# Heat-Reflecting (Oxide-Metal-Oxide) Coating Technology Based on Magnetron Sputtering Method

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**Abstract – For industrial production of heat-reflecting coating on architectural glass, "TOPaz – 3M" plant was manufactured with lockage of two-side dispensing cassette of glass and system of transportation through working chamber. Maximum size of glass plate is 2750×1605. The Plant has automatic monitoring and management system of vacuum and technological equipment performance. Cycle duration is 20–25 min in "mirror" mode, 40–60 min in "low emissive (Low-E) glass $TiO_2$" mode, 25–30 min in "Low-E glass $SnO_2$" mode. For deposition technology the following were used: 2 band-shaped ion sources for cleaning of surface, 2 main magnetron sputtering systems (MSS) with cathodes Ti, Sn and loop magnetron for precise coverage of Ag film simultaneously on two surfaces. Optical thickness was measured *in situ* by monochromatic refractometer. Technical characteristics of Plant, technological parameters of TOP coverage with composition $TiO_2$-Ag-$TiO_2$, $SnO_2$-Ag-$SnO_2$, as well as optical features of resulting multiple coatings in visible and infrared band are represented.**

## 1. Introduction

Magnetron vacuum plants with sluicing and displacement of goods relatively to stationary technological equipment are more widely used for large area production of optical multilayers by average manufacturers, aiming at satisfaction of needs of minor region, city. This schematic solution allows using developed systems of technological equipment without restrictions in weight-overall dimensions, provides stability of processing environment and accessibility of close control. Module structure of such systems allows producing of 50–100 thousand square meters of plate glass a year in minimum launching configuration, and provides the possibility of further upgrading and modernization. [1].

Plant construction for TOP deposition of MeO-Me-MeO type must provide the following technological procedures:

– Ion cleaning, preparation of surface;

– Deposition of adhesion oxide sublayer;

– Deposition of metallic mirror;

– Deposition of antireflective oxide layer.

Basic aspects of technological solution of the task are in reflector nanostructure stabilization (usually, argentums films of some nanometers wide), as well as optical parameters and coating oxide structure control.

Nanostructural metallic films are prone to destabilizing factors. In the first place, they are diffusion and chemical interaction with substrate material – glass. During deposition processes are activated by heating and corpuscular influence on surface (basically – by electronic).

Antireflective layer is achieved due to both traditional obstacles of sputtering in oxidant medium [2]: low processing speed, oxidation and spark formation on cathode, anode "poisoning", and a range of specific conditions in gas medium of large area production.

Below, we offer a variant of not expensive, highly technological plant, which allows processing multiple depositions in automatic mode.

## 2. «TOPaz» Description

Basic configuration of «TOPaz» Plant [3] includes the following main units:

1. Working chamber;
2. Sluicing chamber with slit shutter;
3. Accumulating pocket;
4. Feeding assembly for 2 cassettes;
5. Feeding cassettes – 2;
6. Cassette displacement system;
7. Band-shaped sources of gas ion –2;
8. Line magnetron – 2;
9. Loop magnetron for two surfaces;
10. Dual gas feeding system;
11. Optical control unit;
12. Cooling system;
13. Multi controller system of management;
14. Power unit for magnetron discharge – 2;
15. Power unit for ionic sources – 2;
16. High-vacuum system based on АВПМ-400 and on fore pumps НВР-16Д – 2.
17. Bypass vacuum system (НВР-250Д);
18. Managing computer.

Power sources of Magnetron discharge with micro controller management provide stabilization modes of capacity or discharge current, stabilized current feeds ionic sources.

Control system allows cycle processing: from feeding cassettes with glass plates to outcome of complete product – in automatic mode. It sets modes of plant operation, monitors and locks emergency accidents and provides automatic implementation of technological program on goods processing. Managing Computer Interface has a user-friendly procedure of design and adjustment of technological modes. Program stores Plant processing protocol, it has technologies library, production information.

Plant provides the following modes:

– Cleaning and activation of substrate by the flow of accelerated ions.

– Deposition of metal films by line magnetrons.

– Precise deposition of nanostructural films by loop magnetron.

– Deposition of coating in the result of reactive sputtering with control of optical thickness.

– Deposition of magnetron coatings in the mode of ion assistance.

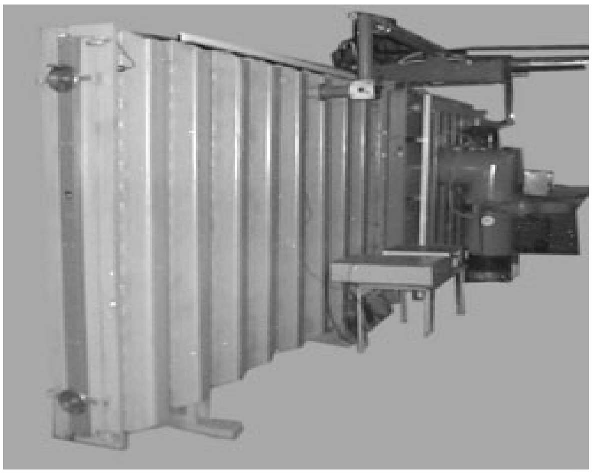

Fig. 1. "TOPaz" Plant for Large Area Production of Optical Multilayers

Combination of these modes in technological program allows successful using of Plant for manufacturing of high quality mirrors, interferential-colored and low emissive architecture glass, other special coatings.

Small volume of locking chamber from processed stainless steel (0.46 м$^3$) and specially designed high vacuum slit shutter of "TOPaz" Plant provide minimum locking time and stability of vacuum environment in the processing area

Fundamentally the Plant and Control System Construction has module structure that is why basic arrangement can be upgraded with the purpose of output increasing and/or obtaining new technological characteristics.

Working chamber Construction provides easy access and convenience of maintenance of technological equipment. Usage of cathodes with increased stock of materials ($S = 14$ mm) in the construction of main magnetrons makes one set enough for production of 6000–7000 m$^2$ of mirrors, ≈ 10000 m$^2$ of toning, ≈ 7000 m$^2$ heat-reflective coatings. Composing cathode of loop magnetron provides simplicity of its production from expensive materials (Ag, etc.), as well as a high use factor ≈ 50%.

Fig. 2. «TOPaz» Plant Working Chamber

Table 1. Basic Functional Parameters of MSS Plants

| Basic characteristics | TOPaz |
|---|---|
| Scale | Industrial |
| Maximal overall dimensions of the sheets | 2.750×1.605 (4.16 m$^2$) |
| Number of loaded units | 2 |
| Total area of processed sheets, m$^2$ | 8.32 |
| Maximal thickness of glass plate, mm | 8 |
| Number of MSSs | 3 |
| Preparation of the surface | Ion gun |
| Stabilization of DC discharge | $I$, $W < 2\%$ |
| Stabilization of gas medium | flow rate |
| Monitoring of film parameters | monochrome refract meter |
| Vacuum system control | automatic |
| Control of technological process | automatic |
| Film thickness equability over the processed area, % | < 3 |
| Volume of slues chamber, m$^3$ | 0.46 |
| Volume of working chamber, m$^3$ | 1.23 |
| Residual pressure limit in the working chamber, Pa | $1.3\times10^{-3}$ |
| Scanning speed, m/s | $10–50\times10^{-3}$ |
| Rate of film concretion (tin target, $SnO_2$ loading), nm×m/s | 0.3–3 |
| Average performance, m$^2$/h | 35 |
| Power supply voltage (3-phase, 50 (60) Hz), V | 380/220 |
| Power consumption, kW | 67 |
| Cooling water discharge (20 °C), m$^3$/h | 1.2 |
| Total weight, kg | 5370 |
| Occupied area, m$^2$ | 75 |

Composition of coatings: nonmagnetic metals, their oxides, nitrides etc.

There are the following elaborated options of upgrading:

– usage of automatic ellipsometer for optical control;

– a scope of cathode units for different types of targets: cylindrical, direct cooling, segment, fused;

– dual magnetron systems on the basis of line and loop cathode units;

– magnetron power sources of medium frequency (MF) bipolar asymmetric and symmetric, 20 kW.

Technology of heat-saving coatings production on glass plate with composition $TiO_2$-Ag-$TiO_2$ и $SnO_2$-Ag-$SnO_2$ was polished on the "TOPaz" Plant with basic configuration.

Additionally the usage of asymmetric bipolar power sources MF for oxides deposition was investigated.

### 3. Deposition Technology for Multilayer Coatings

Technological program of "TOPaz" Plant is a map of succession of modes – operations. Each line sets the condition for the beginning of process, gas parameters, kind and mode of power sources, condition for the end of process: time, number of passings and parameters of optical control system.

Typical succession of heat-reflecting coating was the following:

1. Locking of the cassette with goods till the set level of residual pressure.
2. Gas feeding and training of ion sources.
3. Ion cleaning and preparation of glass plates.
4. Training of MPC in reactive sputtering mode.
5. Deposition of oxide sublayer.
6. Training of loop MPC in argon environment.
7. Deposition of metallic layer Ag.
8. Displacement MPC in reactive sputtering mode.
9. Deposition of antireflecting oxide coating with set numbers of passings.
10. Complete deposition of oxide till set thickness with optical control.
11. Cassette parking, depressurization of lock.

To optimize characteristics of technological map the following process were investigated:

– Ion processing influence on adhesive and chemical resistance of coatings.

– Dependence of composition and homogeneity of deposited films on electro physical characteristics of the process.

– Influence of partial composition and gas type on film characteristics by reactive sputtering.

– Space discontinuity and dynamical processes of changing plasma-generating environment.

– Influence of deposition parameters on stability of metallic layer.

Basing on functional requirements to coatings, the thickness of Ag-mirror ~ 6–8 nm (~ 30% reflection in optical band) was examined. The research showed, that almost all the parameters of deposition operations influence its stability to some extend. The following criteria were put in the basis of optimization:

a. Total destruction of film structure – quantity and density of visible defects;

b. Slowdown of coating conductivity in the result of tribological trials.

Analysis of results showed that fluence and density of ion cleaning power are restricted from the top by the growth of the defects of glass in the result of sputtering with ions and trickling of surface discharge. Besides, there was lowering of structure stability as the result of deposited Ag film at high range of prior processing. This can be the result of increasing of free active radicals in near-surface area of glass. Best values were achieved at 3–5 kJ/m$^2$ and < 50 W/m$^2$.

Oxide sublayer significantly stabilizes Ag, the degree of film oxidation (value of partial pressure of oxidant) being more significant than its thickness.

Apart from oxides the variants of Ag stabilization by subnanolayers 1–2 nm on the basis of Ti, NiCr, TiN were examined. Positive effect was observed in all cases, but for Ti, NiCr there was noticeable lowering of coating transparence. Best result was achieved at usage of titanium nitride; however, the difference is not significant in comparison with oxides.

Deposition parameters of Ag film itself define the quality of the whole coating. Loop magnetron due to high degree of plasma localization (width of erosion area ≈ 10 mm) provides high local capacity of Ag sputtering at minimum transport distance of targets – substrate. From the one hand, it helped to provide the high quality of film, from the other – controlled speeds of nanolayer deposition.

Coating layer deposition influences the mirror due to electronic bombing and substrate heating. This factor is the most important in deposition of $TiO_2$ film, requiring high capacity and duration of process. Positive result was achieved by measures in increasing of magnet system equilibrium and lowering of electronic influence on substrate by additional electrodes.

Antireflective layer production is the longest process in technological cycle. It is defined by layer thickness, low speeds of oxides deposition, high demands for homogeneity. $O_2$ and $CO_2$ were investigated as an oxidant additives. Usage of carbon dioxide provides smoother transfer from oxide deposition mode to metal. This helped to increase the $TiO_2$ deposition speed on 70–90%. However, as it was mentioned, increasing of deposition speed by increasing of discharge capacity was restricted by stability of argentums sublayer.

At conducting a series of technological cycles connected to successive deposition of $TiO_2$ an effect of "poisoning" of anode appeared, which revealed in continuous increase of discharge voltage till maximum. This required the introduction into cycle of operation on anode regeneration by deposition of metallic titanium.

The other clear effects at $TiO_2$ deposition were dynamic processes, connected to change of speed of oxidant consumption according to the position of substrate. Fig. 3 shows the change of general pressure, connected with cycles of substrate disposition.

In most cases corresponding change of discharge impedance could be compensated by stabilization of capacity (Fig. 4), however, in modes with low partial pressure heterogeneity of optical thickness was observed.

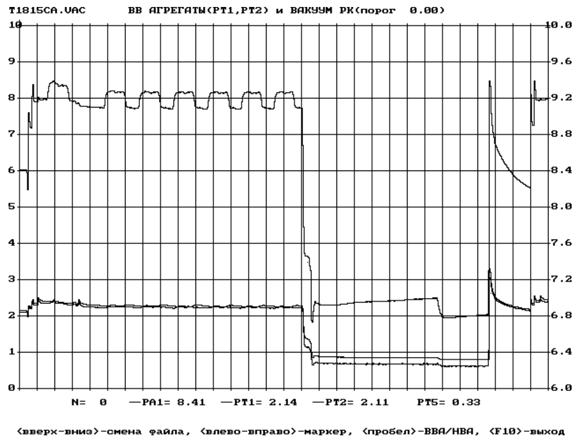


Fig. 3. Influence of substrate disposition on the pressure in working chamber

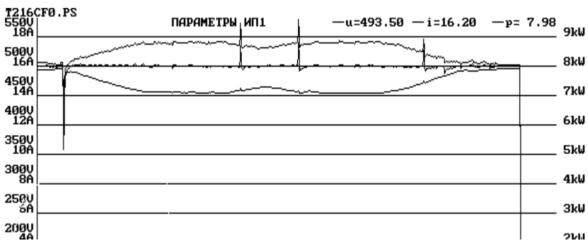


Fig. 4. Change of discharge impedance

Usage of $SnO_2$ film as an interferential coating allowed significant lowering of processing time and negative influence on argentums layer. However, some decrease in chemical and tribological durability of $SnO_2$–Ag–$SnO_2$ does stay within the limits needed by TOP. Negative factor is inclination of Sn – cathode for spark breakdowns in reactive sputtering modes and, due to low melting temperature, explosive energy of its surface.

Usage of discharge on Sn – cathode of asymmetric bipolar source of MF for feeding allowed excluding breakdowns in the whole range of gas compositions. Source working frequency is 30–60 kHz, plus pulses duration ≈ 2 mcs.

Optical characteristics of coating in infrared band were investigated on spectrophotometer "Specord M80" in the mode of reflection at angle of 70°. The research works allowed developing technological processes of achieving of Low-E coating on glass plates for TOPaz plant, having IR-reflection in a range between 85 and 96% by corresponding transmission factor in visible area between 94 and 86%.

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