Vacuum Equipment for TCO and AR Coatings Deposition by Reactive Magnetron Sputtering

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Tasks of the work

- Obtain similar initial conditions on substrate for coating deposition
- Find new possibilities for reactive magnetron deposition of TCO
- Develop control method used in longtime reactive deposition process

Tasks of the work

- Obtain stability of gained coatings parameters during long time cycles
- Design new generation of vacuum machines for TCO and oxides for AR layer deposition

Influence of initial film drying and outgasing on ITO coatings

Sources of water:

- Adsorption water on chamber walls and on in-chamber devices
- Absorption water
- Polymer film itself is another source of water vapor (PET film can contain absorbed water up to 0.8 %, and polyimide – more than 1 %.)

Experiments

- Substrate – PET Melinex 453
- Drying speed – 0.5 m/min
- Two stages – heater and drum
- Sheet resistance – 350 Ohm/sq.
- Thermal stability test – 150 ºC, 1 hour

Table 1. Thermal stability of ITO layer at alternative drying conditions.

No.	T_h, °C	T_d, °C	R_T/R_O, Thermal stability	R_D/R_T, Durability
1.	150	85	0.91 – 0.95	1.03 – 1.14
2.	155	85	0.87 – 0.88	1.0 – 1.1
3.	160	85	0.60 – 0.69	1.0 – 1.03
4.	170(*)	85	0.60 – 0.62	1.0 – 1.03

Influence of water vapor admission in vacuum chamber

- Performed exploration of the influence of drying on coating properties brought us to an assumption, that with dosed water vapor admission in vacuum chamber during deposition, coating properties can be controllable.

Experiments

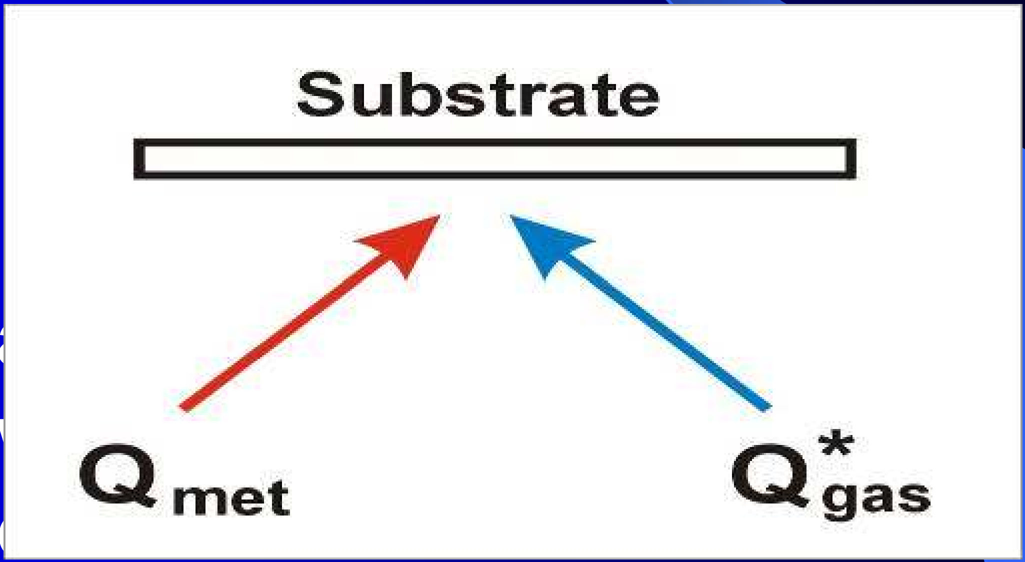
- Substrate – PET Melinex453
- Drying – 160/85 °C, 0.5 m/min
- Deposition – Q(Ar)=375 sccm, P=3.9 kW, $Q_{O2}+Q_{H2O} \approx 20$ sccm → Rs=350-390 Ohm/sq.
- Thermal test – 150 °C, 1 hour

Table 2. Thermal stability of ITO layer at alternative water vapor input flows to vacuum chamber

No.	Q_{O2}, sccm.	Q_{H2O}, sccm.	R_T/R_O Thermal stability	R_D/R_T Durability
1.	19.2	0	0.61 – 0.65	1.0
2.	18.5	1.0	0.73 – 0.80	1.0 – 1.05
3.	17.5	2.0	0.97 – 1.08	1.0 – 1.08
4.	16.5	4.0	1.31 – 1.53	1.01 – 1.08
5.	14	6.0	2.25 – 2.4	1.03 – 1.15

Extended OES method for reactive processes control

- During coating deposition by reactive magnetron sputtering on substrate go t flows: metal fl Q_{met} and reactive gas flow Q^*_{gas}.



Dynamic control of coating deposition process is reduced to two processes:

- Setting of balance between a reactive gas flow Q^*_{gas} and sputtered metal flow on substrate Q_{met}. Criterion for balance setting is coating physical properties.
- Keeping of balance between reactive gas flow Q^*_{gas} and sputtered metal flow Q_{met} on a substrate in time.

Conditions for deposition:

$$\frac{Q^{*}_{gas}}{Q_{met}} = const \qquad or \qquad \frac{p_{gas}}{I_0} = const$$

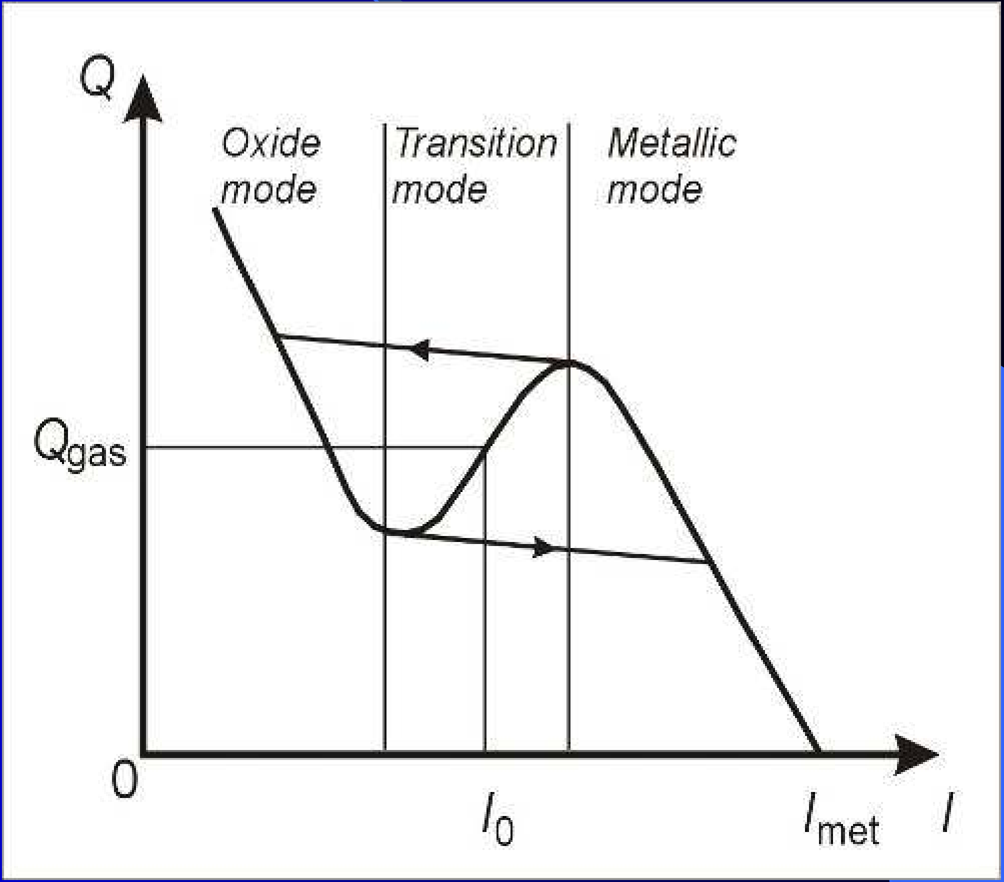
where p_{gas} – partial pressure of reactive gas

I_0 – intensity of metal emission line

- The analysis of above relationship shows that change of metal emission line intensity (that is equivalent to change of metal flow) will result in change of partial pressure of reactive gas.
- It is necessary not only to keep balance between flow of reactive gas Q^*_{gas} and flow of sputtered metal Q_{met} on a substrate, but also one of values Q^*_{gas} or Q_{met}.

Fig.1. Dependence of reactive gas flow on intensity in controlled reactive process.

- In controlled process at the given sputtering power two parameters (***I_0***, ***Q_{gas}***) determine properties of coating



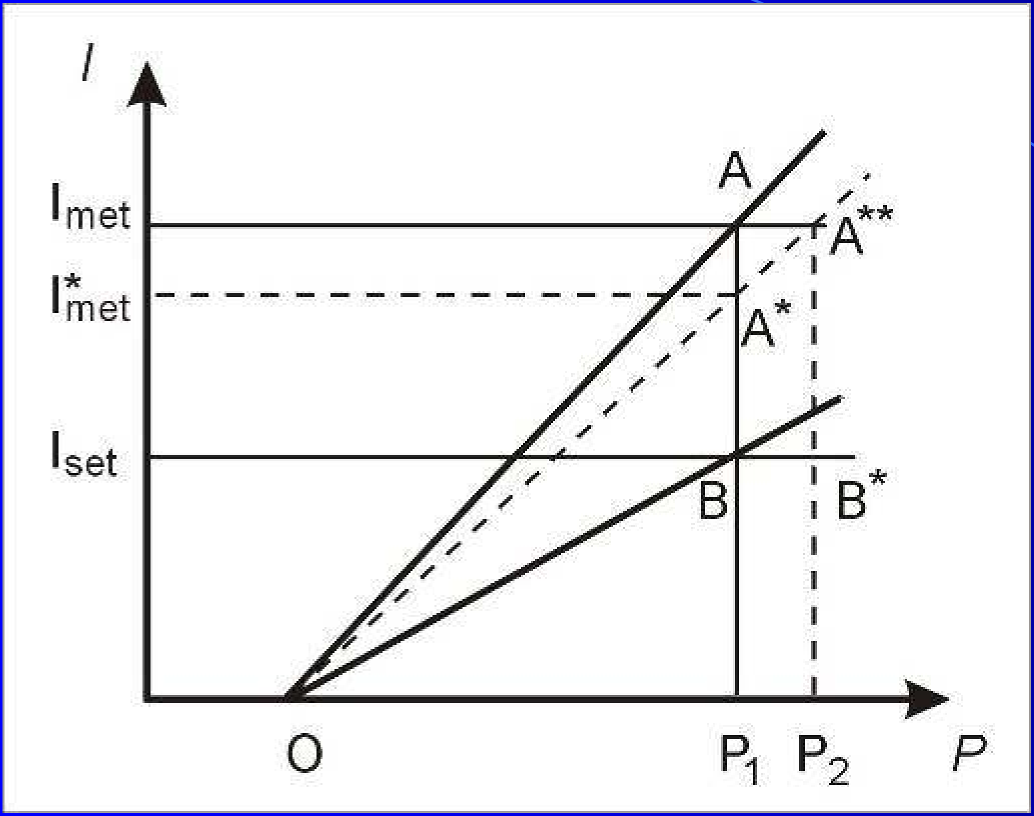


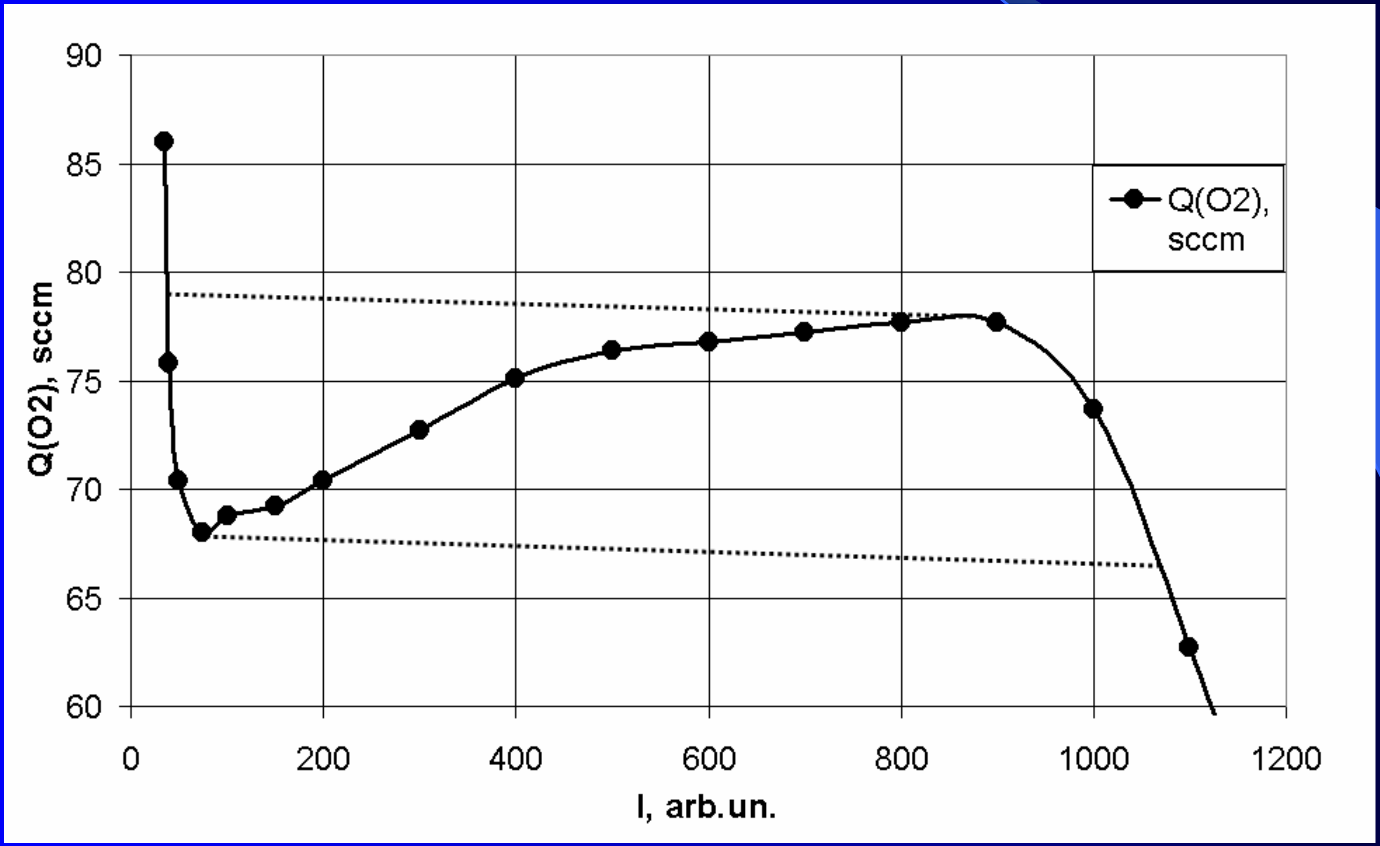
Fig.2. The scheme of extended OES method for control of reactive process.

- In a metal sputtering mode intensity of radiation is proportional to sputtering power (line ***OA***).
- In reactive process at the given sputtering power P_1 process control system with the help of OES uniquely determines necessary flow of gas Q_{gas} for maintenance of the set intensity of radiation of metal I_{set} (point ***B)*** **(first fast loop of control)**

- At keeping of all parameters of process by constants sputtering rate of a target in time is monotonously reduced.
- If in process sputtering rate of metal is reduced (line OA** on fig. 2), point ***A*** passes in ***A****, and it means, that intensity of radiation decreases up to $\boldsymbol{I^*_{met}}$ in metal sputtering mode.
- Control system to keep set intensity will reduce gas flow and as a result we will obtain material enriched with metal.

- To prevent this situation we need to increase sputtering power to return to the previous sputtering rate (point A**)
- In this case we will obtain the same product at set intensity of metal emission line.
- Criteria: it is necessary to increase sputtering power up to P_2 so that at the given intensity of radiation I_{set} to receive former value of a reactive gas flow (to go to point B*) **(second slow loop of control)**

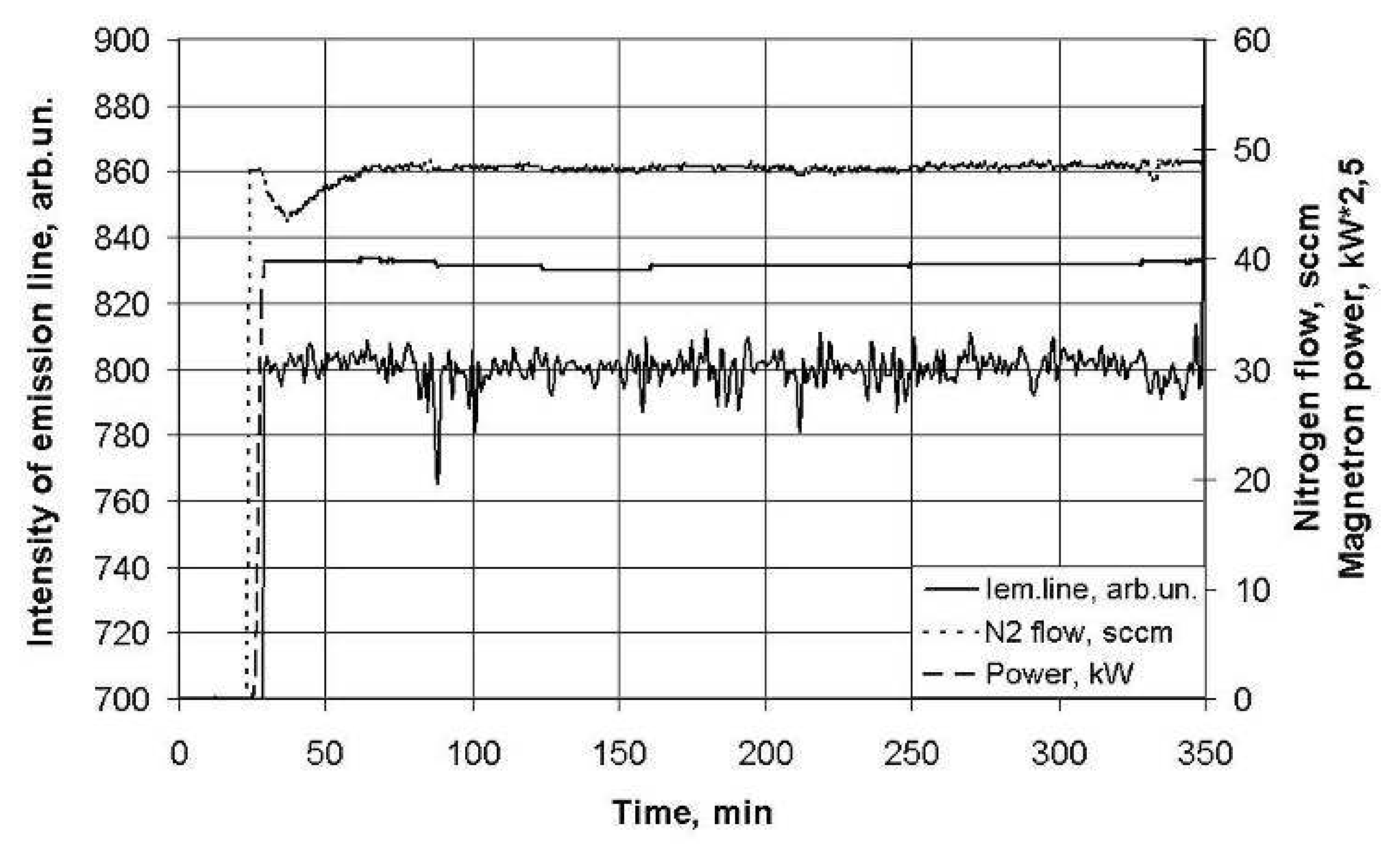
An experimental curve of interdependence of intensity of emission line and oxygen flow in controlled reactive process of titanium sputtering.



- For work of control system in transition mode the speed of measuring system is rather essential. This speed is determined substantially by speed of OES data updating. In our case good results have been received at the measuring speed of OES data more than 3 measurements per second.
- The suggested scheme of process control has allowed to work stably in a transition mode on roll to roll machine for oxide coating deposition on PET film during the long period of time.

- More difficult are processes in which it is necessary to keep such parameters as structure stoichiometry
- Such process is deposition of titanium nitride on a metal foil.

Time diagram of titanium nitride coating deposition process.



- During deposition of titanium nitride on aluminum foil for maintenance of pair parameters (intensity / flow of nitrogen in the chamber) during 7 hours power has been increased on 3.3 %, the nitrogen flow was kept with accuracy ± 2 %. It has allowed receiving during all time stoichiometric coating of titanium nitride.

Vacuum machine P600

- On the basis of above described and a number of separately performed experiments, Sidrabe Inc. developed and manufactured complex equipment for film pre-treatment and coating.

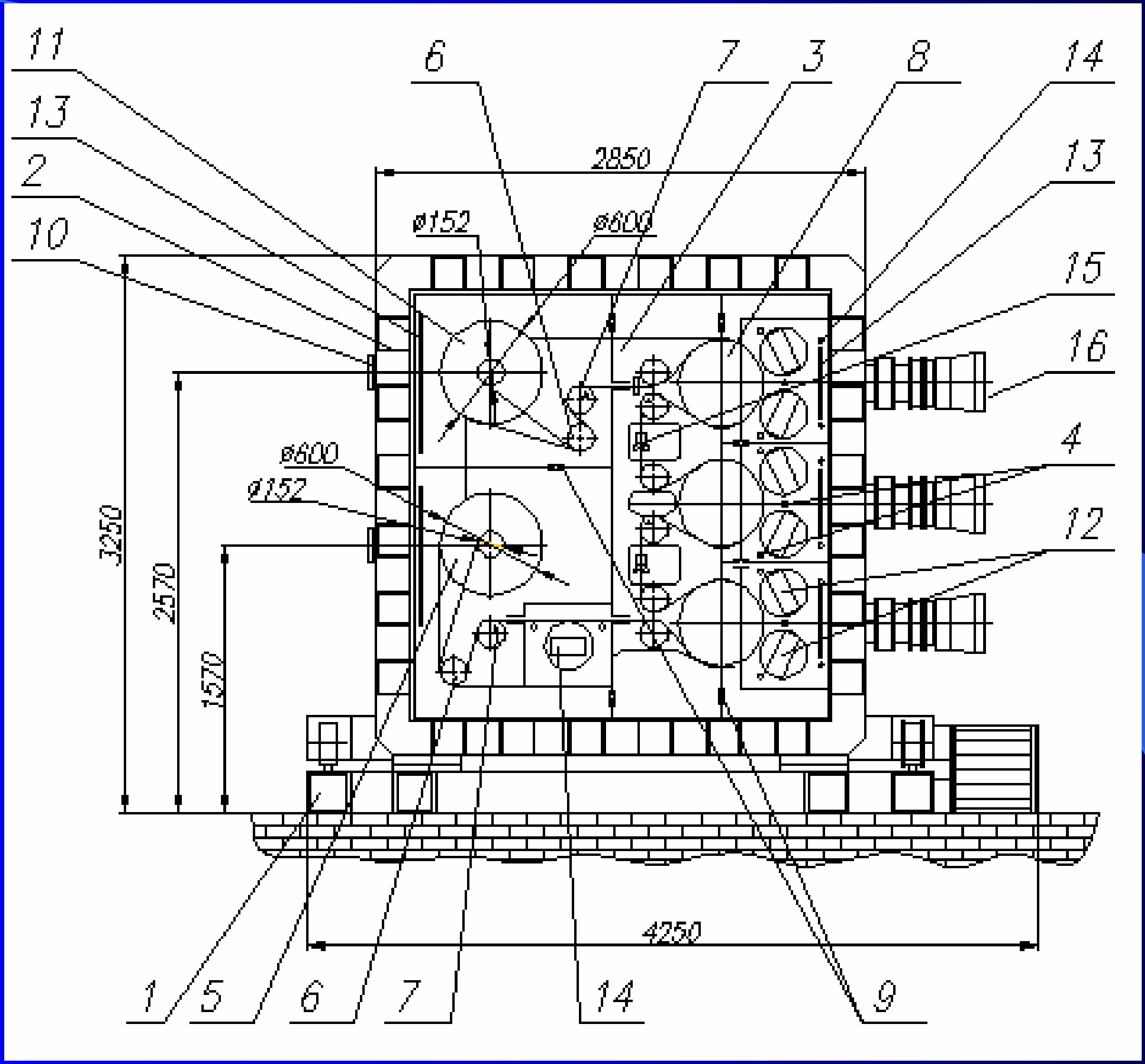


Fig.2. Layout of 3-drum's web coater:

P600MR.5

P600 advantages

- Vertical arrangement of all sputter magnetrons and deposition surfaces;
- Separated, sufficiently insulated zone with individual drum for each deposition process;
- Application of high-effective plasma treatment device for film pretreatment before the deposition;
- Symmetrical, uniform pumping is accomplished for each deposition zone.

P600 advantages

- This design allows control of sheet resistance and optical characteristics (transmission, reflection) after each deposition compartment;
- There are machine versions with turbomolecular and with cryogenic pumping of the magnetron compartments;
- There is a version with 6 drums with 8 insulated compartments, developed especially for films with AR layers.

C2P600.6

C2P600.6

Main Technical Features:

Batch type operation
Material width up to 670 mm
Film thickness – 50…200 microns
Maximum roll diameter – 500 mm
Heating temperature – 80…200 °C
Adjustable winding speed – 1…20 min
Pressure in the heating zone – $5x10^{-2}$…$1x10^{-1}$ Torr
IR heaters power – 30 kW
Control system
Footprint of the machine – 8.1x2.8x2.7 m

CONCLUSION

- 1. The performed researches proved the important influence of preliminary film drying and outgasing on such basic characteristics of ITO thin films, as thermal stability and wear resistance. Preliminary drying brings film to uniform initial state, ensuring high outcome reproducibility

CONCLUSION

- Application of dose admission of water vapor during ITO deposition cycle enables control of thermal stability of product, with a capability to increase and decrease it. In-situ water vapor supply stabilizes the process and allows to decrease specific resistance

CONCLUSION

- 2. Application of carbon dioxide as reactive gas for ITO with ceramic target In_2O_3/SnO_2 in reactive magnetron proved that, compared with oxygen, carbon dioxide ensures smooth alteration of sheet resistance and specific resistance at altered gas flows at constant emission intensity, and also, considerable gas flow interval, at which minimum sheet resistance and minimum specific resistance can be attained.

CONCLUSIONS

- 3. Addition of OES method by a feedback power – flow of reactive gas allows to remove influence of sputtering rate change at change of target sputtering conditions.
- 4. In suggested method control is made on two parameters of process: to a reactive gas flow and sputtering power at preset emission line intensity.

CONCLUSIONS

5. Two control loops in extended OES method provide with:

- Stable course of process of reactive deposition of a coating in a transition mode by keeping of intensity of radiation of a spectral line of metal by control of reactive gas flow in the chamber.
- Stability of properties of a coating in time by control of sputtering power for maintenance of reactive gas flow at the set intensity of radiation of a spectral line of metal.

CONCLUSION

- 6. On the basis of carried out researches Sidrabe Inc. developed high-productive vacuum equipment for ITO thin films, and for multilayer oxide and nitride coating's deposition.

Thanks for your attention

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