Development, synthesis and production of multilayer thin-film filters for infrared sensors for monitoring of flammable gases and vapors of flammable liquids

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Abstract. We present the results of development and production of multilayer thin-film filters for infrared sensors for monitoring of flammable gases and vapors of flammable liquids. This paper addresses technological peculiarities of their production. A theoretical simulation of multilayer filter parameters to provide the transparency in wavelength range 3.3 - 3.5 μm was carried out. It is demonstrated that spectral characteristics of produced multilayer filter correspond to the calculated ones.

1. Introduction

Along with other global problems that arise in the modern world, concentration monitoring of ambient atmosphere components is a critical challenge in both production and household spheres. At the present time, the gas sensors based on various methods of measurement are used to monitor the concentrations of components in the air. The most widespread sensors are sensors based on electrochemical, thermochemical, optical, semiconductive and photoionization measuring principles [1–3]. In the concentration range of Lower Explosive Limit (LEL), catalytic sensors are most widely used, due to high sensitivity and selectivity, linear response and low cost. One of the main disadvantages of catalytic detectors is their degradation in toxic environment due to contamination. NDIR (non-dispersive infrared) optical sensors have crucial advantages, which allow to refer this sensor type to the most in-demand and perspective measuring devices. Advantages of optical sensors are: higher zero stability, sensitivity, selectivity, better performance, resistance to aggressive media and poisoning by high concentrations of monitored and produced gases, capability of functioning in anoxic environment.

Vis-IR lamps, light emitting diodes (LEDs), laser diodes and tunable distributed feedback lasers (dfb-lasers) are used as the light sources. However, the most widespread are xenon lamps. Vis-IR lamps have very wide spectra. So, one of the basic elements in optical sensor design that considerably defines its technical characteristics is multilayer thin-film filter that allows to select the required wavelength range from a wide range.

This paper presents experimental results of development, synthesis and production of multilayer thin-film filters for infrared sensor for monitoring of flammable gases and vapors.

2. Design of multilayer thin-film filters

Thin-film filter comprises several layers of transparent dielectric material with different refractive indexes applied one after another onto an optically transparent substrate [4].
Key parameters of filter spectral characteristic are the target wavelength ($\lambda_{\text{max}}$), the transmission level in transmission band maximum (T$_{\text{max}}$), the half-width ($\delta \lambda$) – width of spectral characteristic at T$_{\text{max}}$/2, the transmission level in suppression zone (T$_{\text{min}}$).

Values of these spectral characteristic parameters are defined depending on the type of measured component, concentration of which is monitored, and composition of gas medium, in which measurements are taken. It is commonly known that polyatomic molecules of substances can absorb the optical radiation [5]. The light absorption by substances can be typically observed within 1 – 12 $\mu$m infrared range. Data on spectral infrared absorption lines is given in specialized reference literature [6] and in data bases on the Internet [7]. This information gives a complete idea of spectral absorption intervals (absorption spectra) of the monitored component and components of the medium, in which measurements are taken, allowing to decide upon parameters of spectral characteristic of the optical filter.

Flammable gases and vapors of flammable liquids are mostly the products of extraction and refining of hydrocarbons, chemical structure of which is described by C-H, C-C, C-O molecular links. Based on the analysis of valence and deformation vibrations of these molecular links [6], we can distinguish infrared spectrum range most typical for them: 3.3-3.5 $\mu$m. From the analysis of absorption spectra of methane and its homologues [7], the most common hydrocarbons, it follows that wavelength $\lambda_{\text{max}}$ of the developed filter must be within 3.3-3.5 $\mu$m wavelength range.

Therefore, optical filter of infrared sensor for monitoring of flammable gases and vapors of flammable liquids was assigned the following parameters: $\lambda_{\text{max}}=3.4$ $\mu$m; $\delta \lambda=160$ nm; T$_{\text{max}}\rightarrow100$ %, T$_{\text{min}}\rightarrow0$ %. Suppression zones of the required filter are defined with allowance for medium composition, in which measurements are taken, as well as spectral characteristics of emitters and detectors included in optical sensor. Filter suppression zones we selected: short-wave region – from 2 to 3.2 $\mu$m, long-wave region – from 3.7 to 5 $\mu$m.

3. Results and discussions

Assigned parameters of spectral characteristic are initial conditions for completion of multilayer thin-film filter designing or synthesis stage. A wide variety of publications that describe existing calculation techniques is dedicated to the synthesis of interference filters [8]. Among them are recursive and matrix methods. Existing methods of thin-film filter synthesis are used as the basis in dedicated software products, such as FilmMgr, Essential Maclleod and Optylaer, which are designed for the given tasks.

At the first stage, we used Optylaer version 5.49 to simulate parameters of multilayer thin-film filter. Filters are made using vapors of materials with low and high refractive indexes. However, these materials must have minimal absorption within operating wavelength range. Due to technological capabilities of layer sputtering, requirements for optical parameters of filters and their operation conditions, as functional layers we selected: germanium (as material with high refractive index n=4.22, H) and silicon oxide (as material with low refractive index n=1.43, L). Quartz plates with thickness of 1 mm were used as substrate. The simulation we had conducted showed that the following filters have the best optical parameters:

- narrow-band filter: 13 layers, sputtering formula: LHL2HLHLHL2HLHL, layer thickness: L=0.593 $\mu$m, H=0.202 $\mu$m
- cut-off filter: 12 layers, sputtering formula: 0.45HLHLHLHLHL0.6H2.4L, layer thickness: L=0.348 $\mu$m, H=0.117 $\mu$m.

Figure 1 illustrates the calculated and experimental dependencies of narrow-band, edge and combined multilayer thin-film filters. The curve 1 (Figure 1) is the calculated dependence of narrow band filter. The curve 2 is the calculated dependence of cut of filter. The multilayer thin-film filter dependence (curve 3) is obtained by a combination of a narrow-band (curve 1) and cut-off (curve 2) dependencies.

For creation of multilayer thin-film filter, we considered several variants of vacuum coating equipment that provides the following sputtering options: electron-beam, ion-beam, magnetron, resistive. According to the results of the analysis of suggested sputtering variants, regarding imposed tech-
nical requirements and requirements to furnish filters with high performance attributes, we selected ion-beam (for silicon oxide layers) and magnetron (for germanium layers) sputtering methods.

![Graph](image-url)

**Figure 1.** The calculated and experimental spectral characteristics of multilayer thin-film filter: 1 - the calculated dependence of narrow band filter; 2 - the calculated dependence of cut of filter; the calculated dependence (curve 3) and experimental dependence of produced multilayer thin film filter (curve 4).

We used Aspira-150 vacuum coating equipment manufactured by “Isovac” (Belarus) as the vacuum unit. The schematic view of the vacuum unit is given in figure 2.

Sputtering of multilayer thin-film coating was under the following conditions: residual pressure – 10⁻³ Pa, substrate heating temperature – 100 °C, used working gases – argon (Ar), oxygen (O₂). For layer growth control, we used the built-in Invisio M optical monitoring system.

Figure 1 shows the calculated (curve 3) and experimental (curve 4) dependencies for the developed multi-layer filter. For measurement of spectral characteristic, we used Varian 640-IR infrared spectrometer (USA).

It can be seen that the calculated and experimental dependencies coincide well with each other. The slight difference in T\text{max}, and δλ can be explained by the following influential factors: deviation of actual refractive index values of used materials from the estimated values, disorder of the film stoichiometric composition due to the presence of contaminants in vacuum environment, thickness variations in applied material layers.

4. Conclusions

Using vacuum coating equipment with magnetron and ion-beam options for thin-film filter sputtering, we produced the optical pass filter in the range 3.3-3.5 μm for infrared sensor for monitoring pre-explosive concentrations of flammable gases and vapors of flammable liquids.

Germanium (as material with high refractive index) and silicon oxide (as material with low refractive index) were chosen as functional layers for multilayer optical pass filter. The ion-beam deposition (for silicon oxide layers) and magnetron sputtering (for germanium layers) were used for the producing optical pass filter.

The calculated and experimental dependencies are in a good agreement. The slight deviation of experimental filter parameters from calculated values is explained by consistent process influential factors. The presented development stages can be used for synthesis and production of multilayer thin-film filters for optical sensors for various applications, for example, for non-dispersive open path detectors [9].
Figure 2. Layout of Aspira-150 vacuum assembly. 1 – vacuum chamber; 2 – substrate holder rotary actuator; 3 – light source of optical monitoring system; 4,5 – entrance/exit window of optical monitoring system; 6 – substrate load lock system; 7 – substrate holder; 8 – substrate; 9 – ion-beam sputtering system; 10 – DC magnetron; 11 – ion-beam purification system; 12 – monochromator of optical monitoring system (Čzerny-Turner); 13 – controller; 14 – PC; 15 – gas puffing system; 16 – pumping system: 16.1 – dry pump, 16.2 – turbomolecular pump.

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