Control and optimization of technological processes for forming nanoscale films for sensitive sensor elements

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Abstract. Modern metal film sensors are used in products and systems of rocket, space and aviation equipment, in extreme operating conditions. In order for the sensors to function correctly in such conditions, they must have a high temporary stability over the entire life of operation and storage. In addition, metal film sensors are often operated at elevated temperatures and under the influence of large-scale vibration and shock. Therefore, they must have high mechanical static and dynamic strength. Increased requirements for the reliability, accuracy and strength of metal film sensors force designers and technologists to develop new design and technological solutions that ensure the regular operation of sensors in products and systems of rocket, space and flight equipment.

1. Introduction

The basis for the temporary stability and reliability of physical quantity sensors are correctly selected materials, structural and functional materials, as well as proven standard and special technologies. This is especially true for metal film sensors of physical quantities (SPQ), which use nanoscale resistive, conductive and insulating films formed on metal elastic elements (EE) (figure 1). In essence, such heterogeneous films and materials form a heterogeneous structure, the properties of which depend both on the electrophysical characteristics (EPC) of individual films and on their totality [1, 2]. With correctly selected materials, technological methods and modes of film formation, it is possible to obtain acceptable technical characteristics (TC) of the SPQ. To achieve high reliability and temporary stability, it is necessary to carry out special technological operations for stabilization of EPC, in which the microstructure of films is stabilized by controlled thermal, mechanical and electrical influences.

When analyzing the physical structure of the SSE metal film, it is possible to identify those structural elements and systems that are directly involved in the conversion of the measured non-electrical value, for example, pressure into electrical (figure 2). Since these elements are involved in the information and energy measurement process, they determine the stability of the entire SPQ [3].
2. Research methods

It should be noted that when creating sensors for modern launch vehicles and new-generation aircraft that are operated in extreme conditions, multi-stage measures are provided to ensure the stability of the main metrological parameters. A conditionally accepted strategy for ensuring reliability and stability can be represented by the structural graph shown in figure 3 [4]. In figure 3, notation is the following: 1 – evaluation of the influence of external destabilizing factors (EDF) on the reliability and stability of SE and SPQ, 2 – stabilization and adaptation of SE and SPQ, 3 – manageability of EPC of functional materials for SSE and SPQ, 4 – development of physical-mathematical models (PMM), 5 – forecast of changes in the metrological characteristics of the SPQ, 6 – appointment criteria for stability, 7 – synthesis of stabilizing feedback.

It should be noted that the presented concept of development includes feedback links that allow monitoring and optimization of the developed design and technological solutions at all stages of the SPQ creation with increased stability and reliability. These feedbacks are similar to the feedbacks recommended by international quality standards, in which they are present throughout the life cycle of any product.

The need to complicate the traditional manufacturing technology of strain-resistor SPQ is dictated by the requirements to ensure high reliability and stability of sensors under extreme operating conditions (low and high temperatures, high levels of vibration and shock, the presence of aggressive media, etc.) [5, 6].
Figure 3. Graph of the process of creating highly stable SPQ designed for extreme operating conditions.

The graph contains two main technological block-modules: "Development and research of individual technological processes for the formation of thin-film heterostructure..." and "Development and research of special methods for tuning, diagnostics, technological training, stabilization and accelerated testing....". These technological modules show the complexity and laboriousness of ensuring guaranteed stability and metrological reliability of SPQ intended for use in strategic industries. Performing special stabilization, diagnostics, and fitting operations significantly increases their cost. For general industrial SPQ, the above-mentioned procedures for stabilizing parameters are usually not available, so the cost of such sensors is an order of magnitude lower than for special ones. Using the example of strain-resistant metal film pressure sensors, we will consider in more detail the material science and technological component of the process of developing highly stable sensors that work under the influence of extreme EDF.

The following basic technological requirements for the materials of metal film SE should be immediately noted:
- technological compatibility (preservation of stoichiometry of films during their formation on EE, stability of phase composition during heat treatment, selectivity of etching)
- controllability of technological processes for forming a film heterostructure to optimize the EPC of the composition.
- ability to measure and control the main parameters of films (surface resistance, insulation resistance, thickness, continuity, etc.).

As a result of numerous scientific and applied research in the field of special materials science for measuring transducers, precision alloys based on iron and nickel with additives of titanium and aluminum were recommended for elastic elements [7]:
- alloy 36NHTYu has good plastic properties in cold and hot conditions, is subjected to stamping, and is welded by argon-arc welding. Retains guaranteed elastic properties up to +200°C.
- for higher operating temperatures (+300°C), precision alloys 42NHTYu and 44NHTYu are used.

A new cobalt-chromium-nickel non-magnetic corrosion-resistant alloy 40KHMN is recommended for EE SPQ. The alloy can work in contact with many aggressive media: solutions of salts, acids and alkalis, in sea water and in tropical climates [6]. EE made of this alloy can work up to a temperature of +400°C.

3. Dielectric layer (film)
The main requirements that the dielectric layer must meet are reduced to the following: unambiguously transmitting EE deformations to the strain gages; high adhesion to the EE material; minimal difference in thermal expansion coefficients (TEC) with the EE material; high and stable
values of the elastic modulus and insulation resistance; chemical inertness in relation to the EE materials and strain gages.

If some particular characteristics of the selected dielectric material do not meet the requirements due to their inconsistency, then certain technical solutions are used to eliminate the contradiction. For example, the inertness of the dielectric layer to the EE material and, at the same time, the presence of high adhesion to this material is resolved by forming a thin film of the active chromium metal, which provides adhesion to both the EE and the insulating material. As practice has shown, the most acceptable insulation materials are SiO-SiO₂, Al₂O₃ and Cr₂O₃ [7].

4. The materials of the strain gages

The selection of materials of strain gages (SG) are the most complex and ambiguous. This is due to the fact that the metrology and stability of the entire sensor depend most on the EPC of SG materials. In this case, the insulation and contact layers are inactive transmitters of information and energy of the measured process. The resistive layer is active, since it converts a mechanical value – deformation, into an electrical value - change in resistance [8]. Therefore, it has a number of special requirements for metrology, EPC, and so on. The main requirements are as follows: high and stable strain sensitivity; high EPC (mechanical strength, resistivity, reproducibility, adhesion to the insulation and contact layers, a wide range of operating temperatures), etc.

There are a number of resistive materials, both traditional, widely used, and new, designed specifically for high-temperature, highly stable SPQ. The first of them is nichrome H20N80, which is technologically advanced for sputtering and has a satisfactory time stability, but has a narrow temperature range (-80...+100)°C. For more extreme conditions, the alloy H20N75Yu is used, which is operable at temperatures from -180 to +150 °C, while providing high temporary stability.

The second group of load-bearing materials includes a silicic alloy of the P65HS type, which is technologically the most complex in terms of labor intensity, yield of usable materials, and so on. On the other hand, this alloy is promising for creating high-precision, stable SPQ [9].

In addition to the specified strain-resistive materials, the possibility of creating a resistive layer of thin-film SG from two strain-resistive materials having opposite signs of temperature coefficient (TC) is investigated. Such materials were selected alloy H20N75Yu, which has a positive TC α₁ = +(1...3)·10⁻⁴ 1/°C and molybdenum – rhenium alloy MR-47VP with negative TC α₂ = minus 7.0·10⁻⁴ 1/°C. These film compositions were formed by magnetron sputtering of films from a composite target containing materials of sputtered films. In this case, two-layer, multi-layer and composite film structures formed on a single SE were obtained.

5. Contact group materials

The main requirements for contact materials are: minimum ohmic resistance between the materials of the strain gage and the contact; no oxidation of the upper and lower surfaces of the contact film; high adhesion to the strain gage, no appearance of intermetallic phases during sensor operation, high film continuity and no pores, performance in a wide temperature range, low contact resistance, the possibility of obtaining a strong welded (electro-contact, ultrasonic) or soldered connection with external terminals; inertness to moisture and gases. In this regard, only pure gold meets most of these requirements to the greatest extent. Only it is inert to the material of the strain gage and has a low adhesion to it. To increase adhesion, the formation of a sublayer of the active metal chromium or vanadium (approximately 1.5 nm) is used, on the surface of which the main contact material - gold is sprayed. The transition layer, while observing the deposition modes, practically does not affect the transition resistance of the contact and is not an element of instability.

To illustrate the application of the materials considered, figure 4 shows an example of a beam type of fuel cell used in sensors for measuring the pressure of rocket fuel supplied by the pumping unit of the first stage of a “Proton M” – type launch vehicle.
6. Technological issues

As noted earlier, for the formation of film structures and CHE elements of high-stable SPQ, it is necessary to determine the basic technology and appropriate equipment, which should be able to control and regulate the process of film formation. Of all the known technologies for the formation of films and film compositions (thermal spraying, electron beam, gas-phase, magnetron, etc.), we selected magnetron sputtering. This choice is due to the availability of equipment, the possibility of upgrading old installations, including simple re-equipment of traditional vacuum spraying units of the UVN-type, by installing purchased or original magnetrons in the vacuum chamber. With the use of a magnetron installation, it is possible to spray both pure metals and alloys, and complex materials with the preservation of stoichiometry under sparing conditions. The performance of such installations is satisfactory and can be improved by simultaneous use of several magnetrons mounted in a vacuum chamber [10]. In addition, the use of several magnetrons makes it possible to form polyfilm compositions with new properties: a giant magnetoresistive effect, thermoactuators, etc.

In relation to magnetron spray installations and technologies, we will consider ways to control the characteristics of formed films.

As you know, the working body of any magnetron installation is a magnetron, the design of which is shown in figure 5, there is also a working target, the material of which is sprayed by the bombardment of electrons accelerated by the magnetic field of the magnetron.

![Diagram of the magnetron sprayer](image)

**Figure 5.** The design of the magnetron sprayer (a) and the type of erosion zone (b): 1-target; 2-radiator; 3-central magnet; 4-annular cooling channel; 5-peripheral magnet; 6-screen-anode; 7-magnetic conductor; 8-insulator; 9-current drive-water supply connection.
An important parameter of a magnetron sputter is the target material utilization factor. The annular zone of the target located between the poles of the magnetic system, in the area where the magnetic field lines are located above the surface of the target and parallel to its surface, is sprayed. The shape of the erosion zone of the spent target is shown in figure 5b. When the depth of the erosion zone is equal to the thickness of the target, the width of the zone \( L \) is 20 mm and the diameter of the middle line is 59 mm. With a target diameter of 130 mm and a thickness of 5 mm, the target material utilization rate is 6.9%.

The developed magnetron sprayer allows to set targets with a diameter of 120 mm. When installing a chromium target with a thickness of 10 mm, the measured magnetic induction on the surface of the target was 70 mT, when installing a nickel target with a thickness of 5 mm – 30 mT.

Two magnetron atomizers are mounted in the vacuum chamber of the UVN-71-P3 type vacuum spraying unit. The supply voltage of negative polarity is supplied to the cathode of magnetron atomizers through the water supply connection. Cooling water is supplied and drained to the sprayer via a PVC hose. Figure 6 shows a photo of an in-chamber device for vacuum spraying with two magnetron atomizers. In the near position, there is a magnetron sprayer with a nickel target, in the far position – with a chromium target. Erosion zones are visible on both targets.

A special feature of the magnetron is the method of attaching the target to the surface of the radiator, which is implemented by soldering with low-melting solder. When using a target made of non-solderable materials, it was applied by vacuum deposition or galvanic build-up of a layer of copper, which is then tinned with solder.

![Figure 6. Inside-chamber device of the modernized installation of a magnetron sprayer with two magnetrons.](image)

Due to the fact that the magnetron discharge is a source of a wide range of electromagnetic interference and false leads, it is therefore difficult to control the properties of the formed films by directly measuring their electrical or optical parameters. Therefore, the task is to study the performance characteristics of a magnetron sprayer in order to determine methods for controlling coating modes.

The experimental study of the characteristics of magnetron atomizers was carried out as follows: the vacuum chamber was pumped to a pressure of \( 2 \cdot 10^{-5} \) mm Hg, then the shutter of the vacuum chamber was switched to throttling mode. After that, an inert working gas argon was fed into the chamber at a pressure from \( 5.0 \cdot 10^{-4} \) mm Hg to \( 10^{-3} \) mm Hg. For a magnetron with a target made of chromium, the magnetron discharge is ignited at a pressure of \( 8 \cdot 10^{-4} \) mm Hg. At a pressure of \( 1 \cdot 10^{-3} \) mm Hg, the magnetron discharge enters a stable phase and burns steadily. For a magnetron with a nickel target, the magnetron discharge is ignited at a pressure of \( 1.5 \cdot 10^{-3} \) mm Hg, and at a pressure of \( 1.8 \cdot 10^{-3} \) mm Hg, the magnetron discharge burns steadily. Figure 7a shows the dependence of the maximum ion current of the magnetron on the pressure in the chamber and the deposition rates on the power for the two materials.
Figure 7. Controlled characteristics of the magnetron: a - from the pressure in the chamber; b - the deposition rate for two materials Cr and Ni from the power; c – VAC of a magnetron atomizer with a nickel target 1-at a pressure of $8 \times 10^{-3}$ mm Hg, 2 - at a pressure of $3 \times 10^{-3}$ mm Hg.

At continuously maintained operating pressures, the deposition rate depends almost linearly on the applied power and is determined by the sprayed material, the amount of induction on the target surface, and the working gas pressure. Figure 7b shows the dependence of the deposition rate of chromium and nickel on the power supplied to the magnetron at a distance of 50 mm from the target surface to the substrate.

The most complete information about a magnetron discharge is provided by its volt-ampere characteristics (VAC). VAC is a complex function of the geometric characteristics of the magnetron sputter, working gas pressure, and induction on the target surface. As can be seen from the VAC of a magnetron atomizer with a nickel target shown in Figure 7c, they represent a nonlinear dependence in the field of operating modes. When the pressure of the working gas in the chamber increases, the volt-ampere characteristics shift to the region of high currents, which is explained by the presence of more ionized particles. This creates difficulties in automating the control of spray modes and requires the use of pressure maintenance systems in the working chamber.

7. Conclusions

Studies in the field of sensitive elements of metal-film pressure sensors made it possible to choose materials and technologies for the formation of heterogeneous film structures. As the basic technology, the magnetron technology was chosen, which allows controlling the deposition of films from various materials.

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