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Increasing the Observability of the Thin Films Manufacturing Process

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Introduction

The rate of deposition of the vapor have the substantial influence to the properties of the condensed thin film during the thin films manufacturing [1]-[5]. The observability and controllability of the deposition rate depends on the actual manufacturing technology used. The ”precise” or ”advanced” technologies, such as laser deposition, get more potential for the deposition rate to be determined and controlled, and the ”rough” technologies, such as thermal evaporation, are less observable and controllable in terms of the deposition rate [5]. However, thermal evaporation, being less precise, has the widest use in the mass-production, thanks to the possibility of unsophisticated industrial application. As the technological requirements are constantly extending towards the specific or more precise properties of films, the challenge to develop a new ways to meet these requirements arises.

The main factor determining the vapor deposition process is the evaporation rate, which is obviously dependant on the evaporation temperature.

The interaction of the evaporator with the sample of material during the thermal vacuum evaporation process is complex and sometimes hardly predictable. In addition to the cooling of the evaporator, caused by the parting of the vapor energy, the electrical properties of the evaporator may also become the functions of the aggregate state of the sample, when it is being heated, melting, irrigating the evaporator and evaporating (Fig.1). Therefore, the actual evaporation rate and the deposition rate, as a consequence, at certain moment remains unknown, unless it is measured.

Several conventional film deposition rate measurement methods, used in thermal evaporation technologies [3],[4], provide for the information of the quasi-static nature, i.e. the measurement channel does not provide for the relevant dynamics or is hardly applicable at the mass production conditions.

Moreover, a thin layers production, using thermal evaporation, canonically consists of several phases:

- initial heating, followed by the effervescence of gases from the heated body;
- evaporation, which is related with the multiple thermal and electrical effects again;
- melting, usually followed by the drop of the temperature of the evaporator and the change of the electric properties of it;

In order to control the vapor deposition of desirable rate and quality, it is necessary to control the temperature of the evaporator during all these phases. For example, when in gas effervescence phase, the rise of the temperature needs to be hold for several seconds to prevent the evaporation from starting before the evacuation of the gases from the vacuum chamber finishes. The temperature has not to exceed a boiling point during the melting phase to prevent undesirable sputtering; it also has to be kept constant during the evaporation phase to ensure the steady evaporation rate.

Usually the temperature of the evaporator is controlled under the open loop conditions, i.e. by simply setting appropriate working point or working program for desirable operation. Such an approach does not ensure the repeatability of results, when requirements to the sophisticated properties of the films rise.

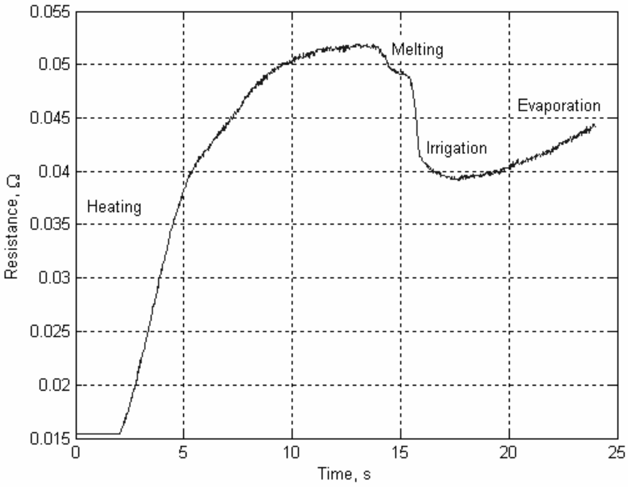

Fig. 1. Evaporator resistance is influenced by the informative (temperature) and non-informative (conductance of the evaporating material) factors

The controllability via observability

The essential issue to make the process controllable with the closed loop scheme is to make the output of the process observable in situ, meeting the real time constraints.

Consequently to the need of satisfactory measurement dynamics, a selection of several physical principles was made:

- direct thermocouple pyrometry;
- evaporator power circuit properties measurement;
- measurement of a thermo electron emission current.

We rejected the pyrometry, because even the platinum thermocouple is unstable at the temperatures over 1000 ^{o}C. A thermo electron emission current is the most promising carrier of the temperature information, but needs the sophisticated methods of elimination of the influence of non-informative electric fields. The second option regards the evaporator itself as a thermistor, and the temperature can be measured as a function of the evaporator resistance. Similar approaches were used during the vacuum tubes cathodes temperature measurements [6],[7]. No add-on instrumentation in the vacuum part of the system is needed, so, it seems to be the most attractive option. In order to be able to adequately measure the temperature in such a way, the mathematical description of the evaporator power circuit has to be developed.

The model of the evaporator power cirquit

Conventionally we use the resistive evaporator, directly heated by the low voltage power. Together with the temperature effect, a resistance of the evaporator is influenced by several constructive and electric properties of the system:

- the aggregate state, electric contact, conductivity and the residual mass of the material sample (Fig.1);
- the temperature of surrounding constructions, which increase with time, even if the heating power is stabilized;
- temperature drift of the elements of power circuit.

The current in the circuit on the Fig.2 can be described as follows:

$$\frac{di}{dt} = \frac{u_m - i(r_0 + r_t + r_a + r_g)}{L}. \qquad (1)$$

In the general case the resistance of the evaporator r_g is a composition of two resistances: resistance of evaporator itself and a resistance of the sample of material. For the first approach we model an empty evaporator (this is evenly the case, when the resistance of the sample is much bigger than the resistance of an evaporator, eg. when evaporating the dielectric material). Therefore, the temperature dependency of r_g :

$$r_g(T_g) = r_{g0}(1 + \alpha_g(T_g - T_0)); \qquad (2)$$

where r_{g0} stands for initial evaporator resistance (at room temperature); Tg - for evaporator temperature; α_g – for the temperature factor of the evaporator resistance.

Substituting (2) to (1) we get:

$$\frac{di}{dt} = \frac{u_m - i(r_0 + r_t + r_a + r_{g0}(T_g))}{L}. \qquad (3)$$

The temperature of the evaporator T_g is dependant on the temperature effect of the current, passing thru it and the heat, transferred to the surroundings. Without detailing the ways of the heat transmission, we get:

$$\frac{dT_g}{dt} = \frac{i^2 r_g(T_g) - W_g}{m_g c_g}; \qquad (4)$$

where m_g is the mass of the evaporator, c_g – the evaporator specific heat constant, W_g – the heat transmitted to the environment.

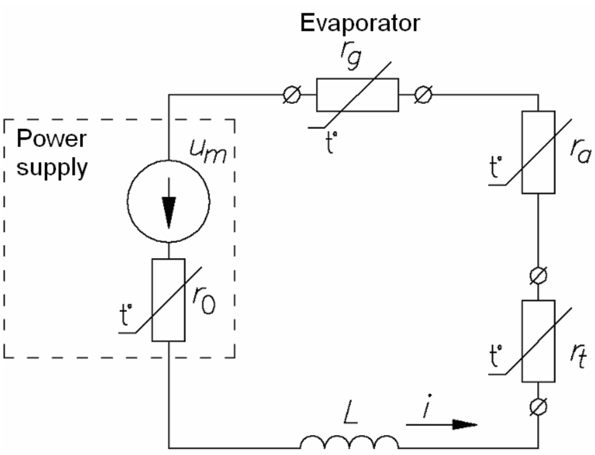

Fig. 2. The equivalent power cirquit of the evaporator: u_m – voltage of the power supply; r_0 – internal resistance of the power supply; L – equivalent inductance of the whole circuit; r_t – resistance of the power regulator; r_g – resistance of the evaporator

The temperature transmitted to the environmental objects:

$$\frac{dT_k}{dt} = \frac{k_k(T_g^4 - T_k^4) - k_0(T_k^4 - T_0^4)}{m_k c_k}, \qquad (5)$$

where m_k and c_k stand for the mass and the specific heat constant of the surrounding objects, correspondingly; T_0 stands for the temperature of the vacuum jar walls, which are water-cooled, so it can be regarded as a constant.

Model of the thermal interaction with the evaporating material

The relationship between the visible thermal phenomena and the evaporator temperature dynamics during the part of evaporation process is shown on the Fig. 3. Initially the peace of solid material barely contacts with the evaporator (only some points), therefore the heat transfer by the radiation is dominant. When the body of the material is starting to melt, thermal contact improves, and the evaporator is getting to be locally cooled by the melting body, which accumulates the energy. After the material comes to the liquid state, it suddenly pours out over the evaporator and cools it out entirely. Then the temperature balance tend to be established by transmitting the heat from the evaporator to the liquid material via the direct contact. The material starts to evaporate and therefore consumes a part of the supplied energy. Another part of the energy is transmitted to the environment. During the evaporation of the material its mass and the occupied place are decreasing until it fully goes out from the evaporator.

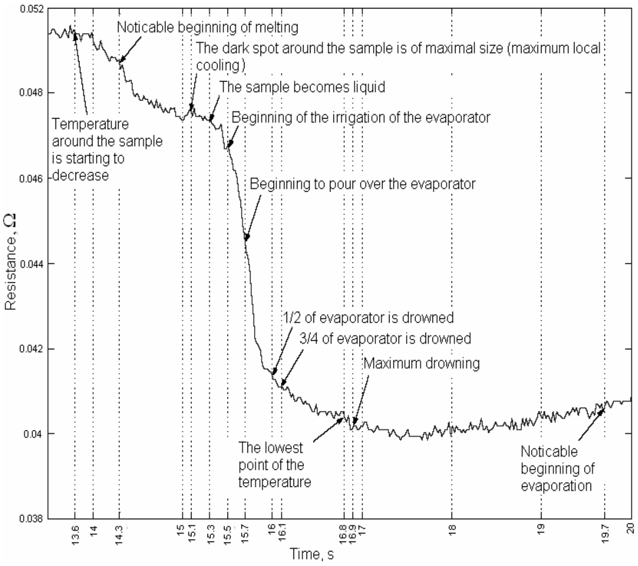

Fig. 3. The relationship between the visible thermal phenomena and the evaporator temperature dynamics during the part of evaporation process

The equation below represents the process described, when the temperature of the material does not equal to the temperature of the evaporator neither to the temperature of the environment:

$$\frac{dT_m}{dt} = \frac{k_{m,i}\left(T_g^4 - T_m^4\right) - k_{m0,i}\left(T_m^4 - T_k^4\right)}{m_m c_{m,i}}. \qquad (6)$$

An index i represents the aggregate state of the material: i=1 corresponds to the solid state; i=2 – melting state and i=3 corresponds liquid state and the vapor state; $k_{m,I}$ stands for the heat transfer factor from the evaporator to the material; $k_{m0,i}$ stands for the heat transfer factor from the material to the environment; $c_{m,I}$ – stands for the specific heat constant of the evaporating material; m_m stands for the mass of the material. An index i can be calculated as:

$$\begin{cases} i = 1 & \text{if} \quad T_m < T_L \quad \text{and} \quad Q_m \le 0, \\ i = 2 & \text{if} \quad T_m \ge T_L \quad \text{and} \quad Q_m \le Q_L, \\ i = 3 & if \quad T_m \ge T_L \quad \text{and} \quad Q_m > Q_L, \end{cases} \qquad (7)$$

where T_L – melting temperature of the material, Q_L –the amount of the heat needed to bring the material to the melting state, Q_m – accumulated amount of heat in the melting state.

Overall mathematical description and simulation

The system of equations below represents the overall mathematical model of an entire system; it is based on the equations 1 to 6.

$$\begin{cases} \dfrac{di}{dt} = \dfrac{u_m - i\left(r_0 + r_t + r_a + r_{g0}\left(T_g\right)\right)}{L}, \\ \dfrac{dT_g}{dt} = \dfrac{i^2 r_{g0}\left(T_g\right) - k_k\left(T_g^4 - T_k^4\right) - k_{m,i}\left(T_g^4 - T_m^4\right)}{m_g c_g}, \\ \dfrac{dT_k}{dt} = \dfrac{k_k\left(T_g^4 - T_k^4\right) - k_0\left(T_k^4 - T_0^4\right)}{m_k c_k}, \\ \dfrac{dT_m}{dt} = \dfrac{k_{m,i}\left(T_g^4 - T_m^4\right) - k_{m0,i}\left(T_m^4 - T_k^4\right)}{m_m c_{m,i}}, \ when\ i = 1, 3, \\ \dfrac{dQ_m}{dt} = \dfrac{k_{m,i}\left(T_g^4 - T_m^4\right) - k_{m0,i}\left(T_m^4 - T_k^4\right)}{m_m c_{m,i}}, \ when\ i = 2. \end{cases} \qquad (8)$$

It does not take into account the conductivity of the evaporating material, therefore could be regarded as a model for the dielectric evaporating material. An output of the model is shown on the Fig. 4.

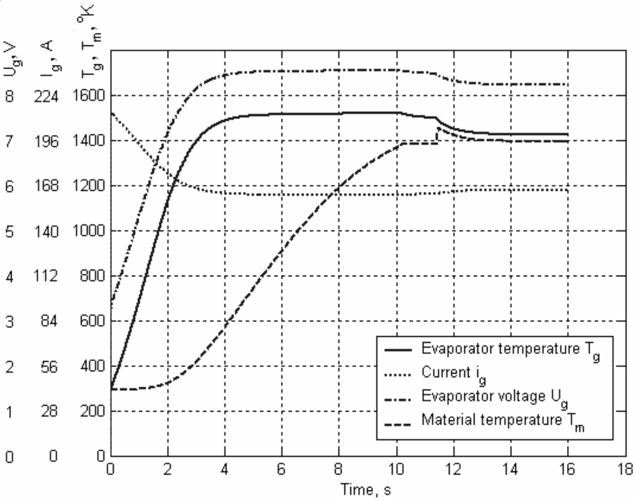

Fig. 4. Simulated dynamics of an electric and temperature parameters. The melting point is reached after the 10-th second

Conclusion

The experimental and theoretical research, being carried in this work, allows distinguishing between the non-informative influence to the electrical properties of the evaporator power cirquit and the evaporator resistance temperature dependance.

It provides for the possibility of in-situ temperature measurement by simple measurement of the evaporator resistivity. The measured data are of sufficient dynamics and could be used for closed loop control of the thin films manufacturing process.

The application of this temperature measurement method does not involve the need for add-on instrumentation in the vacuum part of the system, therefore this method can be applied at the mass-production conditions by adding the adequate processing of the power cirquit voltage and current measurement data.

This method for improving the observability of the evaporation process is not applicable, when the power of the evaporator is compleatly switched off. Though this could be noted as a disadwantage, in practictice such a situation is rare.

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D. Viržonis, V.Sinkevičius. Plonų sluoksnių gamybos proceso stebimumo didinimas // Elektronika ir elektrotechnika. – Kaunas: Technologija, 2004. – Nr. 4(53). – P. 42-45.

Kietų medžiagų vakuuminis išgarinimas yra labiausiai paplitusi pramoniniu būdu gaminamų plonų kondensuotų plėvelių gamybos technologija. Kylant kokybiškai naujiems reikalavimams šių plėvelių kokybei, kyla poreikis surasti naujus priimtinus šio gamybos proceso valdomumo ir stebimumo gerinimo būdus. Manoma, kad dinaminis rezistyvinio garintuvo maitinimo grandinės elektrinių parametrų matavimas yra priimtinas sprendimas, siekiant didinti proceso stebimumą. Darbe pateikti eksperimentų rezultatai ir garintuvo maitinimo grandinės bei šiluminės sąveikos tarp garintuvo bei garinamosios medžiagos matematinis modelis. Matematinis elektrinių ir šiluminių procesų modelis leidžia atskirti informatyvius ir neinformatyvius išmatuotuosius dydžius. Daroma išvada, kad aprašytas metodas yra pakankamas garintuvo temperatūros matavimui in situ, gamybos metu. Tam nereikalinga jokia papildoma įranga vakuuminėje garinimo sistemos dalyje, todėl šis metodas yra potencialiai tinkamas naudoti pramoninėmis sąlygomis. Il. 4, bibl. 7 (anglų kalba; santraukos lietuviškai, angliškai ir rusų k.).

D. Viržonis, V.Sinkevičius. Increasing the Observability of the Thin Films Manufacturing Process // Elektronika ir elektrotechnika. – Kaunas: Technologija, 2004. – No. 4(53). – P. 42-45.

Thermal evaporation of solids is a technology of most common use during the industrial manufacturing of thin condensed films. The qualitatively new requirements for these films call to investigate a new common ways to improve the controllability and observability of the manufacturing process. The dynamic measurement of the electrical properties of the resistive evaporator power circuit is thought to be a reasonable solution for the observability improvement. An experimental data and the mathematical modeling of the evaporator power circuit and the thermal interaction between the evaporator and the evaporating material are described. The mathematical description of an entire process allows distinguishing between the informative and non-informative quantities measured. It is concluded that presented method is sufficient for in-situ evaporator temperature measurement during the manufacturing process. It does not require for the add-on instrumentation in the vacuum chamber of the evaporation system, therefore is acceptable for application in industrial conditions. Ill. 4, bibl. 7 (in English; summaries in Lithuanian, English, Russian).

Д. Виржонис, В. Синкявичюс. Увеличение наблюдаемости процесса производства тонких пленок // Электроника и электротехника. – Каунас: Технология, 2004 – № 4(53). – P. 42-45.

Термическое выпаривание твердых тел является одной из самых распространенных технологий производства тонких конденсированных пленок. Качественно новые требования к качеству этих слоев вызывает потребность исследовать новые способы повышения контроллируемости и наблюдаемости за процэссом. Динамическое измерение электрических параметров цэпи питания резистивного испарителя считается подходящим решением, повышая наблюдаемость процэсса. В статье представлены экспериментальные данные и математическая модель силовой цэпи испарителя и теплового взаимодействия между испарителем и испаряемым материалом, которая позволяет отличить информативные измеряемые величины от неинформативных. Делается вывод, что данный метод подходит для промышленного измерения температуры испарителя, не вызывая нужды в доработке вакуумной части системы испарения. Илл. 4, библ. 7 (на английском языке; рефераты на литовском, английском и русском яз.).

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