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INFRARED HEATERS WITH THIN-FILM CONDUCTIVE LAYERS WERE SYNTHESIZED ON THE GLASS BY THE MAGNETRON SPUTTERING

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This paper examines the infrared heating panels with thin conductivite layer. An oxide semiconductor thin film layer is proposed for improvements of the heating layer's reliability and stability. Characteristics of the oxide semiconductor thin film's materials are analyzed and tin oxide film are selected for further research. The results of the experiments and the characteristics of the films depending on technological factors had been given. Considered parameters of infrared radiant heating glass panels with a thin film heating layer. Established maximum allowable electrical parameters of the IR radiating panels with a thin film heating layer, as well as the possibility of increasing the efficiency of IR panels and power savings.

INTRODUCTION

Infrared heaters are widely used in various fields of human activity. They are conventionally divided into "light" and "dark", depending on the operating temperature and, hence, the emission wavelength. "Light" are short wavelength and "dark" are long wavelength. There are many designs of both types' infrared heaters.

Our research in this paper were limited to one of the "dark" of heating devices, namely, heating panels on the glass. These infrared panels are very widely used for heating various buildings such as industrial, office, warehouse, residential. The same panels are produced in several countries: Germany, Belgium, Holland, England, Sweden, China, and have insignificant constructive and technical differences. They all have a heat capacity 0.5 to 2.0 kW/m^2 and the temperature of the glass radiating surface 60…135 ^{0}C. These limits are defined by a thick film's quality and capabilities of the heating layer based on graphite paste used in these panels.

The heterogeneity of the resistance in the thick layer leads to a limited lifetime in films obtained by sintering the paste (studied IR heater samples were represented in EU market).

This paper presents the results of the research IR heaters on the glass with a thin-film heating elements. The main types of radiant heaters and their parameters, conditions of using are investigated.

The variants increasing heater emissivity are suggested for the possible using of these heaters in process equipment that requires significantly higher temperatures,

1. MATERIALS AND EXPERIMENTAL EQUIPMENT

The choice of material for the thin film heating element. The main requirements for the thin film heater layer are the ability to operate at standard 220 V power supply and at the same time, electric power consumption must be 0.5…2.0 kW/m^2. This requirement necessitates the current load of 2 to 12 A on the device, therefore, the resistance of the heating thin film of the Infrared heater should be in the range of 20…110 Ω.

If we take the size of their foreign counterparts IR panels (Belgium, Netherlands, Germany) 60x120 cm, the resistance of the thin film heater is 20…40 Ω/□. Consequently, as the thin film heater it is impossible to use metals (low resistance), cermet (high resistance) and semiconductors (strong temperature dependence of conductivity). Thus option is the use thin films of degenerate wide bandgap semiconductor with a dopant amount of the base material (5…15)% [1]. In case of a certain amount of impurity centers conductivity material does not depend on the temperature, and, hence, resistance of the film remaine stable. Such materials include degenerate semiconductor oxide In_2O_3, SnO_2, ZnO, CdO and others. Thin films of such materials are widely used as the transparent electrodes in the display devices (oxides of indium and tin) and photoelectric converters (zinc oxide).

To select an oxide conductive material we considered the most famous of them, namely, tin oxide, indium oxide and zinc oxide. We are interested in the basic properties of these materials which are given below.

Tin oxide (SnO_2). Dopants providing the conductivity of tin oxide films are Fluorine (F) and Antimony (Sb). The carrier concentration is $10^{18}…10^{21}$ cm^3, resistivity is $0.1…4{\cdot}10^{-4}$ Ω·cm, the mobility is 10…50 cm^2/(V·s), band gap 3.6 eV. The average transparency films in the visible range is 86…87%. The conductivity of tin oxide films doped with Fluorine is higher than the conductivity of films doped with Antimony, however, their stability is much higher. Tin oxide films have a high stability, strong adhesion (when deposited onto glass, quartz, glass ceramics and other substrate materials), resistant to moisture and acids [2–4].

Indium oxide (In_2O_3). The main dopant providing conductivity film is tin (Sn). Other impurities, providing a highly conductivity of the indium oxide films are titanium (carrier mobility is 120 cm^2/(V·s), band gap

3.75 eV, the carrier concentration is 10^{20} cm^3) and zirconium (carrier mobility is 170 $cm^2/(V \cdot s)$), the carrier concentration is $8 \cdot 10^{19}$ cm^3) [4]. The resistivity of indium oxide films is $2 \cdot 10^{-4} \ldots 10^{-2}$ Ω·cm, carrier concentration is $10^{19} \ldots 2 \cdot 10^{21}$ cm^3, the carrier mobility is 15…70 $cm^2/(V \cdot s)$. The transparency of the films in the visible range is 92…94%.

The conductivity and transparency of tin oxide films doped with tin is higher than the tin oxide films. Indium oxide films differ strong adhesion to various substrates, including a glass, quartz and glass ceramics, resistant to moisture, however, have poor stability at elevated temperatures and easily etched by acids.

Zinc oxide (ZnO). The main doping material is Aluminum (Al). The resistivity is about $8 \cdot 10^{-4}$ Ω·cm high carrier concentration is $5 \cdot 10^{-20}$ cm^3 and mobility is 15 Ω·cm, bad gap 3.35 eV. The average transparency of zinc oxide films is 90…92% with a slightly higher temperature stability than the indium oxide film. The adhesion of the zinc oxide films to glass is high.

Cadmium oxide (CdO). Doped with Sn, i.e. cadmium stannate (Cd_2SnO_4) is the least common conductive film's material with a carrier concentration $10^{17} \ldots 10^{21}$ cm^3, mobility 8…70 $cm^2/(V \cdot s)$, low enough resistivity ~ $5 \cdot 10^{-4}$ Ω·cm. Transparent of cadmium stannate films is relatively low ~ 76…82%. It is associated with a multiphase system.

The data obtained from the literature [5–10] with their own impurities in the films of In_2O_3, SnO_2, ZnO. Measurements of the electrical characteristics of the oxide films we carried out according to standard procedures [11, 12] on the ECMC-4 device for measuring the surface resistance.

2. RESULTS AND DISCUSSION

The main characteristics of the SnO_2 films are shown on Figs. 1 and 2, namely the dependence of Antimony doping level at the same magnetron sputtering regime: power 3 W/cm^2, the amount of oxygen in the plasma is 4%, the vapor pressure of the air Oxygen mixture Argon + Oxygen equals 1 Pa.

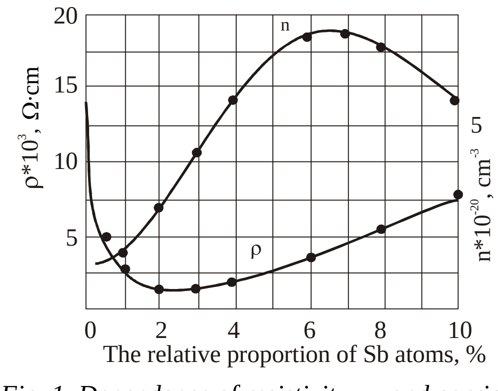


Fig. 1. Dependence of resistivity ρ and carrier concentration n from the impurities in the SnO_2 doped with Sb films

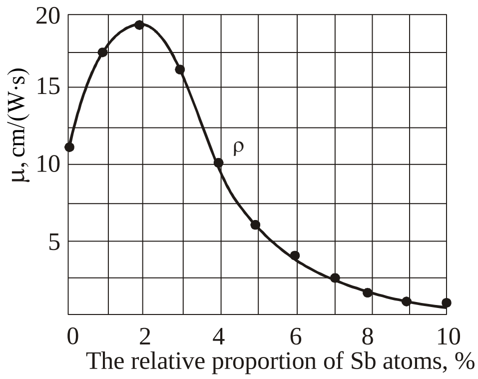


Fig. 2. The mobility μ dependence on the impurities in the films of SnO_2 doped with Sb

As can be seen from Figs. 1 and 2 the characteristics of the films greatly vary quite up to 1…2 orders of magnitude with the changing of the amount of impurities. However, you can get the necessary change of parameters at a fixed value of impurities even. As an example, Figs. 3–5 show resistivity of obtained SnO_2 doped by Sb films at a fixed impurity concentration about 3%.

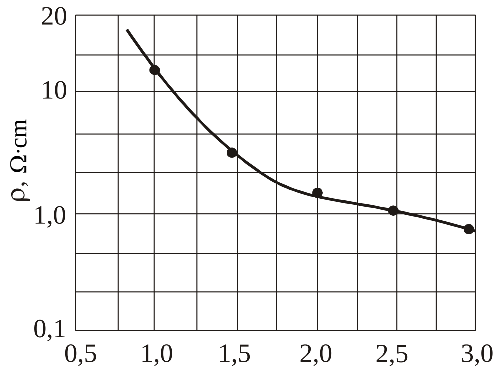


Fig. 3. The dependence of the resistivity in the SnO_2 doped with Sb (3%) films from the magnetron power density at (P_{O_2} = 4%, $P_{Ar + O_2}$ = 1 Pa)

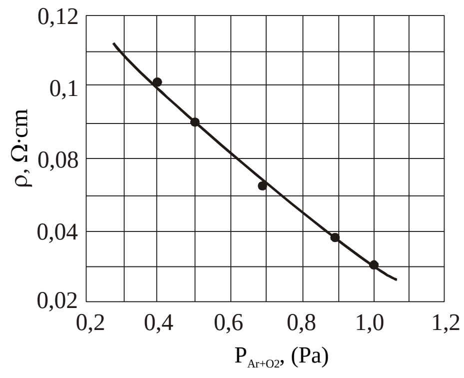


Fig. 4. The dependence of the resistivity SnO_2 doped with Sb (3%) films from the of the (Ar + O_2) gas pressure at (P_{O_2} = 4%, P = 3 W/cm^2)

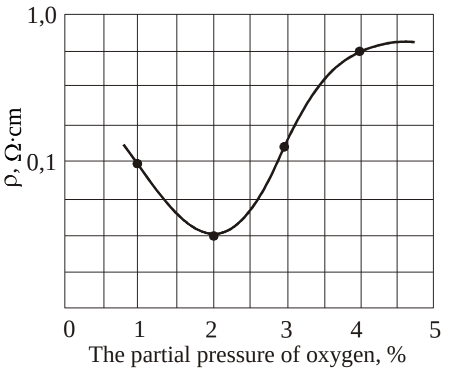


Fig. 5. The dependence of the resistivity of the SnO_2 doped with Sb (3%) films from the oxygen partial pressure at ($P_{Ar + O_2}$ = 1 Pa, P = 3 W/cm^2)

Similar results for In_2O_3:Sn films was obtained in the case of RF magnetron sputtering. Some of these data are shown in Table.

The oxygen partial pressure, %	Carrier concentration, cm^3	Mobility, cm^2/(V·s)	Resistivity, Ω·cm
0.1	$5.3 \cdot 10^{20}$	17.8	$6.5 \cdot 10^{-4}$
0.2	$4.9 \cdot 10^{20}$	28.8	$4.4 \cdot 10^{-4}$
0.5	$1.8 \cdot 10^{20}$	20.4	$9.0 \cdot 10^{-3}$
1	$9.2 \cdot 10^{19}$	0.40	$1.7 \cdot 10^{-2}$

As can be seen from this table, the changing of the oxygen partial pressure in the vacuum chamber considerably changes the parameters of the obtained indium oxide films. Detailed data and analysis of the technological research results will be published in a separate paper. The stability of the parameters and, above all, the resistance stability at elevated temperatures is one of the main characteristics of thin conductive oxides films which can be used as resistive heating elements in the IR radiator. Comparing the temperature stability of the indium and the tin oxides films, it was installed significantly better stability of the tin oxide films over a wider temperature range. Fig. 6 shows the results of measurements of the indium and tin oxides films. Measuring time the thermal stability was not more than 10 minutes. Films were deposited on the tested quartz substrates.

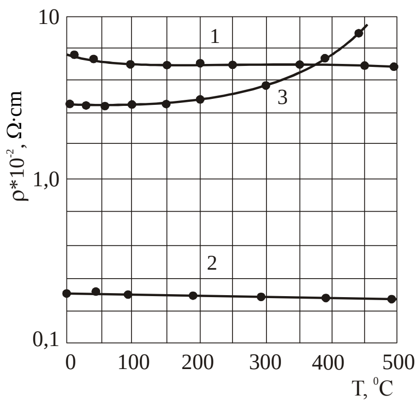
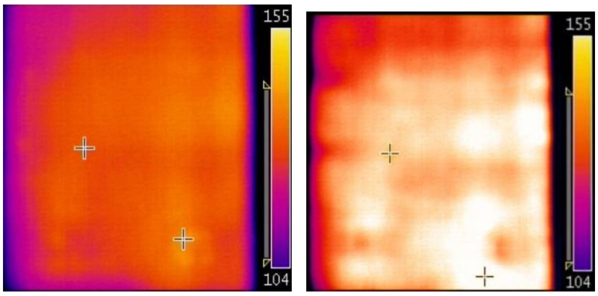


Fig. 6. The dependence the indium tin oxide films (thickness is about 0.3 μm) resistivity on an elevated temperature: 1 – basic film SnO_2:Sb; 2 film – SnO_2:Sb after heat treatment at 500 ^{0}C for 2 hours; 3 – In_2O_3:Sn initial film

As seen in Fig. 6, the tin oxide film is stable over time at temperatures up to 500 ^{0}C. At the same time, the indium oxide films change their resistance upward essentially.

Therefore, the tin oxide films were chosen for deposition in the line of the vacuum deposition “TechnoRay”. Their resistance is chosen so that the IR 600x1200 mm panel (similar manufactured in EU market) could have a resistance of 50 to 25 Ω at 220 V by changing the process conditions what corresponds to the power in the infrared heating panels from 1000 to 2000 W, and the radiating surface temperature from 100 to 160 ^{0}C.

Our studies have shown a high uniformity of the radiator in operations. The temperature increasing on the surface of the radiator at design regime was (130±5) ^{0}C. Fig. 7 is a picture obtained using thermal camera when the temperature of the rear surface of the radiator did not exceed the room temperature more than 5 ^{0}C.



a *b*

Fig. 7. The temperature distribution on the surface area which were determining of the most “cold” and “hot” zones: a – experimental infrared irradiator, b – infrared radiator in case of a typical market sample

For comparison, Fig. 7 shows the data on the measurement of the temperature uniformity on the surface of the infrared radiator in case of a typical market sample. As seen from Fig. 7 the homogeneity of the glass-infrared radiating panel with a thin film electrode is significantly higher. Such IR panels on the glass can be widely used for heating of various buildings. However, for using in the process equipment, such as nondestructive active thermography control or high temperature furnaces, this temperature is insufficient. Therefore, a separate task was to determine the boundary of electrical parameters for the using of the infrared heating panels on the glass in the process equipment.

To determine the maximum allowable power of the IR panels, assume that the resistance of the heated film (in the simplest case) does not change with the temperature. Therefore, increasing the power supply, we increase the current capacity. Determination of the maximum current load will be the boundary condition of the film heating element using. Assuming that the current density is the same throughout the heating surface of the film, the per linear decimeter contact will counts for about 1.5 A, while power consumption is 1.5 kW and a surface temperature is 135 ^{0}C. Based on the measurement results, it can be concluded that the investigated radiator has the possibility of using when the current load is to 2…2.5 A on the 1 dm contact, which corresponds to the power consumption and power output up to 6 kW. At the same time, a heater's thin oxide film has shown its efficiency at a current load up to 5 A/dm.

Our studies of infrared radiant panels on glass showed that an increase in temperature of the glass significantly changed its emissivity. Thus, at 20 °C temperature, η_{CT} (emissivity) = 94…96%; at T = 100 ^{0}C, η_{CT} = 92%; at T = 250 °C, η_{CT} = 87% and at T = 500 °C, η_{CT} = 76%. In addition, as the temperature rises, internal stresses can appear [15] and also the role of regions with an island fractal-like conductivity can be increase [16]. This leads to a considerable decreasing in the efficiency of the glass panel when the panel is used in the process equipment. To increase the emissivity, we used additional coverage of the glass panel's outside part. For this purpose, the glass coating with a thin film of chromium oxycarbide (black chrome) was used [17]. Emissivity of the film changed from 96 to 94% slightly when the temperature increases from 20 to 400 °C.

In addition, we measured the emissivity of the glass covered by a black high-temperature paintings (450…600 °C). The coated glass radiant ability remained at the 98…96% level [18]. Thus, infrared radiant panels with an additional black layer on the glass may be used in process equipment.

CONCLUSION

It is shown that the conductive oxide semiconductor films SnO_2, In_2O_3, ZnO, CdO are the most promising materials of the heating elements in the panels. Analysis of the properties of these materials allowed to identify the most suitable In_2O_3 and SnO_2 films for using as the heating elements. We studied their characteristic's dependences on the technology of preparation. It was found that the line with continuous magnetron sputtering of the films was the most suitable. The dependence of the characteristics of the films, not only on the composition of the target (the amount of impurities) but also on the technological regimes of their application, had been found.

It is shown that the necessary change of the heating characteristics of the films can be obtained by changing the process conditions of their deposition.

Measurement of temperature stability has allowed to choose the most stable films namely tin oxide film. It is shown that the power of the infrared panel and the temperature of its radiation can be regulated by changing the parameters of the films. Methods of producing these films had been analyzed. We studied their characteristics depending on the technology of preparation. It was found that the line with continuous magnetron sputtering films was the most suitable for the mass production. The dependence of the characteristics of the films, not only on the composition of the target (the amount of impurities) but also on the technological regimes of their deposition had been found.

It is shown that the necessary change of the film's heating characteristics can be obtained by changing the process conditions of their deposition.

Measurement of temperature stability has allowed to choose the most stable films namely the tin oxide film. It is shown that by changing the parameters of the films, the power of the infrared panel and the temperature of its radiating surface can be changed. In order to determine the possibility of the using the infrared panels in process equipment, their maximum permissible electric characteristics are defined.

The advantages in the maximum temperature uniformity and the surface temperature of the glass panels with a thin film heater layer had been shown in comparison with typical market samples. The ways of increasing the efficiency of infrared radiating glass panels had been shown.

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ИНФРАКРАСНЫЕ НАГРЕВАТЕЛИ С ТОНКОПЛЕНОЧНЫМ ПРОВОДЯЩИМ СЛОЕМ, СИНТЕЗИРОВАННЫМ НА СТЕКЛО МЕТОДОМ МАГНЕТРОННОГО РАСПЫЛЕНИЯ

В.В. Литвиненко, И.Н. Шмидко, Е.В. Родионов

Рассматриваются инфракрасные нагревательные панели с тонким проводящим слоем. В качестве проводящего материала предлагается тонкопленочный слой оксидного полупроводника, что повышает надежность и стабильность нагревательного слоя. Анализируются характеристики материалов тонкой пленки оксидного полупроводника и обосновывается выбор пленки оксида олова для дальнейших исследований. Приведены результаты экспериментов и характеристики пленок в зависимости от технологических факторов их синтеза. Рассматриваются параметры инфракрасных лучистых нагревательных панелей с тонкопленочным нагревательным слоем. Установлены максимально допустимые электрические параметры инфракрасных излучающих панелей с тонкопленочным нагревательным слоем, а также возможность повышения эффективности инфракрасных панелей и экономии энергии.

ІНФРАЧЕРВОНІ НАГРІВАЧІ З ТОНКОПЛІВКОВИМ ПРОВІДНИМ ШАРОМ, СИНТЕЗОВАНИМ НА СКЛО МЕТОДОМ МАГНЕТРОННОГО РОЗПИЛЕННЯ

В.В. Литвиненко, І.М. Шмидко, Є.В. Родіонов

Розглядаються інфрачервоні нагрівальні панелі з тонким провідним шаром. Як провідний матеріал пропонується тонкоплівковий шар оксидного напівпровідника, що підвищує надійність і стабільність нагрівального шару. Аналізуються характеристики матеріалів тонкої плівки оксидного напівпровідника і обґрунтовується вибір плівки оксиду олова для подальших досліджень. Наведено результати експериментів і характеристики плівок у залежності від технологічних факторів їх синтезу. Розглядаються параметри інфрачервоних променистих нагрівальних панелей з тонкоплівковим нагрівальним шаром. Встановлено максимально допустимі електричні параметри інфрачервоних випромінюючих панелей з тонкоплівковим нагрівальним шаром, а також можливість підвищення ефективності інфрачервоних панелей і економії енергії.