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# SMART POLYMER COATINGS FOR THE DEFECTS CONTROL IN METALS

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**Abstract**

This letter demonstrates the possibility of using thin polymer films for non-destructive diagnostics of metal products and structures. The method is shown to be quite simple and reliable. To implement the method, the development of complex electronic solutions is not required. The resulting sensors based on thin polymer films can operate in both constant and alternating electric field modes. The use of this method will help prevent man-made accidents. This method can make a significant contribution to the development of industrial safety.

## Introduction

When planning the development of society and territories, an important aspect is the prevention of incidents related to technologies for the construction of buildings and structures, bridges and overpasses, oil and gas pipelines, electrical networks, and so on. This issue is relevant, for example, in the field of ensuring safety during threatening natural phenomena, such as earthquakes, floods, hurricanes [1]. These problems can be approached from different angles. In particular, when developing buildings and structures, the tensile strength of various materials is taken into account and the development of new materials with improved characteristics, be it heat resistance, mechanical strength, or impact resistance, is actively underway [2,3].

However, there is also another approach. This refers to the development of warning and alarm systems. One of the promising areas of research in this area is the development of various sensors that make it possible to identify the danger of destruction of man-made elements. Such methods include non-destructive testing and diagnostic methods [4].

Indeed, using the example of metal products and structures, one can operate with such parameters as fatigue strength, tensile and tensile strength, and corrosion resistance. Despite the excess indicators included in engineering calculations, which are several or more times higher than the nominal ones, unforeseen disasters, destruction, damage that cause colossal harm to the economy, ecology, life and health of people happen with alarming frequency [5].

In this regard, the development of simple, economically feasible methods and technologies for non-destructive testing of the condition of infrastructure elements is becoming a very relevant and sought-after branch of science and technology. The solutions proposed today, despite their high efficiency, are expensive and difficult to implement and implement, not to mention ensuring the individual safety of citizens and individual elements of buildings and structures [6, 7].

The solution can be found in the field of technologies that do not require large amounts of energy, the development of a separate information network and are capable of rapid scaling and prototyping. One of these areas may be the development of non-destructive testing systems based on the achievements of organic printed electronics and additive technologies. Indeed, polymers and composites based on them are accessible and cheap materials with great potential for use in electronics. There are already a large number of electronics industries where polymer materials are used commercially successfully. These are organic light-emitting diodes, solar energy, flexible electronics and renewable energy sources [8,9,10].

In this paper, we propose a simple, commercially available, but at the same time quite knowledge-intensive method for creating and developing devices based on organic materials for non-destructive testing of metal elements of buildings, structures, pipelines and other things.

As is known, most unmodified polymer materials are dielectrics in nature [11, 12]. Based on the band theory of solids, it is known that dielectrics with an ideal structure should not conduct electric current. In local areas of a material, this can be observed, and polymers are no exception, that is, in polymer films, there are always structural disturbances, defects and impurities that cause the generation of mobile charge carriers. In those polymers that are classified as dielectrics in terms of electrical conductivity, the role of free charge carriers can be assigned to ions (or mole ions), which arise as a result of the dissociation of molecules, or, for example, during the injection of electrons from the external environment (metal contacts).

To describe the movement of charge carriers, two mechanisms can be distinguished:

1) The carrier motion is considered as the propagation of a delocalized plane wave in the conduction band.

2) The charge carrier is highly localized and moves by hopping or jumping, accompanied by scattering of the charge carriers. From the point of view of practical use, it is very important to recognize the type of conductivity and the mechanism of transport of charge carriers in polymers.

Let us consider a dielectric whose unit volume contains n charge carriers with charge q. Under the influence of an electric field of intensity E, the ordered movement of free charges begins in the dielectric, which creates an electric conduction current in the material. When studying the intrinsic electrical conductivity of dielectrics, one should also consider the issue of the dependence of the current on the time of exposure to the electric field. When a constant voltage is applied, the value of the current that passes through the polymer sample, called the charging current, will monotonically decrease, asymptotically approaching some equilibrium value. When the voltage supply is stopped and the electrodes are short-circuited, a discharge current appears, flowing in the direction opposite to the charging current, which also decreases over time.

Polymers contain free and loosely bound electrons. The reason for their occurrence may be thermal ionization of macromolecules and impurity molecules, ionization of these molecules under the influence of light and radiation, injection of electrons into the polymer, as well as the introduction of additives. It should be mentioned that electronic conductivity can be observed in polymers, which are dielectric in nature, although this type of conductivity is primarily characteristic of polymer semiconductors and electrically conductive materials.

One evidence of the presence of electron-hole conductivity in some polymers is the Hall effect [13]. However, for a large number of polymers this conductivity cannot be detected due to the low mobility of charge carriers.

When considering the mechanisms of movement of electrons and holes in polymers, the following models can be applied: non-activation band, tunnel and activation hopping. Also in some cases it is necessary to take into account the emission of electrons from the electrodes or, in other words, the Schottky effect and conductivity, which is caused by impurities and defects [14].

At the interface between two media (metal/dielectric), various phenomena occur that affect the electrical conductivity of the dielectric. Below are several main types of contacts:

“Neutral contact” (metal/dielectric contact), which provides conditions for stationary current flow due to the carriers that are present in the volume of the sample, is called.

A “blocking contact” that prevents charge carriers from passing through the metal/dielectric interface. If, under conditions of an applied electric field, charge carriers do not come from the side of the electrode, then in the adjacent region of the dielectric, layers depleted of charge carriers with increased resistivity are formed, which leads to a decrease in the current passing through the dielectric over time. This process is called molding or the formation of barrier layers.

"Injection contact", which is a source of excess carriers. The role of such a contact can be an electrolyte solution (salt or acid) that is in contact with a polymer dielectric. Such systems are used to study conductivity in polymers caused by ions that are injected from solution under the influence of voltage applied to the electrodes. Electronic injection currents in dielectrics can occur even in cases where the dielectric itself is almost ideal and the concentration of its own charge carriers is close to zero. To describe the processes of electron injection, the model proposed by Schottky is used [14].

One of the interesting electronic properties of organic materials is the ability to change the state from dielectric or semiconductor to a state with high conductivity [15]. This phenomenon is called the conductivity switching effect or simply the switching effect.

The switching effect manifests itself as follows. In the normal state, when an electric field is applied to chalcogenide glass, the current through the sample increases linearly. The current in this state is small, that is, the resistance of the sample is high (according to Ohm’s law for the circuit section I=U/R). When a certain voltage value Uon, called threshold or critical, is reached, a sharp increase in the amount of electric current through the sample is observed (Fig. 1). This state is called "on".

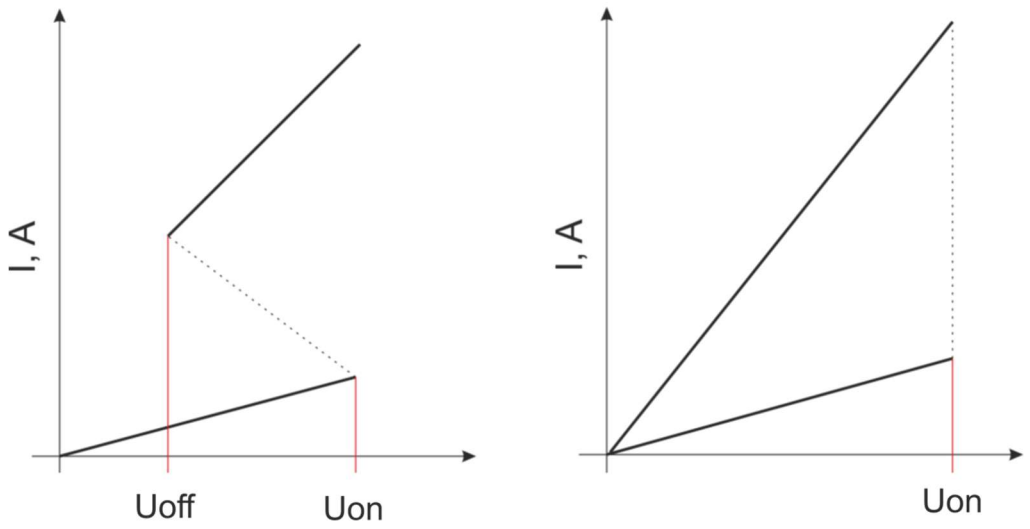



**Figure 1.** Illustration of switching effects: a) switching effect and b) memory effect

When moving towards a decrease in voltage, the switched state will be maintained until a certain voltage value Uoff, after which the sample returns to its original "off" state. With this effect, the dependence of current on voltage, that is, the current-voltage characteristic (CVC) has an S-shaped form (Fig. 1a). It is noteworthy that the change in resistance can reach several orders of magnitude, that is, a transition of the material from a dielectric or semiconductor to a highly conductive state is observed.

A second type of switching, called the memory effect, can also be observed (Fig. 1b). The difference between this effect and switching is that the state of high conductivity is maintained even after turning off the voltage on the sample and when the voltage is subsequently applied, the sample is in a state of high conductivity [15].

A change in the electrical conductivity of an organic material can be triggered by minor external factors. One of these factors may be a change in electronic parameters in the metal contact. This is exactly what the subject of research was.

**Materials and methods**

The problem of monitoring in real time the condition of metal structural elements is relevant. Different groups of researchers propose different ways to organize this process [6,7]. Despite the obvious simplicity of the proposed solutions, they have a number of significant disadvantages. They consist in the fact that a source of optical radiation, its receiver and a signal analyzer are required. All this also requires an additional source of electrical energy. Our approach will minimize the costs of these components and allow us to implement a device with non-volatile principles. Another disadvantage can be considered the limited miniaturization and complexity of providing a matrix arrangement of tens, thousands, or even an unlimited number of identical sensor elements. Thus, the necessity and possibility of simultaneously carrying out local, in areas less than 1 $\mu m^2$, and distributed measurements of the state of metals is revealed.

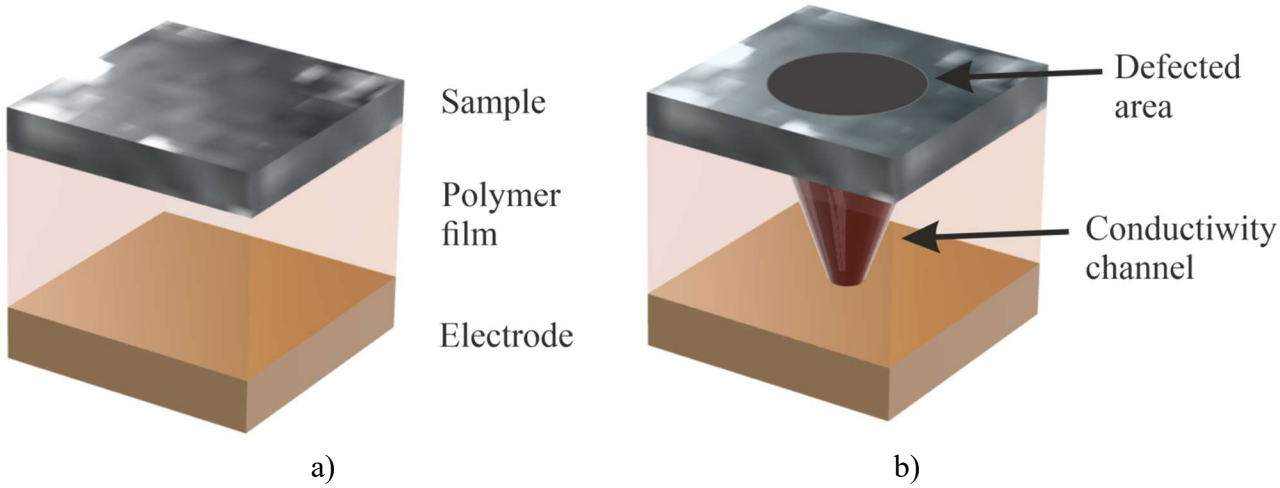



**Figure 2.** Structure of the samples: a) before switching and b) after switching

Figure 2 shows the schematic structure of the samples studied in this work. As a sample, a steel pipe segment was cut out, which was subjected to compressive deformation during the measurements. A polymer film was applied to the surface of the sample by casting. A copper electrode was pressed on top of the polymer film.

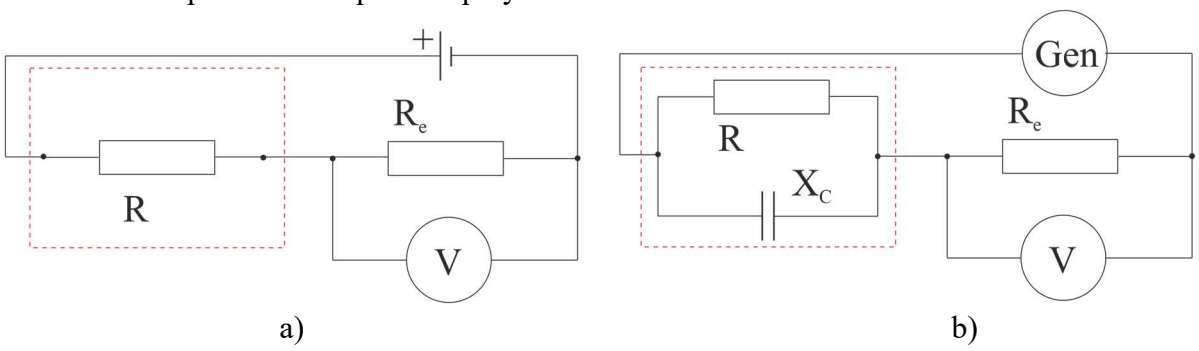



**Figure 3**. Scheme of measurements: a) in direct current mode b) in alternating field mode

The final form was a metal/polymer/metal structure, with varying thickness of the polymer film, shape, size and overlap area of the electrodes. The thickness of the polymer film from 50 to 3000 nm was set by the concentration of the polymer solution and ranged from 0.5 to 20 wt. %. Since it was assumed that the polymer film could transition to a highly conductive state (Fig. 2b), direct measurement of the resistance of the polymer film was not permissible. Measurements were made in both constant and alternating electric fields according to the circuits shown in Figure 3. A reference resistance of Re = 100 kΩ protected the circuit from short circuits, and the currents in the circuit were recalculated based on the voltage drop measured across it. Let us consider as an equivalent circuit the simplest RC circuit, the operating principle of which is based on the dependence of the capacitor reactance on the signal frequency.

If a resistor with resistance R and a capacitor with capacitance C are connected in series to a source of alternating sinusoidal voltage U with frequency f, the voltage drop across each element can be calculated based on the division coefficient with impedance Z.
Impedance is the complex (total) resistance of a circuit for a harmonic signal:

$$Z^2 = R^2 + X^2; Z = \sqrt{(R^2 + X^2)}$$

where X is the reactance.
Then the voltage UR at the resistor terminals will be:

$$U_R = U\frac{R}{Z} = U\frac{R}{\sqrt{(R^2 + X_C^2)}}$$

where $X_C$ is the reactance of the capacitor, equal to $\frac{1}{2\pi f}$
If R = $X_C$ at frequency f, the expression will be simplified by abbreviating R and take the form:

$$U_R = U_c = \frac{U}{\sqrt{2}} \approx 0.7U$$

Consequently, at frequency f, the equality of the active and reactive resistances of the RC chain will provide the same amplitude of the alternating sinusoidal voltage on each of the elements, √2 times less than the input voltage, which is approximately 0.7 of its value. In this case, the frequency f will be determined based on the resistance R and capacitance C with the expression:

$$f = \frac{1}{2\pi RC} = \frac{1}{2\pi\tau}$$

where τ is the time constant of the RC circuit equal to the product RC.
The equilibrium concentration of charge carriers is determined by the condition of equality of the equations of the dependence of current on voltage for the linear Ohm's law:

$$j = en_0\mu\frac{U}{L}$$

and a quadratic plot. In the simple case when there are no traps and the space charge limited current is carried by carriers of the same sign, the current density is described by Child's law:

$$j = \frac{9}{8}\varepsilon\varepsilon_0\mu\frac{U^2}{L^3}$$

where j – current density, L – distance between electrodes, U =Un – the voltage corresponding to the transition point of linear approximations of these sections in logarithmic coordinates, n0 – equilibrium concentration of charge carriers, μ – effective mobility of charge carriers, e – elementary electric charge, ε0 – vacuum permittivity, dielectric constant of the material ε at room temperature, it is assumed to be equal to [16]. Hence, we get the expression for concentration:

$$jn_0 = \frac{9}{8}\frac{\varepsilon\varepsilon_0 U_n}{eL^2},$$

Carrier mobility was calculated using the following equations:

$$\mu = \frac{8}{9}\frac{jL^3}{\varepsilon\varepsilon_0 U_n^2}.$$

It should be noted that changes in the resistance, mobility and concentration of charge carriers correlate well with changes in the potential barrier. At the same time, the graphs are not monotonous; fluctuations are observed on each of the dependencies.

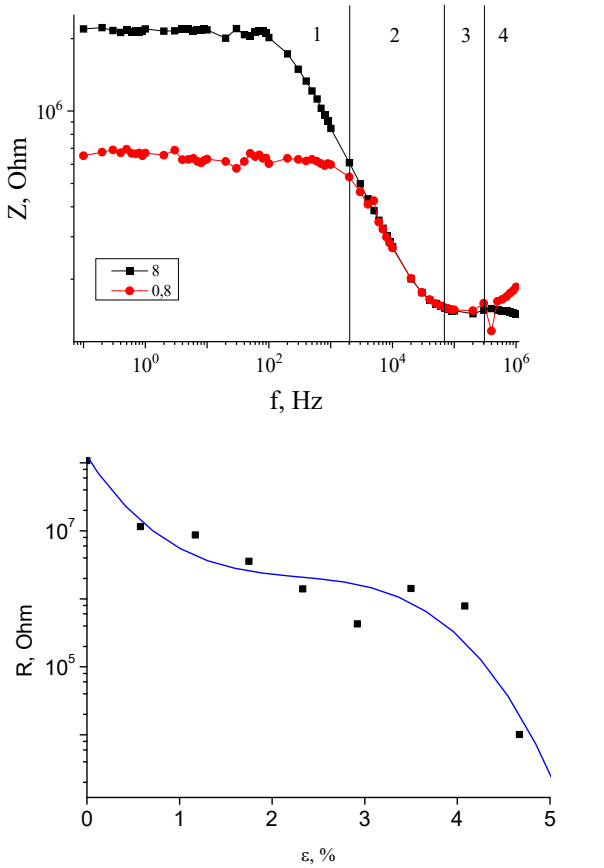
The height of the potential barrier at the metal/polymer contact $\varphi_{B0}$ can be estimated using the well-known formula for the Schottky barrier:

$$\varphi_{b0} = \frac{kT^3}{q_n} \ln\left(\frac{AA^*T^2}{I_S}\right)$$

where T is temperature, k is Boltzmann's constant, q is electron charge, A is contact area, A* is Richardson's constant, $I_S$ is saturation current. The saturation current or current at zero voltage can be determined at the point of intersection of the line of approximation of the saturation region of the current-voltage characteristic, constructed in semi-logarithmic coordinates (ln(I) ÷ V) with the current axis.

**Results and discussions**

It should be noted that the switching circuit of the structure containing the polymer film corresponded to the operating circuit of the high-frequency RC filter. This means that the experimental dependences of the output voltage on frequency should have reflected the features of such a connection.




a) b)

**Figure 4.** Dependence of the impedance of the metal/polymer/metal structure on the frequency of the alternating field at fixed voltage values: a) from 0.1 Hz to 1 MHz, to 1 kHz and the dependence of the resistance on the amount of deformation of the metal sample b).

Since the electrophysical phenomena observed in polymer films, in particular, the effect of electronic switching from a dielectric state to a state with high conductivity, excluded the possibility of measuring frequency characteristics using a circuit with a parallel connection of a reference resistance, the dependence of the impedance on frequency was plotted based on calculations carried out using experimentally measured output characteristics of the sequential RC chain structure.

Figure 4a shows the dependence of the impedance of the metal/polymer/metal structure on the frequency of the alternating field. The dependences are plotted for two voltage values: 0.8 and 8 V. The dependences for both voltage values can be conditionally divided into four sections.

In section 1, the impedance weakly depends on the frequency of the alternating signal; a monotonic decrease in resistance with small fluctuations is observed. It should be noted that the resistance of the polymer film at a low applied voltage (0.8 V) is approximately three times less than the resistance at a high voltage (8 V). In section 2 there is a sharp decrease in impedance with increasing frequency. In this area, the resistances of the polymer film at different voltages are close in value. In section 3, the impedance weakly depends on the frequency of the alternating voltage. The impedance values at different voltages and frequencies are close and equal to approximately 1.5 MΩ. In section 4, the impedance at a voltage of 0.8 V increases sharply with increasing frequency. The impedance at a voltage of 8 V begins to decrease monotonically as the frequency increases.

From the above data it is clear that the resistance of the polymer film decreases insignificantly and monotonically with increasing frequency at a voltage of 0.8 V. At the same time, the resistance of the film at a higher voltage, 8 V, decreases significantly and exponentially with increasing frequency. This behavior can be well explained by injection processes in polymer films.

Figure 4b shows the dependence of the active resistance of the polymer film on the amount of deformation of the metal sample. It is clearly seen that the resistance of the polymer film decreases non-monotonically with increasing deformation. At the same time, the resistance decreases by as much as 4 orders of magnitude.

The results obtained can probably be explained by the changes occurring in the metal sample during elastic deformation. Namely, it is well known that with different types of deformation, the electron work function (EWF) of a metal changes [17,18,19]. Indeed, let us turn to measurements made in a constant electric field (Figure 5 a). It is clearly seen that at different voltages across the sample, different mechanisms of current flow are realized. From a straight section to a quadratic section [16]. Since the currents are small, they will be extremely sensitive to changes in contact phenomena. And in the case of this experiment, the EWF was subject to change, which can reach a value of 0.3 eV [17,18]. Apparently, such a change can cause a transition from the linear law of electric current flow to quadratic, which is observed in the frequency characteristics.

To test the assumption that the current depends on the EWF of the metal, the dependences of the resistance of the polymer film on the type of metal (on the EWF of the metal) were measured. The results are presented in Figure 5b. It can be seen that the threshold pressure at which the switching effect is observed [15] depends on the type of metal, and, consequently, its electronic characteristics.

Thus, the results obtained suggest the possibility of non-destructive testing of metal products using thin polymer films. A change in resistance by 4 or more orders of magnitude opens up the possibility of placing low demands on electronics, which can be used in the development of sensors based on the principles described.

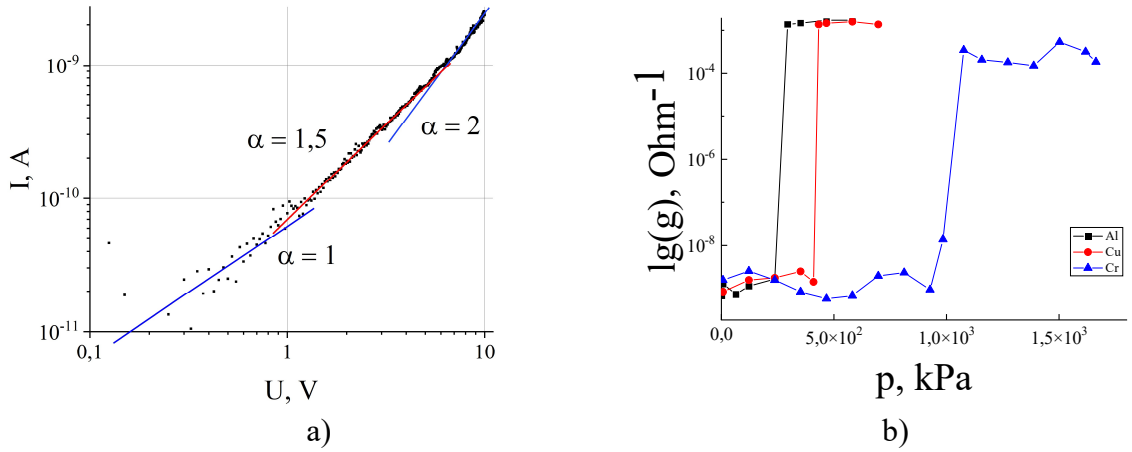



**Figure 5.** Current-voltage characteristic of the structure a) and the dependence of the threshold pressure on the type of metal b).

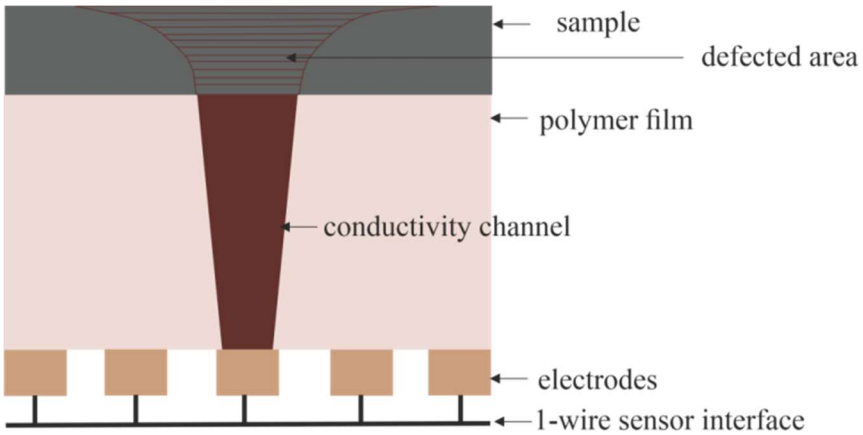



**Figure 6.** Sensor array placement and connection using 1-wire protocol technology

One of the possible sensor connection schemes is shown in Figure 6. The scheme uses 1-wire protocol technology [20]. That is, the system can be configured this way. So that when a certain level of deformation in the metal is reached, the polymer film locally switches to a state with high conductivity. And the coordinates are easy to detect using the proposed protocol.

**Conclusions**

Along with active research into semiconductor and metal-like organic materials, such as electrically conductive polymers, anomalous charge transfer effects in thin layers of metal oxide films, as well as organic dielectrics, are of interest. In thin films of various polymers, such as polypropylene, polyimide and others, not exceeding tens of microns, phenomena associated with a phase transition from a dielectric to a highly conductive state are observed.

It was discovered that such a transition can be initiated by external physical influences: mechanical pressure, temperature, magnetic and electric fields, and others. Despite the fact that the mechanisms of conductivity of polymer films have been studied quite well, insufficient attention has been paid to the state with high conductivity. In particular, the relationship between the chemical structure of the polymer, the supramolecular organization and the electrical properties of

the films remains unclear; influence of barrier effects at metal/polymer interfaces on charge transfer processes; characteristics of a highly conductive state. In addition, recording changes in the EWF of a metal due to phase transitions, as well as the occurrence of elastic and inelastic deformations, opens up a new direction for diagnosing the state of metals.

By using organic materials with high stretchability and the ability to control structure on a molecular scale, it is possible to simplify manufacturing processes and improve functionality. However, there is a relationship between good electronic performance, reflected in high carrier mobility and photoelectric efficiency, and desirable mechanical properties, such as high elasticity. Overcoming this correlation and combining high electronic performance and deformability in a single material will facilitate the development of reliable metal condition sensors. Therefore, it is important to understand which parameters influence these properties and determine whether optimizing them together is truly possible. To control the properties and parameters of organic films, molecular structure control is often used. This is set by parameters such as polymer chain rigidity, macromolecular length and branching of side chains, as well as formation conditions, such as the rate of solvent evaporation and annealing, molecular orientation.

The change in the electron work function of a metal is a fact that testifies to various processes in metals. These may be chemical changes, in particular the oxidation of the surface layers. At the same time, changes related to the structure are of practical interest – the origin of cracks, metal fatigue, elastic and inelastic deformations. Tracking changes in the EWF can be a method of determining all these transformations.

This paper presents the results of a study of a new method of non-destructive testing of metals. At the moment, only the case of deformation is considered. However, the results presented in this paper suggest that this method can also be used to track other changes: elastic deformations, mechanical stresses, phase transitions. All these processes are accompanied by a change in the EWF. These processes can be distinguished by the magnitude and direction of change in the EWF. The method consists in the application of electroactive wide-band non-conjugated polymers to determine the energy parameters of the metal/polymer interface. A change in the mechanical stress, microhardness, and EWF of the metal during deformation leads to a change in the height of the potential barrier at the metal/polymer interface. This change can be determined indirectly, by measurements. Since wide-band dielectrics are used as polymers, the technique can be reduced to determining the resistance or impedance of the structure, which makes the introduction of this method into the metallurgical industry promising.

An electroactive polymer was used in the work. Probably, by changing the chemical composition of the polymer, as well as its thickness, it is possible to achieve selectivity to certain types of structural changes in metals.

Thus, knowing the approach of high mechanical stresses in the metal, it is possible to prevent any man-made accident in production, in buildings and structures, and even in everyday life. We have shown that the use of smart polymers can be very promising in non-destructive testing of metal products.

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