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Research of the Possibility of the Silicon-Carbonic Diamond-like Films Application in the Manufacturing of SmS Strain Gauges on Metal Base.

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ABSTRACT

The object of the present study is the possibility of the application of films which belong to the class of diamond-like (DLF – diamond-like film), i.e. containing a substantial concentration of carbon atoms linked by SP3 bonds typical for the structure of diamond to be further used as the dielectric and protective layer to obtain strain-sensing gauges based on samarium monosulphide (SmS). Unlike other traditional diamond-like films (a-C:H) films under consideration contain circuits ... -Si-O-Si-O- .... This feature provides diamond-like silicon-carbonic films (SC DLF) with a unique set of physical and chemical properties and a wide range of applications. The study was conducted in order to create the technology of thin-film interlayer dielectric with high dielectric strength suitable for various types of gauges (piezoresistive, thermal, etc.) intended for abrasive and chemically aggressive media.

Keywords: strain gauges, samarium monosulphide, diamond-like coating, plasma-chemical deposition, precursor, polyphenyl methylsiloxane.

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INTRODUCTION

Semiconducting strain gauges of mechanical values on the basis of samarium monosulphide are superior to all existing strain gauges on the basis of other materials in terms of their parameters [1-3]. The manufacturing technology of strain gauges on the basis of samarium monosulphide includes the preparation of the metallic substrate, the application of an insulating dielectric film, the application of metal bond pads, the application of samarium monosulphide, and the application of protective film. Traditionally, silicon monoxide is used as the dielectric film. However, this material has a number of drawbacks: high porosity, poor adhesion, etc.

METHOD OF THE EXPERIMENT

To create a dielectric that would meet the requirements of dielectric strength, chemical and abrasion resistance, it is possible to use materials from the class of diamond-like coatings (DLF). The condition for chemical and abrasive resistance is carried out automatically for all coatings in this class. To identify materials with maximum dielectric strength, we conducted an experimental study to compare electric strength of DLF obtained by plasma-chemical deposition from a variety of precursors. Deposition was carried out with the help of the installation described below. Installation diagram for DLF deposition is shown in Figure 1.

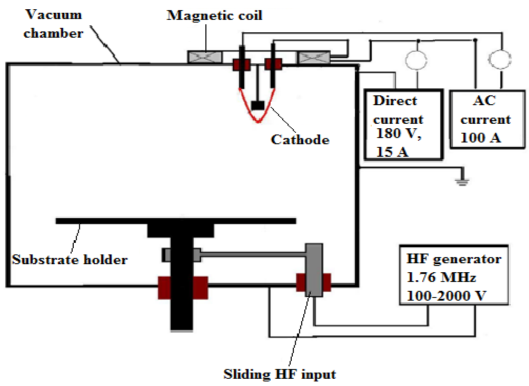

Figure 1. Installation diagram for the deposition of diamond-like films.

The process of deposition was carried out in a vacuum chamber, pre-evacuated to a pressure of $5 \cdot 10^{-5}$ mmHg; then under the pressure of argon $(3\text{-}4) \cdot 10^{-4}$ mmHg, we conducted the cleaning of substrate with HF plasma or partially ionized directional flow of Ar atoms with negative pulse potential on the substrate holder (SH). Precursors used to obtain DLF are listed in Tables 1 and 2. The precursors that are in a gaseous state under normal conditions were put into the operating chamber through a standard adjustable needle leak valve. The precursors that are in a liquid state under normal conditions were put with the help of a specially designed plasmatron [2] shown in Figure 2.

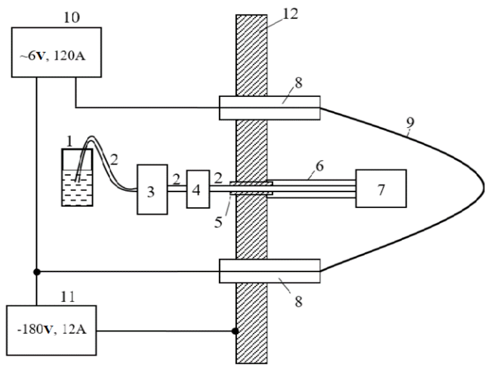

Figure 2. Schematic diagram of the structure of plasmatron for liquid precursors.
1 – container with a precursor, 2 – feeding tube for a precursor, 3 – pressure pump, 4 – back-flow valve, 5 – vacuum seal, 6 – protective tube, 7 – porous ceramic nozzle, 8 – current lead of tungsten cathode, 9 – tungsten cathode, 10 – power unit of cathode glowing current, 11 – power unit of cathode bias potential.

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A detailed description of the technology of the growth process can be found in [3].

RESULTS AND DISCUSSION

Dielectric Strength of Diamond-like Films

We conducted the studies of the dielectric strength of a series of films belonging to the class of DLF which, according to the literary data, possess suitable dielectric properties to be further used as a dividing dielectric of low frequency devices. We used breakdown voltage of the film as a reference parameter. The value of this parameter should comprise at least $1 \cdot 10^6$ V·cm. We used the method of measuring the i-v curve of dielectric films on ceramized substrate with a metal sublayer; we also used a “point” electrode (the sphere with a diameter of 0.5 mm); the negative potential was given in to the metal sublayer on the substrate. The results are summarized in Table 1.

Table 1. Results of the study of dielectric strength of films deposited from plasma of simple hydrocarbons

Precursor	Operating pressure, Torr	Plasma current, A	Speed of growth, µm/h	Dielectric thickness, µm	Voltage of the breakdown field, V/cm
Methane	$3 \cdot 10^{-4}$	3,5	0,2	0,1	$5 \cdot 10^4$
Acetylene	$2 \cdot 10^{-4}$	4,5	0,5	0,1	$2 \cdot 10^5$
Benzene	$3 \cdot 10^{-4}$	6	0,08	0,2	$4 \cdot 10^5$
Acetone	$3\ 10^{-4}$	4	0,1	0,05	$2 \cdot 10^4$

The results of the measurements showed that conventional carbon coatings can not ensure dielectric strength of obtained films in the required range.

As a positive alternative, we tested the films obtained by the deposition of siloxane vapors from plasma – silicon-carbonic compounds containing high carbon concentration in SP3 state and the chain of atoms ... -O-Si-O-Si- .... Literary data and the data of our own research allowed the authors to predict the values of the voltage of the breakdown field that are bigger than 106 V/cm. We selected polyphenyl methylsiloxane (PPMS), methyl silicone (MS) and the mixture of MS with benzene as starting siloxanes.

The results of investigations and tests are shown in Table 2.

Data provided in Table 2 allowed us to make the choice in favor of PPMS (brand: PPMS 2/5L) as the starting material for dielectric films that meets the requirements of the interlayer dielectric. Therefore, further research and development of materials and technologies were based on PPMS.

Table 2. Results of the study of dielectric strength of films deposited from siloxane vapor plasma

Precursor	Operating pressure, Torr	Plasma current (A)	Speed of growth (µm/hour)	Dielectric thickness (µm)	Voltage of the breakdown field, V/cm
MS	$2 \cdot 10^{-4}$	6,0	1,0	0,1	No adhesion
MS + C_6H_6	$3 \cdot 10^{-4}$	7,0	1,0	0,1	$0,8 \cdot 10^6$
PPMS	$1,5 \cdot 10^{-4}$	6,0	1,0	0,1	$3 \cdot 10^6$

The Kinetics of SC DLF Growth

The reproducibility of the deposition technology is provided by means of reliable information about the dependence of film growth rate on the value of the process parameters. It is important to note that there is one set of parameters for different installation solutions of the growth process implementation, and specific values can vary considerably and are identified with the help of technological experiments. In this study, the authors relied on the findings of the earlier work [4], where it was shown that the rate of growth depends on the diameter of the SH and the value of the constant component of its potential. Both dependences are monotone and decreasing with the growth of the corresponding parameter.

The experience of SC DLF deposition also shows that the thickness of the grown film depends on the volume of the PPMS flow in the vacuum chamber. The adjustment of the feed rate in the manual mode of work is performed with the help of the needle valve while the work in automatic mode is performed with the help of a special delivery pump. We have experimentally studied the relationship between PPMS flow volume in three different processes different in volume of the consumed precursor and thickness of the deposited films. The feed rate was the same and the consumed volume was strictly proportional to the timing of the process. Total consumption comprised 17 units, 30 units and 60 units (the volume of one unit corresponds to 0.05 cm^3).

The deposition was performed on a substrate holder fixed on a series of silicon substrates 20 x 20 x 0.4 mm^3 in size placed along the horizontal diameter of SH at a certain distance from each other. Figure 3(a) presents the thickness of deposited layers depending on the position on SH and the amount of consumed PPMS. Figure 3(b) shows the ratio of thicknesses of grown layers to the thickness of the thinnest of them. For comparison, there are ratio lines of consumed volumes of PPMS. As can be seen from these two figures, the thickness of the deposited film is strictly proportional to the volume of consumed PPMS. The obtained result allows getting SC DLF with a predetermined thickness and an accuracy of ± 3%.

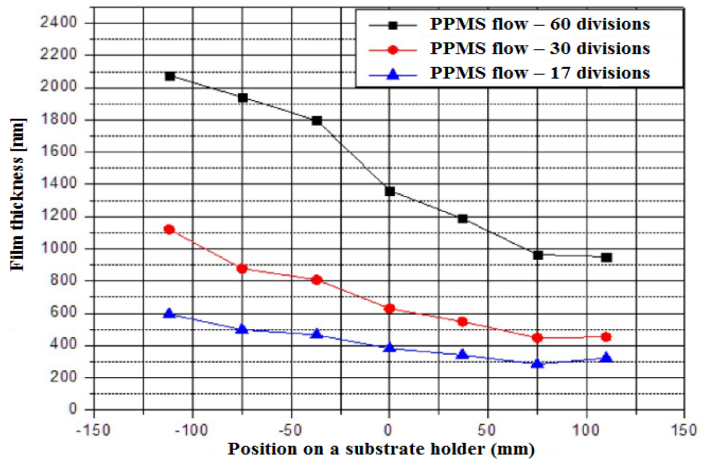


Figure 3 (a). Thickness of deposited layers, depending on the position of SH and consumed volume of PPMS.

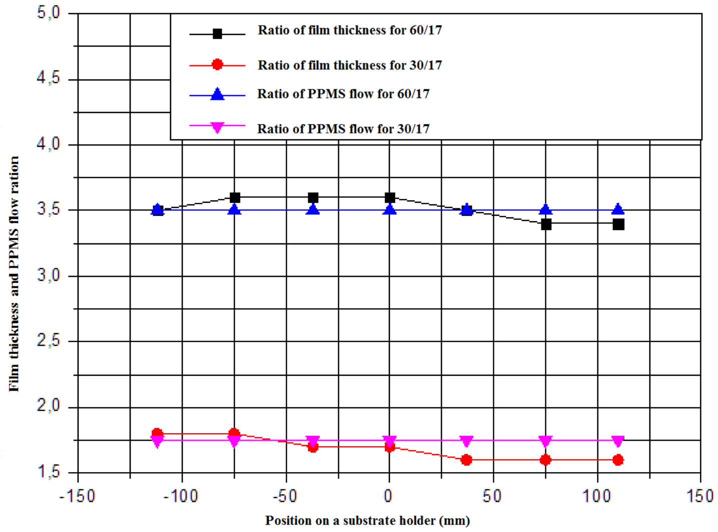


Figure 3 (b). Ratio of thickness of the grown layers to the thickness of the thinnest of them. For comparison, there are ratio lines of the consumed volumes of PPMS.

Uniformity of Film Thickness

Another important parameter of the reproducible technological process of the film deposition is the uniformity of thickness, which plays a significant role when it comes to obtaining sensors based on samarium monosulphide (SmS) with reproducible parameters. In order to maintain this parameter in required limits it is necessary to use uniform distribution of the growth rate (DGR) of the film on the substrate holder. The conducted experiments showed that this parameter depends on two process parameters: the shape of the porous ceramic nozzle of the plasmatron and the distance between plasmatron and substrate holder.

The Study of the Influence of Ceramic Nozzle Shape of the Plasmatron on DGR

To assess the influence of the shape of the porous ceramic nozzle on DGR we selected three types of nozzles given in Table 3.

Table 3. Type and shape of the ceramic nozzle (horizontal section)

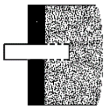
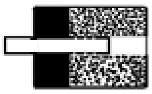
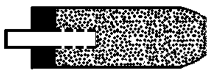
“Rectangle” Nozzle	“Nozepiece” Nozzle	“Cone” Nozzle
 of frontal surface 40 x 20 mm, radius of curvature 140 mm	 Diameter of the nozzle 4 mm	 Diameter of a flat front cut – 10 mm

Figure 4 shows the distribution of thickness of films grown with the help of three nozzles given in Table 3.

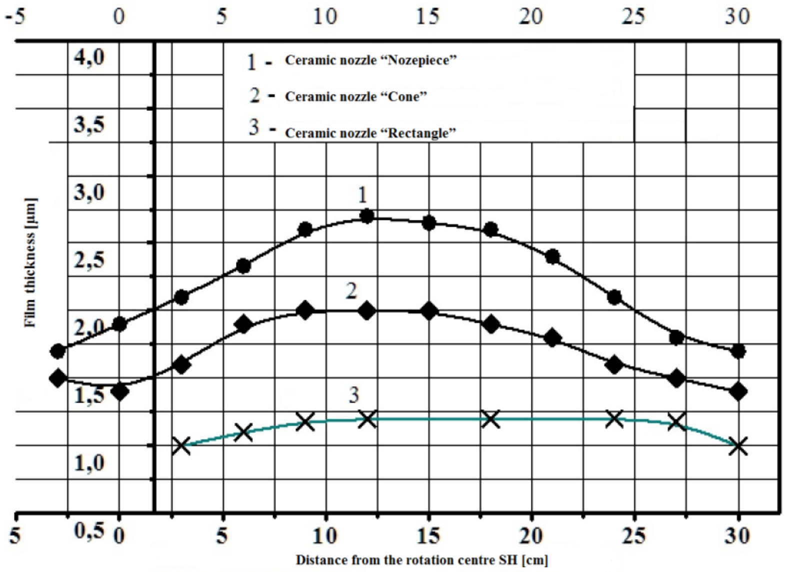


Figure 4. The distribution of the thickness of the film grown with the help of three different ceramic nozzles.

These results demonstrate the possibility of forming precursor streams of varying intensity and uniformity by changing the shape of the nozzle. This feature makes the process flexible and adjustable enabling us to achieve maximum effectiveness for various sizes of substrates, different numbers and different dimensions of the substrate holder.

The dependence of the distribution of the growth rate on the distance plasmatron – the substrate holder is illustrated in Figure 5. The dependence is preserved for all types of ceramic nozzles. The resulting dependences are quite predictable, but their specific parameters are necessary to set the process in order to optimize it. In this installation solution of the process, the change in the distance of plasmatron – SH is also the only way to change the substrate temperature and SH.

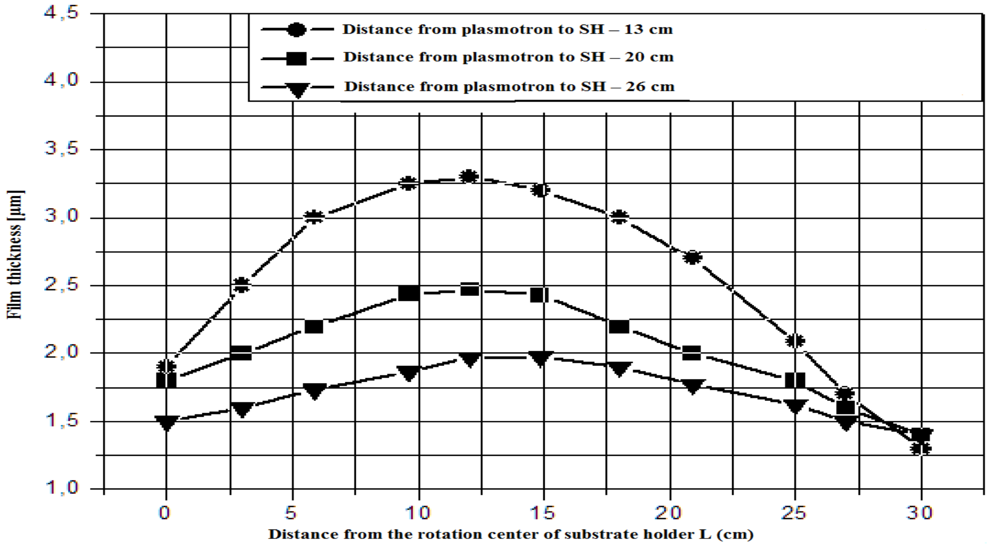


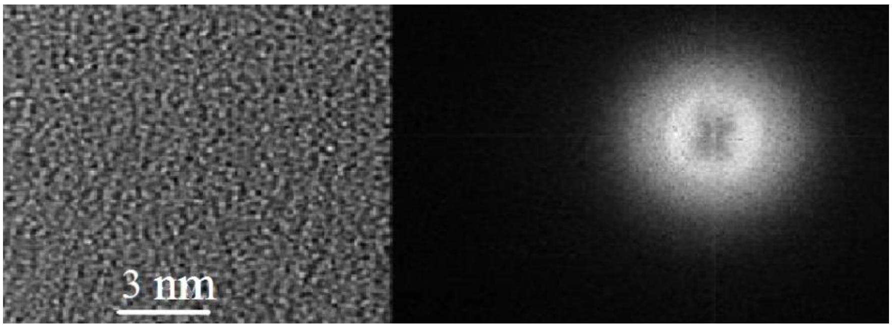
Figure 5. Dependence of the distribution of the film thickness on SH Grown at different distances between plasmatron – substrate holder.

As a result of the conducted technological experiments, we found out that the deposition mode with distance plasmatron – substrate holder is 26 cm and the ceramic "Cone" nozzle enables us to obtain a film with the spread of thickness of less than 3% on a plot of a rotating substrate holder with a diameter of 20 cm, which provides acceptable efficacy of the process.

The results obtained in the framework of technological experiments will be used in the manufacture of sensor gauges on the basis of samarium monosulphide to obtain homogeneous and dielectric and abrasion-proof coatings with equal thickness belonging to the class of DLF.

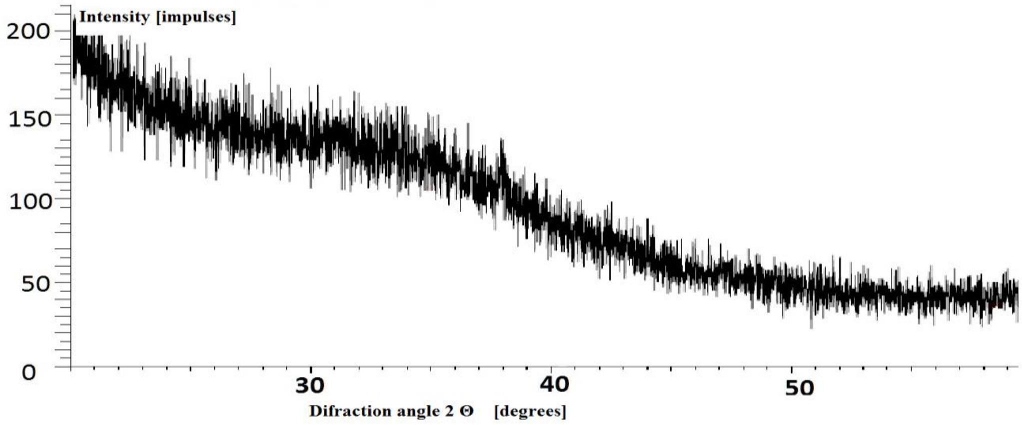
The Structure and Physical-Chemical Properties of SC DFL

As with most solid substances, physical and chemical properties of SC DFL are defined by their atomic structure and the structure of chemical bonds. On the other hand, both of these parameters can depend on the process parameters. In this regard, we conducted studies on the samples obtained with the help of the technology described above. Atomic structure was investigated by means of X-ray diffraction and electron diffraction. Both methods showed that the atomic structure of SC DFL is amorphous. Figure 6 (a) shows electron diffraction pattern on the clearance of a thin cut of SC DFL on the right and the electron microscopic image of the atomic structure of SC DFL. In the latter one, there are short linear formations of atoms, which can be interpreted as fragments of -Si-O-Si-O- chains.



a

Figure 6 (a). Electron diffraction and electron microscopic image of the atomic structure of SC DFL.



b

Figure 6 (b). X-ray diffraction pattern.

The X-ray diffraction pattern shown in Figure 6 (b) corresponds to the amorphous state of the object under consideration (SC DFL) and confirms the data of electron diffraction.

The information about the structure of chemical bonds in SC DFL was obtained by means of infrared spectroscopy. Figure 7 shows IR spectra of the sample absorption of SC DFL and PPMS.

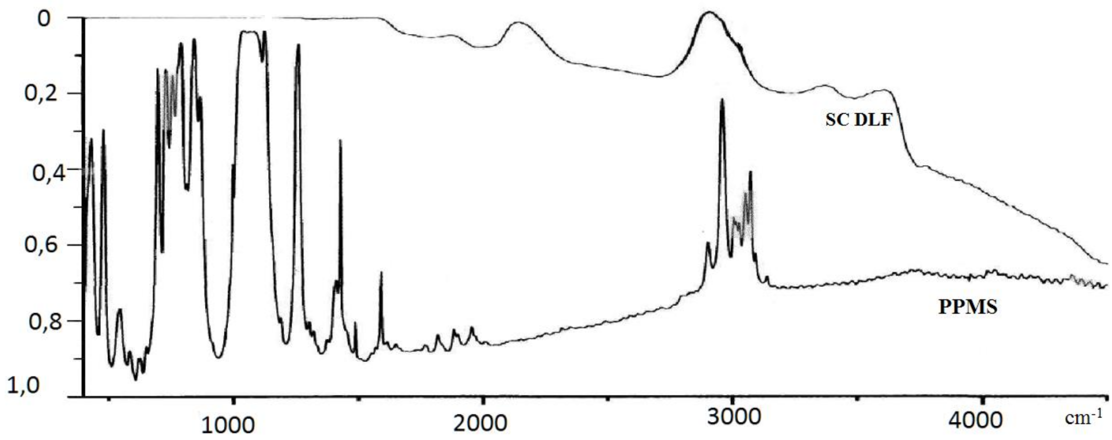


Figure 7. IR absorption spectra of SC DLF and PPMS. Scale Y reflects the value of the absorption spectrum of PPMS. The spectrum of SC DLF is combined with the spectrum of PPMS only on the X axis.

Table 3 shows the IR absorption lines found in the film SC DLF. Some of them are located below 1,700 1/cm and it is not reflected in Figure 7 due to high absorption of the film (the thickness of the film SC DLF under consideration comprised 100 µm). Deciphering of this range was made by other thinner patterns.

Table 3. The maximum of IR absorption radiation detected in SC DLF

Indication of absorption maximum	Value of the wave number	Reason of absorption (functional groups and bands)	
1	810	δ(Si-CH_3)	
2	1010	ν(Si-O-Si) absorption maximum shows the integrity of silicon-oxygen chains	
3	1550	Si-C_6H_5, this peak indicates the presence of aromatic rings in the film under consideration	
4	2130	Si-H these absorption maximums are not observed in PPMS	
5	2410		
6	2860	ν^s (-CH_3) maximum inherited from PPMS	
7	3200	ν (Si-OH)	Both maximums are not observed in PPMS
8	3610	ν (Si-OH)	

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When comparing the IR spectra of SC DLF and PPMS (the spectrum of the latter one can be easily deciphered with the help of the tables [5]), it can be seen that films inherit such structural elements as aromatic rings, chains (... -Si-O-Si-O- ...), (Si- C6H5), as well as methyl groups (valence (-CH3) 2863 1/cm and deformation vibrations (-CH3) 805 1/cm). The difference in the real structure of films from the precursor molecule is caused by the detected absorption peaks at the frequencies of 2130, 2410, and 3606 cm^{-1}. Links responsible for these absorption bands in PPMS are missing; the deciphering with the help of the tables shows that the absorption peaks of 2130 and 2410 cm^{-1} correspond to the link Si-H, and the band of 3606 cm^{-1} corresponds to the band of Si-OH. This outcome suggests that as a result of plasma-chemical reactions PPMS molecule does not fall apart into individual atoms, as it was considered in early models [6-7]; it falls apart into larger fragments which retain their identity to PPMS molecule. Another result of the reactions occurring in the plasma is the emergence of a series of chemical PPMS bonds in the film.

Mechanical Properties of SC DLF

We studied the mechanical properties of the layers of SC DLF. Those studies concerned several parameters related to mechanical properties. This is Young's modulus (E), residual elastic of tension (σ) and adhesion of the substrate layers. For the evaluation of microhardness (H) of thin layers, we used the empirical ratio H = E / 10 [GPa].

To determine Young's modulus we used the method of speed measuring and dispersion velocity of the surface acoustic waves. This method is implemented in the form of installation designed and manufactured at the Fraunhofer Institute in Germany [6]. The results are shown in Figure 8.

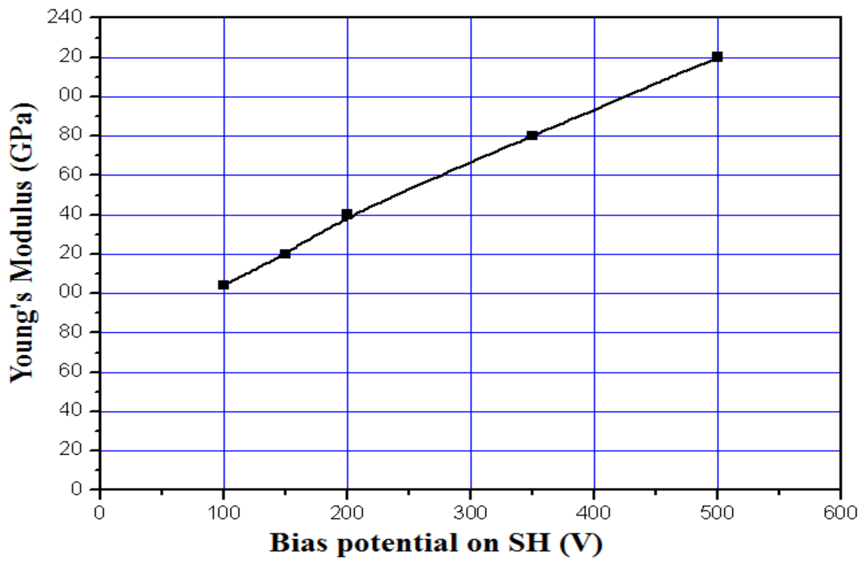

Figure 8. Dependence of the value of Young's MODULUS of SC DLF on the value of the constant component of potential on SH.

The obtained results showed that elastic modulus and hardness of SC DLF increase with the increasing bias potential at the substrate holder, i.e., with increasing energy of positively charged plasma component.

Residual elastic stresses in the films were determined by measuring the radius of curvature of the film-substrate system. As is known, when there are other elastic stresses in the film, the whole structure film-substrate is bent. By measuring the radius of the structure curvature, the thickness of the film and substrate, knowing elastic substrate modules we can easily calculate elastic tension in the film with the help of Stoney formula [7]. Experimental studies showed that residual elastic stresses depend on the constant potential component on SH to a greater extent. Figure 9 shows the dependence of this type obtained in this work.

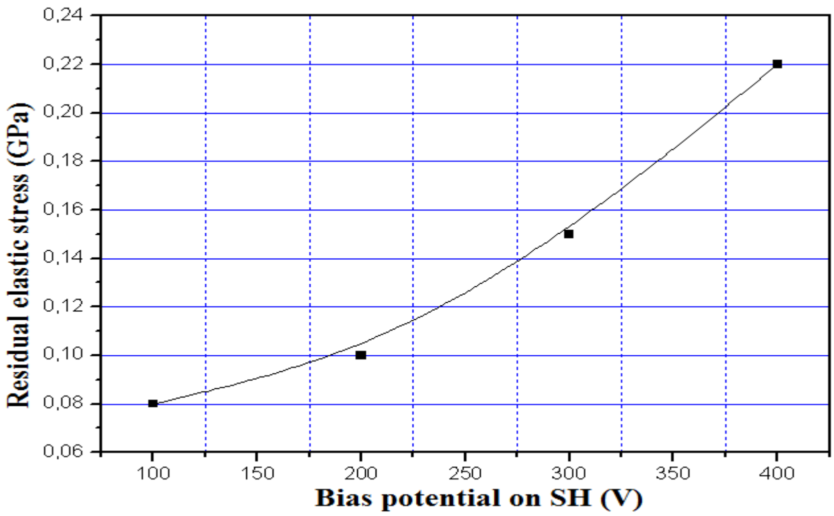

Figure 9. Dependence of the value of residual elastic stresses in the film SC DLF on the potential of SH.

As seen in Figure 9, the stress level increases with the rise in potential on the substrate. This is due to the increase in energy of the positively charged plasma component deposited on the substrate. Additional energy condenses the film by implanting effect, adding elastic stresses. By varying this parameter, we can reduce the stress from 0.22 GPa to 0.08 GPa. As is known, film adhesion to the substrate decreases with the increase of residual elastic tension therein. Therefore, the decrease in potential on SH will lead to increased adhesion.

Thus, the reduction in potential on SH will ensure that we obtain more durable dielectric films and, respectively, better sensor gauges.

CONCLUSION

The conducted study proved that the use of SC DLF as insulating dielectric films in order to create strain gauges based on samarium monosulphide, as well as protective coatings for sensors is very promising.

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