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Investigation of thermal sensitivity and radiation resistance of $SiO_x\langle Ti\rangle$ metal-dielectric films

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HIGHLIGHTS

- We investigated $SiO_x\langle Ti\rangle$ films as materials for thermosensitive detectors.
- In composite $SiO_x\langle Ti\rangle$ films the metal concentration in the dielectric matrix was varied with thickness.
- After annealing at 500 °C neither resistance nor TCR of MDTF were changed during storage.
- The gamma irradiation has very little effect on sample characteristics up to a dose of 10^5 Gy.
- $SiO_x\langle Ti\rangle$ MDTF are promising for thermosensitive detectors operated in γ-radiation fields.

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ABSTRACT

In this investigation the composite $SiO_x\langle Ti\rangle$ films were prepared by the thermal evaporation of a mixture of silicon oxide (SiO_2) and Ti powders. The optical transmission of the films in the IR spectral range and their temperature-sensitive properties are studied. By varying the contents of the metal in vaporizer and time of evaporation it is possible to obtain $SiO_x\langle Ti\rangle$ layers with resistance (for monopixel of 0.8 × 1 mm) from tens kOhms to MOhms and a value of the temperature coefficient of resistance (TCR) is equal to $-2.22\%\ K^{-1}$. IR spectrum of $SiO_x\langle Ti\rangle$ film is characterized by a broad absorption band in the range of 8–12 μm which is associated with the Si–O–Si stretching mode.

Investigations of the effect of gamma irradiation on $SiO_x\langle Ti\rangle$ films have shown that their temperature-sensitive properties, in particular TCR does not change up to a dose of 10^6 Gy.

These results suggest that $SiO_x\langle Ti\rangle$ films can be used as materials for production of radiation-resistant thermosensitive detectors operated in radiation fields of γ-radiation and combining functions of IR-absorption and formation of an electric signal.

1. Introduction

In recent years, much attention has been concentrated on the design of uncooled infrared detectors where the temperature-sensitive element is made in the form of a chain or array consisting of identical uncooled microbolometer structures [1–5]. The microbolometer converts absorbed infrared radiation into heat, which in turn changes its resistance. The uncooled microbolometer thermal detector has advantages over photon detector such as low cost, low weight, low power, large spectral response, and long term operation. The sensitive element of a microbolometer usually contains an active temperature-sensitive layer with a high value of the temperature coefficient of resistance (TCR) and an absorbing coating that absorbs IR radiation.

A wide variety of materials have been used as active element for microbolometer. Vanadium oxide (VO_x) [6,7] is the most widely used material due to its high TCR of 2–3%/K. The main drawback of VO_x is that it is not compatible with IC fabrication process. An IC compatible microbolometer material is the amorphous silicon carbide (SiC) [8], or amorphous semiconductor films [9–11] but their process temperature is high for post-CMOS processing. A low temperature and IC compatible is metal film microbolometers [12], but metals have very low TCR, limiting their performance.

As the absorbing coating in microbolometers can be used thin metal films (an absorption of near 50% in a wide spectral region) or multilayer structures for which the resonant absorption may reach 90% in a certain spectral range (usually second atmospheric window $-\lambda = 8–12$ μm). However, presence of these additional layers increases essentially the heat capacity of a microbolometer and worsens its characteristics. Therefore, search for materials that would combine the functions of temperature-sensitive and

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absorbing layers seems to be perspective. It was shown by us earlier [13,14] that inhomogeneous metal-dielectric films deposited in vacuum are characterized by high selective absorption in IR – spectral area (8–12 μm) which is combined with acceptable values of TCR (>1%). Varying the contents of metal in metal-dielectric film it is possible to change its resistivity in very wide limits: from characteristic for dielectric-oxide up to metal. Offered metal-dielectric thermosensitive films (MDTF) have the following advantages: high stability of physical and chemical properties, absence of toxic components, compatibility with IC fabrication technology, low commercial cost. This makes it possible to accomplish the absorption of IR radiation and the formation of an electrical signal in a single layer.

An important problem of practical exploitation of MDTF bolometers for high-energy physics and space applications is the stability of their parameters under extreme conditions of external factors (ionizing radiation, strong magnetic fields, etc.). In this paper we investigate thermosensitive properties of metal-dielectric $SiO_x\langle Ti\rangle$ coating under the influence of ionizing radiation (gamma-ray photons).

2. Experiment

Samples for investigations were prepared by the method proposed earlier to produce light-absorbing inhomogeneous SiO and Cr-based coatings [15]. Its basic idea is the thermal coevaporation of a mixture of these materials in a vacuum from a single evaporator. In this investigation, a mixture of silicon oxide (SiO_2) and Ti powders was evaporated on glass, pyroceramic, silicon or germanium substrates at a pressure of near $2–3 \times 10^{-3}$ Pa. The average metal-to-oxide mass ratio in a sample thus obtained was roughly determined from the ratio of the SiO_2 and Ti masses in the evaporator, and the distribution of the components was specified by the evaporation regime also. Temperature of intensive Ti evaporation in vacuum is slightly higher than silicon oxide. That is why under conditions of gradual increase of evaporator temperature SiO_2 evaporation and deposition takes place first with small addition of Ti, than simultaneous SiO_2 and Ti deposition with increased addition of Ti. Layers thus obtained are composite $SiO_x\langle Ti\rangle$ films in which the metal concentration in the dielectric matrix varies with thickness: the concentration of the metal is maximal near the film surface and minimum at the interface film-substrate. Such a distribution specifies the optical and electrical properties of these inhomogeneous metaldielectric layers.

The deposition process was controlled by a calibrated quartz resonator. The thickness of the films measured by a microinterferometer and was changed in the range between 0.3 up to 3 μm.

The reflection and transmission spectra in the IR spectral range were taken from the films deposited on Si(1 1 1) or Ge substrates using Perkin-Elmer Spectrum BXII spectrometer. To examine the temperature-sensitive properties (TCR and resistance), we used the films deposited on the pyroceramic and thin glass substrates in a vacuum. Samples had the form of a planar chip of near square millimeter in area. On the top of the chip, Ag contacts were deposited to which thin wires were soldered. During the measurements, these MDTF bolometers were placed in a thermostat U-10 and the bias current was chosen in such a way as to prevent Joule heating.

Gamma irradiation of the MDTF bolometers was carried out at room temperature using a ^{60}Co (E = 1.33 MeV) gamma rays source (MPX-γ-25M) with the dose rate of 0.18 Gy/s from small doses up to integral doses of 10^6 Gy. After each gamma-ray exposition measurements of the sample resistance were made at the temperature range of 273–333 K, and then values of temperature coefficient of resistance TCR (% K^{-1}) where calculated.

3. Results and discussion

Fig. 1 shows IR transmittance of $SiO_x\langle Ti\rangle$ MDTF film (thickness 1.83 μm). The ratio of SiO_2 and Ti mass in the evaporator made 80:20. The mode of deposition was selected such to receive non-uniform distribution of phases in the film with the maximal content of metal on its surface. As follows from Fig. 1, IR spectrum of $SiO_x\langle Ti\rangle$ film is characterized by a broad absorption band in the range 8–12 μm. The nature of this band is associated with the Si–O–Si stretching mode.

To determine the TCR in the inhomogeneous MDTF films, we recorded the temperature dependences of their resistance, and then values of TCR were calculated from temperature dependences of resistance r:

$$\mathrm{TCR} = \frac{1}{r}\frac{dr}{dt}$$

Test samples were 0.8×1-mm chips on pyroceramic substrates. Fig. 2 (curve 1) shows temperature dependence of resistance r of $SiO_x\langle Ti\rangle$ MDTF film (in the coordinates lnr versus $1/t$, where t – Kelvin temperature) thickness of the film was equal 0.86 μm and the ratio of SiO_2 and Ti mass in the evaporator was equal to 80:20, also. With increasing temperature r decreases, i.e. film has negative TCR, and its value at room temperature is equal $-2.21\%\ K^{-1}$ (Fig. 3). The negative TCR value and exponential decreasing of resistance is likely to be associated with the conduction mechanism in these films (tunnel conduction through percolation channels like, for example, in Au–SiO cermet films of similar composition, for which this mechanism has been studied rather extensively [16]).

By varying the contents of the metal in vaporizer and time of evaporation it is possible to obtain $SiO_x\langle Ti\rangle$ layers with resistance (for monopixels the specified size) from tens kOhms to MOhms, which falls into the interval of optimum resistances for microbolometers [1]. Thus, TCR of $SiO_x\langle Ti\rangle$ films at room temperature varies from -1.9 to $-2.22\%\ K^{-1}$. The maximum TCR values were observed for highest resistivity samples.

For investigation how electrical and optical properties of gradient MDTF vary through the film cross-section, we have deposited one $SiO_x\langle Ti\rangle$ coating onto two substrates. During evaporation of SiO_2–Ti mixture we have changed two substrates and have obtained two sub-layers (partial layers) which present different parts (in cross-section) of whole coating – bottom and top: bottom layer, 600 nm thickness, and top layer – 260 nm. Integral thickness

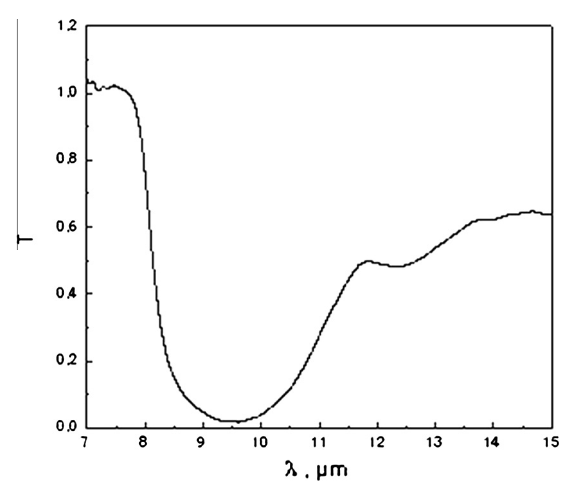

Fig. 1. Spectral dependence of transmittance (T) of $SiO_x\langle Ti\rangle$ MDTF film (λ – wavelength). The ratio of SiO_2 and Ti mass in the evaporator is equal to 80:20, and thickness of the film – 1.83 μm.

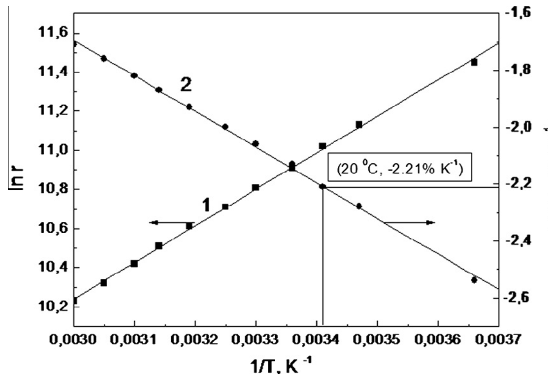

Fig. 2. Resistance (r) of $SiO_x\langle Ti\rangle$ MDTF film (curve 1) and its TCR (curve 2) (the size of sample is 0.8 × 1 mm, thickness 0.86 μm).

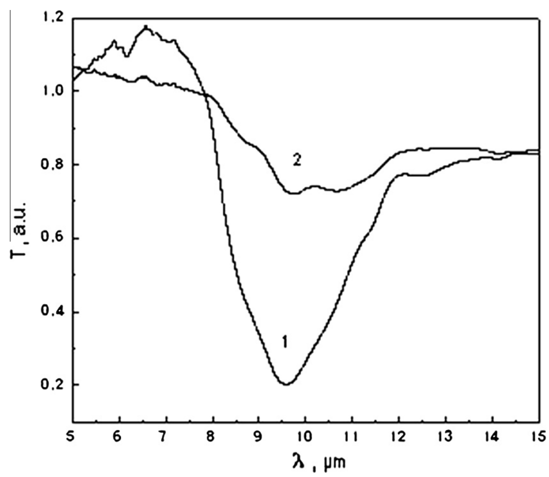

Fig. 3. Transmittance of the two partial layers of $SiO_x\langle Ti\rangle$ MDTF: bottom (curve 1), and top (curve 2) layer.

of partial layers was equal to thickness of MDTF coating deposited onto single substrate (0.86 μm).

Fig. 3 shows transmittance of these two partial layers: bottom (curve 1), and top (curve 2). Bottom layer have a broad absorption band, associated with the Si–O–Si stretching mode. But top layer have only weak absorption band in this spectral range. Bottom layer has high resistance, near 1 GOhm, but resistance of the top layer – near 400 KOhm. These results show gradient structure of $SiO_x\langle Ti\rangle$ films: bottom (near substrate) part of the film consists mostly of oxide component and characterized by high resistance. Top metal- or silicide-rich part of the $SiO_x\langle Ti\rangle$ MDTF (near surface) has sufficiently low resistance, which may be optimized (by varying the contents of the metal in vaporizer and mode of evaporation) for microbolometer applications. This top layer of the film determined the electrical properties of MDTF.

After deposition of $SiO_x\langle Ti\rangle$ MDTF its resistance was changed. For stabilizing of MDTF characteristic we investigated the influence of the different treatments – annealing, cooling. It was revealed that the best results are given by annealing in a vacuum at a temperature about 500 °C. Such annealing results in decreasing of film resistance in several times; after annealing neither resistance nor TCR of MDTF was changed. Fig. 4 shows change of resistance (in relative units) during storage at room temperature of initial, unannealed sample (curve 1) and annealed in vacuum (curve 2). It is seen that resistance of the unannealed sample increases at storage. Resistance of annealed, stabilized samples remains unchanging at storage during 6 years. Does not change and TCR of this MDTF (TCR value for this sample is equal $-2.2\%\ K^{-1}$).

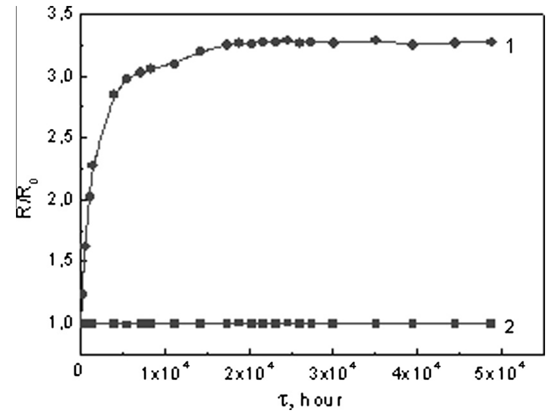

Fig. 4. Aging of resistance of initial (1) and stabilized (2) $SiO_x\langle Ti\rangle$ – film. r_0 – initial resistance of sample after evaporation, r – resistance of sample after storage, τ – time of storage at room temperature.

Investigations of the effect of gamma irradiation on $SiO_x\langle Ti\rangle$ MDTF were carried out at room temperature using ^{60}Co as the source of gamma rays. After gamma-ray exposition measurements of the sample resistance were made at different temperatures and then values of TCR were calculated. Fig. 5 shows the $SiO_x\langle Ti\rangle$ MDTF resistance (a) and TCR (b) as a function of the gamma-ray dose measured at 293 K.

As shown in Fig. 5, the gamma irradiation has very little effect on sample characteristics up to a dose of 10^5 Gy. At higher doses the sample resistance increases, while the value of TCR does not depend on the dose of γ-radiation in the whole investigated range. This means that the change in resistance was caused, most likely, by the degradation of contacts, while the thermosensitive properties of the $SiO_x\langle Ti\rangle$ MDTF in this dose range did not change.

It is known that the main mechanism of contact degradation in a high radiation environment is the mass transfer of the metal layer into the thermosensitive structure [17]. The process of the contact degradation may change the resistance and thermosensitivity of bolometers. Further improvement in the radiation-resistance of $SiO_x\langle Ti\rangle$ MDTF bolometers can be achieved by improving the design and increasing of the radiation tolerance of the electrical contacts to the MDTF sensitive element. This can be achieved by the development of multilayer metal contact structure.

These results suggest that $SiO_x\langle Ti\rangle$ MDTF can be used as material for production of radiation-resistant thermosensitive detectors operated in γ-radiation fields.

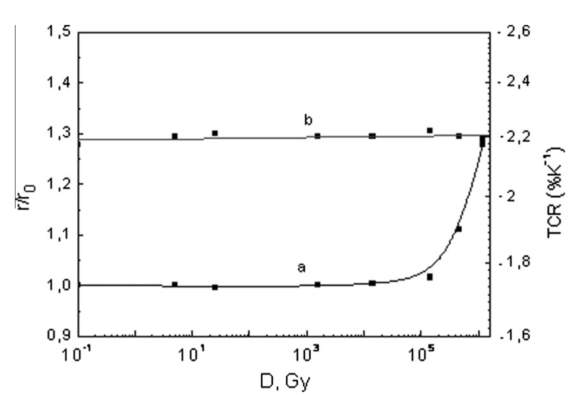

Fig. 5. The dependence of the resistance r/r_0 – (a), (where r_0 = 61.36 kOm – resistance of the sample before radiation), and the temperature coefficient of resistance TCR – (b) on the dose D of the gamma-rays (at 293 K). Film with the same parameters as in Fig. 2.

4. Conclusions

Investigated in this paper $SiO_x\langle Ti\rangle$ MDTF, deposited by thermal vacuum evaporation of SiO_2 and Ti mixture, has gradient structure: bottom (near substrate) part of the film consists mostly of oxide component and characterized by high resistance, but top metal- or silicide-rich part (near film surface) has sufficiently low resistance, and this top layer of the film determines the electrical properties of MDTF. TCR of $SiO_x\langle Ti\rangle$ films at room temperature varies from -1.9 to $-2.22\%\ K^{-1}$. IR absorption spectrum of $SiO_x\langle Ti\rangle$ MDTF is characterized by a broad absorption band in the range 8–12 μm which associated with the SiO–O bond vibration mode.

Annealing of $SiO_x\langle Ti\rangle$ films in a vacuum results in decreasing of film resistance in several times and stabilizing of electrical characteristics; after annealing resistance and TCR of MDTF remain unchanged at storage during 6 years.

Gamma irradiation has very little effect on sample resistance up to a dose of γ-radiation of 10^5 Gy, but TCR does not change up to 10^6 Gy. Such MDTF can be used as a γ-radiation-resistant thermosensitive layer of the microbolometer combining functions of absorption of IR – radiation and formation of an electric signal. Offered thermosensitive layers have the following advantages: high stability of physical and chemical properties, absence of toxic components, compatibility with IC fabrication technology, low commercial cost.

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