamber with a mini-conflat tee used for the inlet of the oxygen-water vapour mixture and for the mini-conflat flange of the pyrex cavity.

The radio frequency (R.F.) apparatus consists of a wave generator (Phillips P.M. 5134) working in a sinusoidal regime between 1 and 100 MHz which drives the R.F.-amplifier (EIN 3100 L) both in intensity and in frequency. The R.F.-amplifier is characterized by an impedance of 50 Ω and a maximum output power of 100 W. In series to the amplifier there are a bidirectional Wattmeter (Bird model 43) to measure the percentage of the transmitted power and a matching network whose electric scheme is reported in Fig. 2. Finally two copper electrodes wrapped around the pyrex cavity end the electronic chain.

The most critical part of the experiment is the R.F. transfering. Indeed without a correct matching we do not produce any glow discharge. The matching network that we have used allows the substitution of the inductance in case of gross mismatching. For a fine adjustment we may use two variable capacitors. A particular care should be also devoted to the cooling of the electrodes because they reach quite a high temperature (200° or 300°C) and may cause vacuum leaks on the pyrex cavity; in our case we have used a forced ventilation which brings the electrodes to an acceptable temperature without any alteration of the impedance matching.

The procedure of the experiment is quite simple: we start out by emptying the receptacle to our best possibilities (≈ 0.5 Pascal), then we open the leak valve of the gas pipe and let oxygen flow in the chamber. To have the humid atmosphere we let oxygen pass through a water cell and collect some water vapour. The humidification is required to improve the cleaning rate. The quantity of water vapour, as reported in ref. 3 should be of about 2% of the oxygen inlet. In spite of the fact

that we were not able to measure the water percentage in the mixture, we got positive results and this suggests that value of this parameter is not crucial. When the pressure is of about 30000 Pascal and a balance between the pumping speed and the gas inlet is obtained we turn on the R.F. tuned at 13.5 MHz and slowly increase its intensity until the discharge gets a characteristic cream colour. A typical time for cleaning the glass, that was artificially contaminated with carbon using a candle flame, was one working day and the results were comparable with those presented in the literature.

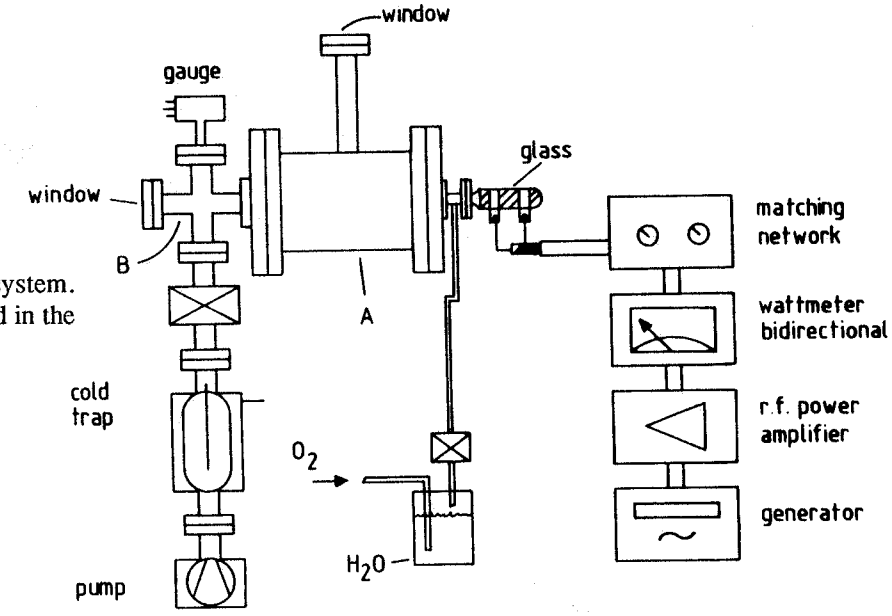
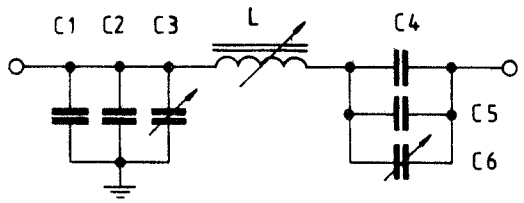


FIG. 1 - Scheme of the RF system. Further explanations are reported in the text.



C1= 220 pF
C2=470 pF
C3=4÷30 pF
C4=47 pF
C5=22 pF
C6=4÷30 pF
L= 4 μH

FIG. 2 - Matching network for R.F. power transfering. The inductance can be easily replaced in case of a gross mismatching. On the contrary for fine adjustments it is possible to use the two independent variable capacitors C_3 and C_6.

In Fig. 3 the result of a cleaning process for a period of eleven hours on an artificially contaminated glass is reported. The glass slide was broken in two pieces after the contamination. The first piece has been processed while the second has been kept untreated for comparison. We also checked the optical reflectivity of these two pieces in a flat portion of the visible region. The ratio of the reflected intensity between the clean part and the smoked part was larger than 80. In Fig. 4 we report a comparison of the cleaning process for slites put in two different positions along the gas stream. The piece 1 was placed in position B (see Fig. 1) while the piece 2 was placed in position A. After 11 hours of the described treatment the second piece was practically unaffected by the cleaning process and its reflectivity increased only by a factor 1.6. This indicates that in order to clean an optical element, one must place it just along the stream of the oxygen-water mixture. This

is very important for the choice of the correct position of the pyrex cavity and of the pumping system in the vacuum chamber where the *in-situ* cleaning should be performed.

Finally in Fig. 5 we report the pictures of a replica grating before (A) and after (B) a cleaning process of 13 hours. The cigar shaped shade due to carbon contamination that appears on the grating was completely removed after the treatment. On the contrary, the rectangular blot which was not removed at all, is probably due to a mechanical alteration of the surface. Indeed the blot was not on the radiation path.

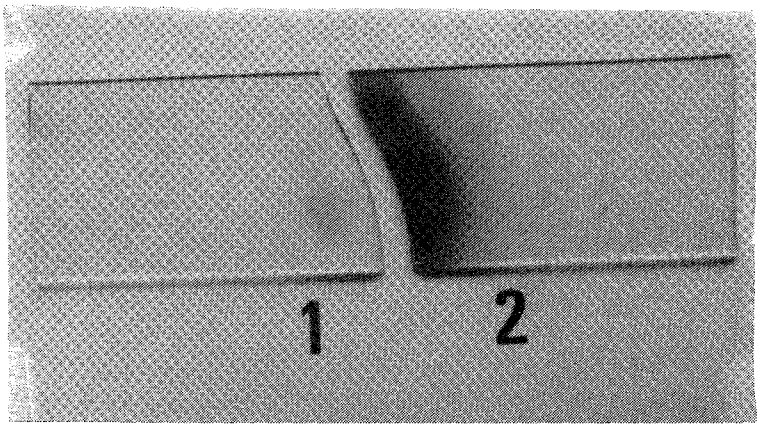


FIG. 3 - Direct evidence of the cleaning process. The original piece of glass was artificially contaminated with carbon and then broken in two pieces. The one on the left was kept under glow discharge for 11 hours.

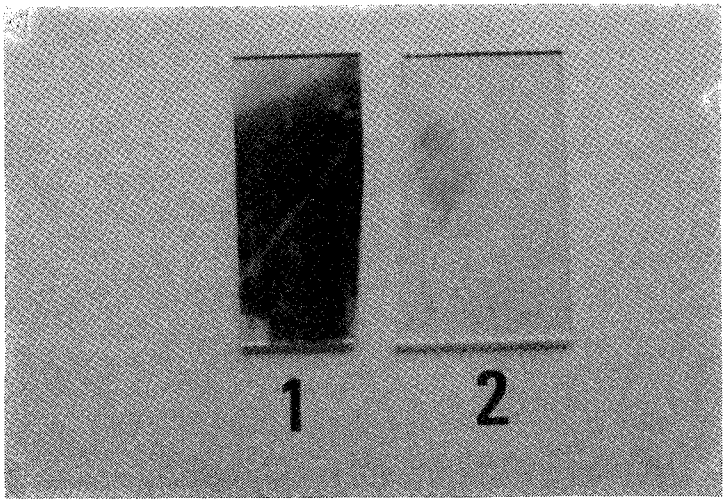


FIG. 4 - Direct comparison of the cleaning process for two different positions with respect to the molecular stream. Two artificially contaminated glass samples were placed in positions B (label 1) and A (label 2) of Fig. 1, respectively.

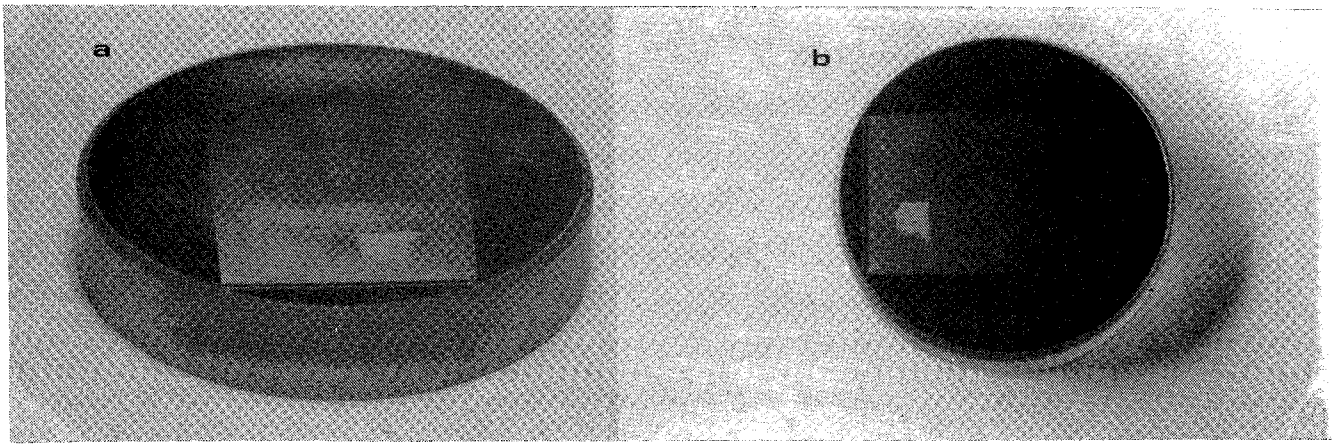


FIG. 5 - Glow discharge cleaning of an holographic grating.

FUTURE RESEARCH AND APPLICATIONS

The apparatus just described was assembled with spare parts of other equipments and therefore it was not optimized for our purposes. Obviously any further development would require a properly designed apparatus. In particular we think that the following aspects should be more thoroughly pursued: 1) the optimization of the cleaning rate by varying the gas composition and in particular the water vapour content; 2) the optimization of the cleaning process by varying the sample position with respect to the molecular stream and the chamber geometry and 3) the implementation of a simple method for measuring the reflectivity during the cleaning process. Of course the major interest of this work lies in the possibility of *in-situ* cleaning of the optical elements in the synchrotron radiation beam lines. In this way labour and time are saved and the difficulties of beam realignment are avoided. In our opinion this goal can be achieved with the method we have investigated, provided care it taken of the geometrical aspects.

ACKNOWLEDGEMENT

We would like to thank Ing. R. Boni and the staff of the R.F. group of ADONE for technical support and Dr. V. Chimenti for useful advices.

REFERENCES

(1) W.R. McKinney, P.Z. Takacs. "Plasma discharge cleaning of replica gratings contaminated by synchrotron radiation". Nucl. Inst. and Meth. 195, 371 (1982) and reference therein.
(2) T. Koide, S. Sato, T. Shidara, M. Niwano, M. Yanagihara, A. Yamada, A. Fujimori, A. Mikuni, H. Kato and T. Miyahara. "Investigation of Carbon Contamination of Synchrotron Radiation Mirror". Nucl. Inst. and Meth. in Phys. Res. A246, 215 (1986).
(3) E.D. Johnson, S.L. Hulbert, R.F. Garrett, G.P. Williams, M.L. Knotek. "In-situ Reactive Glow Discharge Cleaning of X-Ray Optical Surfaces". Rev. Sci. Instrum. 58, 1042 (1987).