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STUDY OF A HIGH-SENSITIVITY PIEZORESISTIVE SENSOR UNIT BASED ON GRAPHENE STRUCTURES FOR THE DETECTION OF EARLY-STAGE FAILURE OF SMALL-DIAMETER METAL TUBES

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A high-sensitivity graphene-based piezoresistive sensor for the early detection of microdefects in small-diameter metal tubes has been investigated. An experimental correlation between defect depth in the range of 10...100 μm and changes in the electrical resistance of the graphene layer has been established. The feasibility of differential detection of the early stages of local damage based on the piezoresistive response of the sensor has been demonstrated.

The energy sector and related industries are experiencing a growing need for highly sensitive non-destructive testing methods for compact cylindrical tubular elements made of electrically conductive materials, aimed at the early detection of localized degradation sites. Conventional methods, including ultrasonic and eddy-current testing, provide effective detection of a range of volumetric and well-developed surface defects; however, their diagnostic sensitivity to the initial stages of damage may be substantially reduced under conditions of small wall thickness, complex curved geometry, and submillimeter characteristic defect dimensions [1–3]. A particular challenge is the detection of early-stage degradation characterized by localized surface inhomogeneities and microcracks with characteristic dimensions on the order of 10...100 μm. At this stage, structural changes do not yet form a pronounced macrodefect but may serve as initial sites for subsequent failure and significantly affect the remaining service life of tubular elements. Therefore, the development of non-destructive testing methods capable of highly sensitive detection and spatial localization of microscale surface damage at the early stages of its formation represents a relevant scientific and engineering challenge.

The study proposes and experimentally substantiates a high-sensitivity method for the detection and diagnosis of microscale defects (10...100 μm). The primary sensitivity mechanism is the modulation of tunneling conductivity in mesoscopic contacts between graphene flakes. When a microdefect (10...100 μm) occurs in the adhesion contact zone of the "steel–dielectric–graphene" structure, a local gradient of mechanical stresses is generated, resulting in an exponential change in the contact resistance of the sensor. For the inspection of a steel tube containing defects with dimensions of 10...100 μm, the graphene layer is positioned on top, between the polydimethylsiloxane substrate and the pressure roller; thus, it does not come into direct contact with the metal. Unlike conventional non-destructive testing methods focused on macrodefects, the developed approach enables the detection of early stages of material degradation associated with the initiation of microcracks and localized damage in cylindrical metal tubes. Numerical modeling was performed to determine the region of maximum sensitivity, which corresponds to frequencies up to 1 kHz. Within this frequency range, an optimal compromise is achieved between probing depth and the influence of high-frequency currents.

A promising approach for detecting microdefects preceding the development of degradation stages is the use of the piezoresistive method. In piezoresistive sensors, a direct correlation exists between the magnitude of deformation and the change in electrical resistance. This is a modern approach that combines nanotechnology with non-destructive testing. The underlying concept is that graphene is extremely sensitive to strain [4].

The aim of this study is to investigate the characteristics of the piezoresistive response of graphene films to defects with depths of 10...100 μm in cylindrical metal tubes containing artificially introduced microdefects corresponding to the early stages of damage.

The present study is aimed at establishing an analytical correlation between the geometric parameters of microscale surface defects (10...100 μm) on cylindrical bodies of revolution and the dynamic response of the complex impedance of a sensitive graphene layer operating in the percolative charge-transport regime. The primary objective of the study is to move from qualitative detection (indication of the presence of a topological inhomogeneity) to precise quantitative analysis and non-destructive mapping of defect geometry.

The presence of defects such as cracks and cavities results in significant changes in the resistive response, including the appearance of relaxation maxima, making this method promising for early-stage defect diagnosis.

Microscale surface defects were modeled by precisely introducing localized geometric damage into the load-bearing structure. Within the framework of equivalent electrodynamic circuits, the defects are interpreted as heterogeneous inclusions with resistive-capacitive impedance, which induce spatial distortion of the local electric field and redistribution of current density [5, 6, 12].

From the initial stages of the study, model verification was performed exclusively on cylindrical steel tubes. This methodological approach made it possible to isolate and accurately differentiate the purely resistive and capacitive contributions of the heterostructure to the overall measurement signal without the influence of extraneous deformation-induced noise. To quantitatively evaluate the piezoresistive response, a series of four reference specimens was fabricated: seamless steel tubes with an outer diameter of 10 mm and a length of 150 mm. Three specimens contained artificially introduced defects (scratches) with fixed depths: h_1 = 10 μm, h_2 = 50 μm, and h_3 = 100 μm, respectively. The fourth specimen retained a nominally smooth surface and was used as a reference baseline to provide high-contrast comparison of the signals. The physics governing the formation of the measurement trend on metallic bodies of revolution differs substantially from that of conventional two-dimensional defect detection on flat plates, owing to the radial anisotropy of the stress field and the constrained bending effect of the thin-film sensor.

The selection of layered graphene structures as the active element of the defect detection system is based on:

– mechanics of deformable solids: evaluation of local stress concentration in elastoplastic shells;

– quantum-mechanical tunneling transport: modulation of conductivity in two-dimensional systems;

– geometric conformability: minimization of the bending stiffness of the sensor on curved surfaces.

Initially, GFC 995 graphene paper was investigated as the most readily available material. In fact, GFC 995 consists of thermally expanded graphite compressed into a flexible sheet. It exhibits virtually unlimited fatigue resistance and does not fail when applied to a cylindrical substrate; instead, it continues to generate a signal, thereby enabling defect diagnosis. GFC 995 is more convenient for engineering experiments than laboratory-grade graphene. However, as demonstrated experimentally, owing to its thickness of 0.1 mm, the graphene paper possesses intrinsic stiffness; therefore, the signal produced by a 10 μm defect was very weak, whereas stable and reproducible responses were observed for defects with depths of 50...100 μm.

A more promising approach is the electrophysical analysis of a heterostructure consisting of a monolayer of graphene synthesized by the chemical vapor deposition (CVD) method and an elastic polydimethylsiloxane substrate, which was performed under stationary conditions. A controlled variation of the internal hydrostatic argon pressure in the range of 0...2.5 MPa was used as the deformation-inducing factor. Under such pressure, in the defect region where the wall is thinner, the steel begins to undergo a local outward deflection on the order of fractions of a micrometer. This volumetric cyclic loading method made it possible to determine the limiting sensitivity of the developed piezoresistive sensor unit to submicrometer displacements generated by local stress concentrators in the defect regions [7].

The proposed method for detecting the early stages of fatigue and corrosion failure of cylindrical shells is based on the use of the piezoresistive effect in two-dimensional carbon systems. The dominant microscopic mechanism determining the high strain sensitivity of the structure is the quantum modulation of tunneling conductivity in mesoscopic contacts between crystalline graphene flakes within a polymer matrix.

When the sensor is positioned above a topological defect (10…100 μm), a pronounced local gradient of the mechanical stress field develops in the three-layer adhesive contact zone of the "steel–dielectric gap–graphene" structure. The spatial redistribution of elastic forces causes an exponential change in the contact resistance and intralayer resistance of the graphene matrix. Unlike conventional metallic or semiconductor strain gauges, for which the sensitivity coefficient is limited primarily by the geometrical factor (changes in the conductor length and cross-sectional area), the developed heterostructure implements quantum-mechanical tunneling-percolative charge transport along a curved surface, providing a substantial increase in the differential sensitivity of the sensor [8–11].

The principles of integrating nanocarbon layered materials for structural health monitoring (SHM) of critical structures have been validated in international practice and are widely represented in modern approaches, such as Electrical Resistance Tomography (ERT) and piezoresistive SHM analysis. At the same time, precise detection of microscale local defects (10…100 μm) in thin-walled objects with a small radius (10 mm in diameter) remains insufficiently studied. The solution to this problem, addressed in the present study, was made possible through the mathematical closure of the correlations between the geometric metrics of the defect, the radial elastic deformation of the shell, and the complex impedance dynamics of the graphene layer operating under a strictly percolative conduction regime.

The following section presents the physical and technical parameters of the experiment using a sensor configured as a conventional planar resistor with linear (uniform) current flow. The current in the graphene monolayer flows strictly parallel to the tube axis, forming a uniform electric field. The transition to a planar-resistor geometry with end current-collecting buses extending across the entire 10 mm width determines the electrodynamics and mechanical stress distribution within the sensor.

The development of the measurement system and the determination of the experimental parameters were based on the need to ensure a uniform electrophysical field combined with a controlled distribution of mechanical stresses. Structurally, the active area of the developed piezoresistive sensor has fixed dimensions of 20×10 mm. When positioned on the test object, the sensor conforms around the 10 mm-diameter steel tube. This spatial configuration provides coverage of approximately 115° of the circumferential arc of the cylindrical surface across the sensor width (10 mm), while its 20 mm longitudinal axis is oriented strictly along the direction of the principal deformation vector, i.e., the longitudinal axis of the tube.

The interface of the structure consists of two parallel copper current-conducting buses, 30 mm in length (based on a highly dispersed silver conductive paste), applied to the opposite end faces of the active area. The formation of reliable electrical contacts on the soft polymer substrate precludes the use of soldering; instead, cold-

curing conductive silver epoxy compounds were employed.

For precise registration of the electrical response, a pair of electrodes from the E7-10 meter is permanently connected to each bus using a four-wire configuration with separate current and potential circuits. Taking into account the actual continuity and defectiveness of the transferred CVD graphene monolayer, the measured range of the sensor's baseline resistance, R_0, in the initial undeformed state is a stable 800…1400 Ω. The average operating point for a high-quality CVD monolayer is approximately 1100 Ω.

The informative signal was recorded by the E7-10 instrument exclusively in the mode of measuring the active component of the complex impedance, R. This requirement is due to the relatively large area of the sensor (200 mm²) and the minimal gap between the sensor and the grounded metal shell of the tube, which generate a significant parasitic capacitance of the dielectric PDMS interface. To minimize the reactive contribution of this capacitance to the total measured impedance, the structure was probed at a fixed alternating-current frequency of 1000 Hz. At this frequency, the capacitive impedance of the dielectric suppresses leakage currents to the metal substrate, localizing percolative charge transport exclusively within the graphene monolayer.

A distributed contact-pressure method was implemented in the study. Mechanical fixation was achieved using a rigid elastic semicylindrical fluoroplastic fixture (support bed), the geometry of which precisely reproduces the outer radius of the tube (R = 5 mm). When CVD graphene on a soft substrate (PDMS) is used for inspection of a 10 mm-diameter cylinder, an additional clamping load is essential. The optimum total clamping force of the fixture, controlled by a dynamometric assembly, is 2…5 N. This ensures a uniform normal pressure sufficient to provide air-free contact and conformal adaptation of the elastomer to the microrelief of the steel surface without irreversible plastic deformation of the polymer matrix.

Since, under uniform current flow across the entire resistor area, the measurement signal is geometrically averaged along the channel length, stable control of the internal stress field is required for reliable detection of submicrometer displacements in the defect regions (10…100 μm). The controlled loading of the system is achieved by varying the hydrostatic argon pressure inside the steel tube within the operating range of 0.5…2.5 MPa. Pressurization with gas forces an elastic local deflection of the thinned metal wall at the defect location. The local deformation of the graphene monolayer induced by this process reaches critical threshold values, ensuring the generation of a high-contrast piezoresistive response.

The relationship between the relative change in the active resistance of the graphene layer and local deformation is described by the Amjadi-Simons tunneling-percolation model [1, 2]. Taking into account that, within the elastic regime, the local deformation of a cylindrical shell in the region of a surface cavity is proportional to the internal pressure through the stress concentration factor [3], the resulting equation describing the electrophysical response of the heterostructure takes the form [8–10]:

$$\frac{\Delta R}{R_0} = A\left(e^{B\cdot k(h)\cdot P} - 1\right), \qquad (1)$$

where A – dimensionless factor determined by the initial topology of the percolation network and the baseline conductivity between the grain boundaries of the monolayer. $A \approx 0.04…0.05$; B – coefficient characterizing the rate of change in the tunneling conductivity of graphene during mechanical stretching of the elastomeric substrate. $B \approx 2.5…3.5$ (for standard optically transparent Sylgard 184 PDMS with the conventional base polymer-to-curing agent ratio of 10:1); P – argon pressure, MPa; $k(h)$ – stress concentration factor, which increases exponentially with increasing defect depth h, MPa^{-1}.

Unlike coefficients A and B, which are determined by the structural and material parameters of the sensor element and can therefore be considered its calibration characteristics, the coefficient $k(h)$ directly depends on the geometric parameters of the local defect and is specific to each value of its depth h. This coefficient was determined by experimental calibration using reference steel tube specimens with specified geometric parameters of the surface defects. Given the complexity of analytically describing the deformation-electrical conversion in the multilayer "steel shell-PDMS-graphene" system, the coefficient $k(h)$ was determined by inverse calculation. For this purpose, the basic equation describing the piezoresistive response was rearranged with respect to $k(h)$, making it possible to directly relate its value to the electrical signal measured by the E7-10 instrument at a specified applied pressure P:

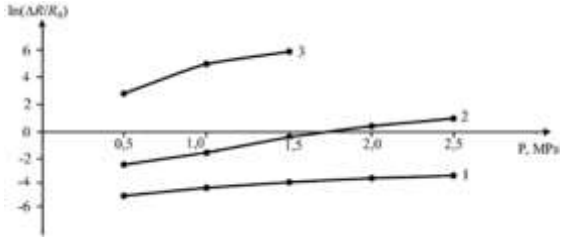
$$k(h) = \frac{1}{B\cdot P}\cdot ln\left(\frac{\Delta R}{A\cdot R_0} + 1\right). \qquad (2)$$

Experimental calibration for the investigated defect geometries yielded three characteristic values of the coefficient: k = 0.3 for a defect depth of h = 10 μm, k = 3.2 for h = 50 μm, and k = 11.5 for h = 100 μm. The obtained values indicate a nonlinear increase in the sensitivity coefficient with increasing local damage depth, which is consistent with the assumption of enhanced local strain concentration in the defect region.

To analyze the nature of the obtained dependence, a logarithmic transformation of the experimental data was performed:

$$ln\left(\frac{\Delta R}{R_0}\right) = ln(A) + B\cdot k(h)\cdot P.$$

In the graph (Figure), the ordinate represents the natural logarithm of the relative change in the electrical response, while the abscissa represents the corresponding parameter of the local defect. The use of the natural logarithm does not alter the physical meaning of the dependence under investigation; however, it provides effective linearization and facilitates comparison of the sensitivity of the sensor unit for defects of different depths. The approximation results are characterized by different slopes for the corresponding ranges of the geometric parameters of the defects, indicating a change in the intensity of the piezoresistive conversion with increasing degree of local damage.



Dependence of the natural logarithm of the relative electrical response on the applied pressure for different defect depths: 1 – defect depth of 10 μm; 2 – defect depth of 50 μm; 3 – defect depth of 100 μm

Thus, the results of the experimental study confirm the existence of a stable relationship between the geometric parameters of a local surface defect and the magnitude of the piezoresistive response of the sensor unit. As the defect depth increases, a systematic increase in the relative change in the active resistance of the graphene layer is observed, which is attributed to the enhancement of the local strain field in the damaged region. The established relationship provides a basis for considering the change in the electrical resistance of the graphene sensor layer as a quantitative diagnostic parameter for assessing the degree of local damage to the metal shell.

For the investigated range of defect geometric parameters, the following characteristic levels of electrical response were established:

– a microdefect with a depth of 10 μm produces a weak but stably detectable piezoresistive response in the range of 0.2…0.4%, corresponding to a change in active resistance of approximately 2…4 Ω at a baseline sensor resistance of approximately 1 kΩ. This signal level corresponds to the lower limit of reliable detection of local damage and characterizes the limiting sensitivity range of the investigated sensor system;

– a defect with a depth of 50 μm causes a more pronounced and monotonic increase in the relative change in resistance, reaching approximately 6% (30…60 Ω). This response level provides a substantial increase above the background signal and creates the conditions for reliable identification of local surface inhomogeneity of the metal;

– a defect with a depth of 100 μm is characterized by a sharp increase in the piezoresistive response to 15…35%, corresponding to a change in resistance of approximately 150…350 Ω. The substantial increase in signal amplitude compared with that of shallower defects indicates a significant enhancement of the local strain field and, consequently, an increase in the sensitivity of the graphene sensor layer to the development of surface damage.

The obtained results demonstrate the possibility of differential electrical monitoring of microscale defects and confirm the potential of the proposed sensor approach for the early diagnosis of localized degradation of cylindrical metal shells.

CONCLUSIONS

A correlation between the geometric parameters of local surface microdefects in the range of 10…100 μm and the dynamic change in the active component of the complex impedance of the graphene sensor layer operating in the tunneling-percolative charge-transport regime has been experimentally established. An increase in defect depth is accompanied by a systematic increase in the piezoresistive response, confirming the feasibility of using electrical resistance as a diagnostic parameter of local damage.

The specific features of measurement signal formation for small-diameter cylindrical metal objects have been established. Unlike conventional two-dimensional defect detection on flat plates, the nature of the electrophysical response is determined by the radial distribution of the mechanical stress field, the curved geometry of the surface, and the constrained bending effect of the thin-film sensor. These factors must be taken into account during calibration and quantitative interpretation of the signals generated by the sensor unit.

The sensitivity of the developed sensor unit to local stress concentrators formed by defects with depths of 10…100 μm has been confirmed. Controlled variation of the hydrostatic argon pressure within the range of 0.5…2.5 MPa provided controlled local deformation of the steel tube wall in the defect region and the corresponding modulation of the electrical resistance of the graphene layer. The obtained results demonstrate the feasibility of detecting microscale defects based on their piezoresistive response.

The possibility of differentiating the degree of local damage based on the amplitude of the relative change in the active resistance of the sensor has been demonstrated. For defects with depths of 10, 50, and 100 μm, qualitatively and quantitatively distinct levels of electrical response were recorded, providing a basis for establishing a calibration relationship between the geometric parameters of the defect and the measured signal.

The proposed approach can be considered a promising basis for the development of high-sensitivity non-destructive monitoring systems for the early stages of degradation of thin-walled metal tubes, particularly at the stages of initiation and development of localized microdefects.

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ДОСЛІДЖЕННЯ ВИСОКОЧУТЛИВОГО ТЕНЗОРЕЗИСТИВНОГО СЕНСОРНОГО ВУЗЛА НА ОСНОВІ ГРАФЕНОВИХ СТРУКТУР ДЛЯ ВИЯВЛЕННЯ РАННІХ СТАДІЙ РУЙНУВАННЯ МЕТАЛЕВИХ ТРУБОК МАЛОГО ДІАМЕТРА

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Досліджено високочутливий графеновий тензорезистивний сенсор для раннього виявлення мікродефектів металевих трубок малого діаметра. Експериментально встановлено кореляцію між глибиною дефекту 10…100 мкм та зміною електричного опору графенового шару. Показано можливість диференційованого виявлення ранніх стадій локальних пошкоджень за п’єзорезистивним відгуком сенсора.