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Investigation of impedance frequency characteristics of sensitive elements

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Abstract. This paper presents the results of investigating impedance frequency characteristics of sensing elements based on composite materials with varying content of carbon nanotubes (MWCNTs) and graphene. Five different substrate designs for sensing elements were analyzed to determine the optimal configuration in terms of noise immunity. The temperature characteristics of samples were studied in the range from -40°C to +40°C, as well as their behavior under high-voltage exposure. It was found that samples with 1% content of MWCNT/graphene mixture in a 70/30 ratio demonstrate the most stable characteristics in the studied temperature range and frequencies up to 50 kHz.

1 Introduction

The development of new sensing elements based on composite materials is an urgent task in modern electronics. Materials with the addition of carbon nanotubes and graphene, which possess unique electrophysical properties, are of particular interest. The main challenges in creating such elements are:

- ensuring stability of characteristics across a wide temperature range;
- achieving high noise immunity;
- technological reproducibility of parameters in mass production.

This work investigates the impedance characteristics of sensing elements based on composites with different percentages of MWCNTs and graphene. Special attention is paid to analyzing substrate design solutions to minimize the influence of high-frequency interference. A comprehensive study of temperature stability and behavior of samples under high-voltage exposure is conducted. The obtained results allow determining optimal parameters for composite composition and sensing element design to ensure required characteristics under real operating conditions.

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2 Experimental studies of the spectra of samples

All measurements were carried out by recording the response when a 1V sine wave voltage was applied to the sample at frequencies of 100 Hz, 1 kHz, 10 kHz, 50 kHz, 100 kHz, 500 kHz, 1 MHz.

Such frequencies make it possible to satisfactorily approximate the data obtained in order to determine the parameters of the replacement circuit. Based on the results of measurements at the specified frequencies R and X, Nyquist diagrams were constructed, which will be subsequently analyzed [1].

The objectives of the study were established:

A. Establishing the parameters of the sample manufacturing process, which ensured the technological repeatability of their production;
B. The choice of the design of the sensitive element having the least sensitivity to high-frequency interference;
C. Determination of the drift of the characteristics of the presented samples under the influence of different temperatures;
D. Determination of the drift of the characteristics of the presented samples under short-term high-voltage exposure;
E. Determination of the characteristics of samples with different percentages of the main and filler components.

1) Selection of the design of the sensitive element having the least sensitivity to high-frequency interference.

The structures shown in (Figure 1) were considered as proposed constructs. The main difference between the proposed designs was the size and location of the copper electrodes. For the purposes of the study, the substrates of the sensitive elements were milled according to these models on copper-plated textolite.

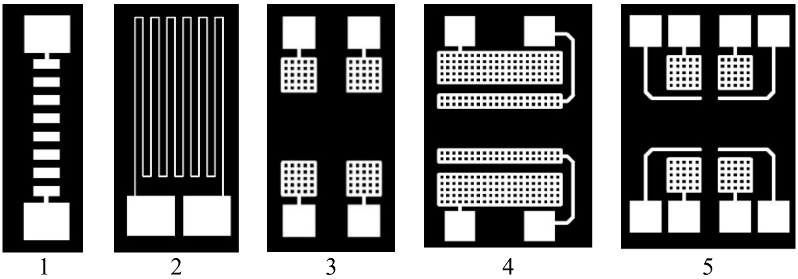


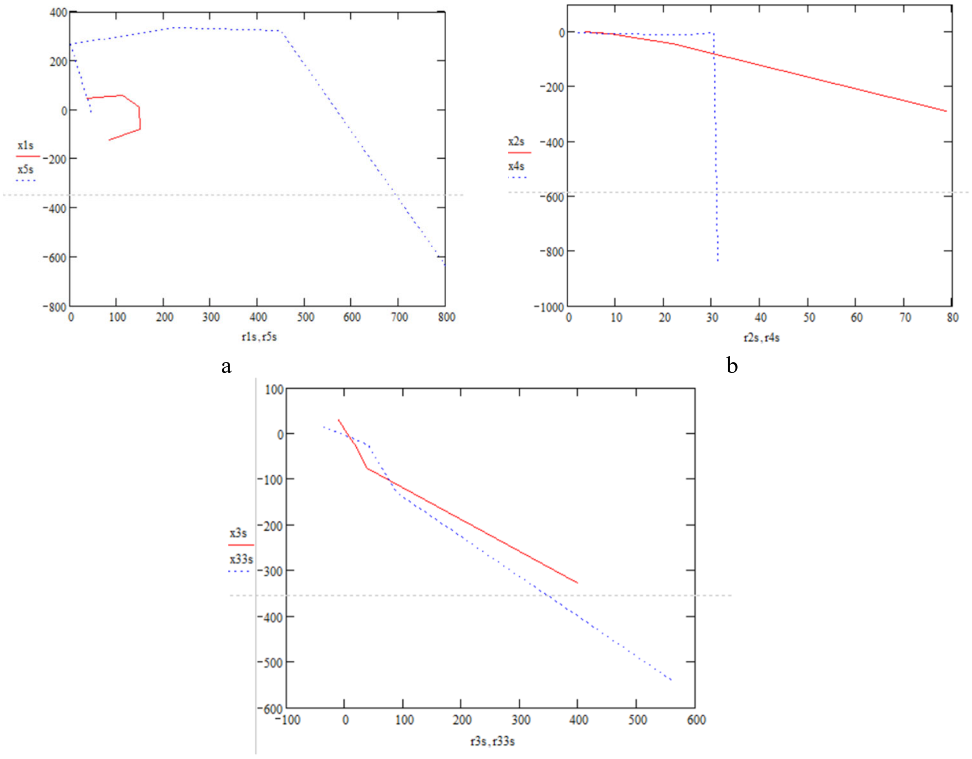
Fig. 1. Investigated types of substrate constructs.

The inductance value is adopted as the main parameter providing sensitivity to the effects of high-frequency induced interference on the sensing element. Therefore, to determine the most promising design solutions, studies of the frequency response of samples with an equivalent composite material were carried out (in the present case, elastosil with 1% 50/50 MUNT and graphene were used). The measurement results are shown in (Figure 1a-c). Where x1s, x2s, x3s, x4s, x5s are the reactance of the sample, r1s, r2s,r3s,r4s,r5s are the active resistance of the sample. As you can see, Figure b shows two graphs of measurements of the sample with the third variant of the substrate [2-3].

As you can see, in (Figure 1a), the graphs are located largely in the upper positive half-plane, which shows a significant sensitivity of the samples to interference and high inductance (this is indicated by a positive value of the reactance). Based on this, the samples of substrates 1 and 5 have shown their unsuitability as a basis for the manufacture of sensitive elements.

Substrate 2 also demonstrates the presence of an inductive component, as can be seen in Figure 15b, in addition, this is clear from the design of the substrate (low resistance and long length of the copper track). Based on the data obtained, the most promising will be 3 and 4 substrate variants (Figure 1b-c), in which the frequency response has a resistive-capacitive component, as well as the Warburg impedance. It should be noted that for 1, 2 and 5 of the substrate, kOhms are marked along the axes in this case, and for samples 3 and 4, the reactance is in kOhms, the active resistance is in Ohms.

So, as an option for studying the repeatability of the technology, three samples with a substrate of option No.4. The results of the spectrum measurement are shown in (Figure 2). The graphs show a repetition of the shape of the spectrum for different samples. So, with an equal active resistance, the reactive differs by 1.15-1.5 times. Further research is needed, including for other materials and fillers. Also, the departure of the graph into the upper half-plane indicates the presence of an inductive component introduced by substrate No. 4, which may have an effect on reducing the noise immunity of sensitive elements based on it.



a b

c

Fig. 2. The results of measuring the spectral component for samples made of elastosil with 1% 50/50 MUNT and graphene on various substrates: a - on substrates 1 and 5, b – on substrates 2 and 4, c – two samples on substrate 3.

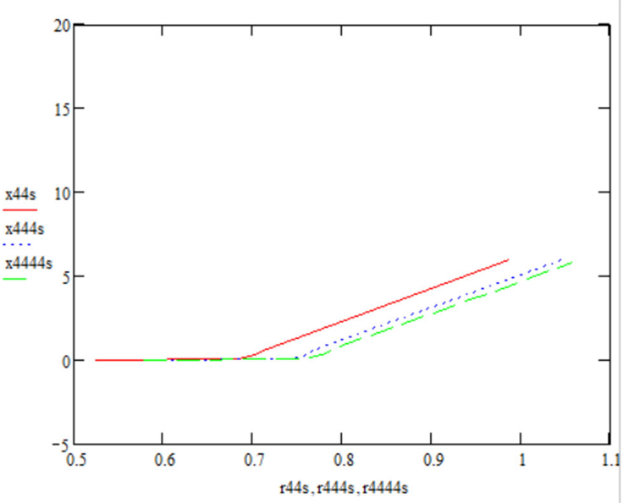


Fig. 3. Study of the repeatability of the electrical properties of samples with substrate No. 4 (elastosil with 1% 50/50 MUNT and graphene), along the abscissa axis – active resistance in ohms, along the ordinate axis – reactive in kOm.

The block of circuit breakers is designed to connect (disconnect) the uninterruptible power supply to an industrial three-phase network ~ 380/220 V ± 10% with a frequency of (50 ± 1) Hz. The uninterruptible power supplies provide uninterrupted power supply to the complex in the event of an emergency power outage.

Each of the twelve charging and discharging devices 160-5 is connected to the corresponding battery and regulates the charge and discharge current of the battery in accordance with the sequence diagram set from the control measuring computer complex. To utilize excess electricity, each charger-discharge device is connected to a corresponding load located in the load block of the BNS-30 automated stand.

2) Determination of the characteristics of samples with different percentages of the main and filler components.

Since the materials used are composite with different distances between the phase boundaries, in fact we will have different time intervals of the free path of the electrons. This circumstance determines the impossibility of linearization of the dependence of conductivity on the signal frequency due to the fact that there will be a significant discrepancy between theoretical and experimental data [4-5].

So, before calculating the resistances of the sensitive elements, it is necessary to make sure that the characteristics of the sensitive elements of the sensors can be repeated. To do this, it is necessary to produce several identical elements of samples of each type.

Samples a, d, e – demonstrated satisfactory repeatability of the characteristics according to the test results (Figure 4). Thus, a large discrepancy in the properties of sensitive elements is due to non–compliance with technological discipline - application errors, micro-gaps and porosity of the composite layer due to non-mixing (Figure 5), which causes a sharp increase in the active and reactive resistances of the samples. In these cases, work on improving the technological process of applying a uniform, equally thick layer of composite material bears fruit and allows you to achieve repeatability of results.

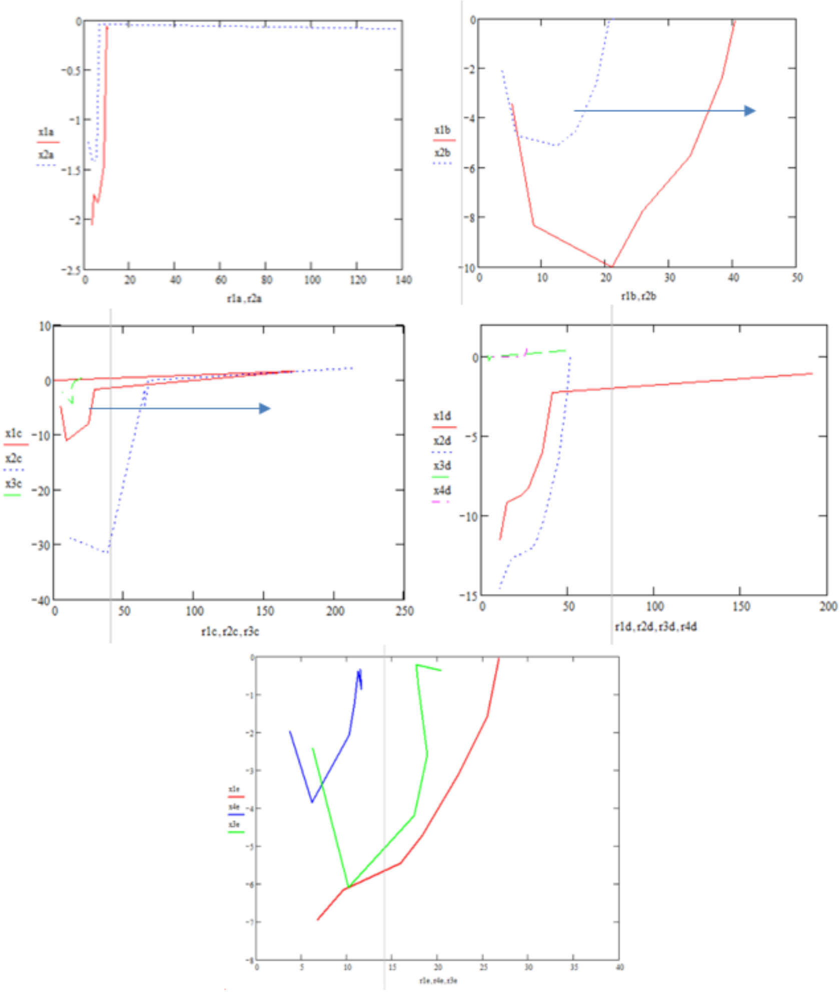


Fig. 4. Study of the repeatability of electrical properties of samples with substrate No. 4 (elastosil with fillers a - 3% 50/50 MUNT and graphene, b - 5% 50/50 MUNT and graphene, c - 1% 70/30 MUNT and graphene, d - 3% 70/30 MUNT and graphene, e - 5% 70/30 MUNT and graphene), along the abscissa axis – active resistance in ohms, along the ordinate axis – reactive in ohms.

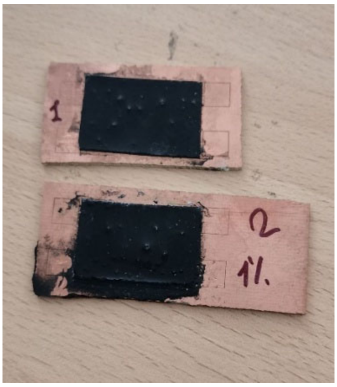


Fig. 5. Appearance of defects in the composite layer in case of non-compliance with technological discipline.

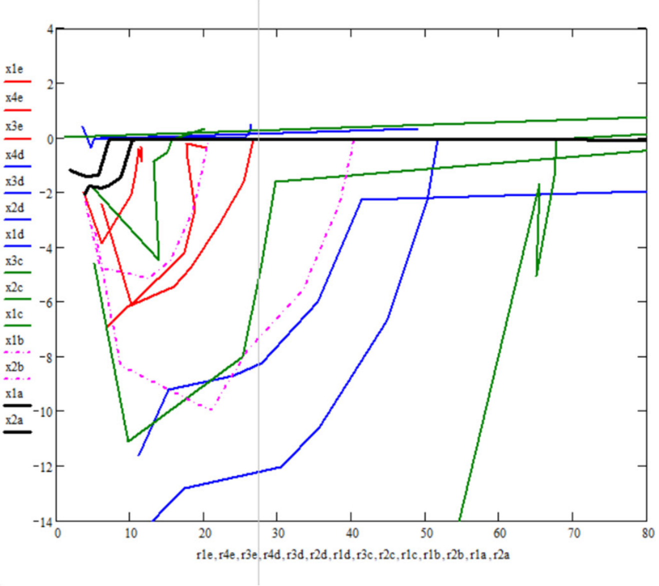


Fig. 6. Comparison of the electrical properties of samples with substrate No. 4 (elastosil with fillers: black - 3% 50/50 MUNT and graphene, pink - 5% 50/50 MUNT and graphene, green - 1% 70/30 MUNT and graphene, blue - 3% 70/30 MUNT and graphene, red - 5% 70/30 MUNT and graphene), along the abscissa axis – active resistance in ohms, along the ordinate axis – reactive in ohms.

Figure 6 shows a comparison of the impedance frequency characteristics of all the samples obtained. The following can be noted:

a. with an increase in the percentage from 1 to 5% of MUNT and graphene in a percentage of 70/30, the sample capacity increases;
b. with an increase in the percentage from 3 to 5% of MUNT and graphene in a percentage of 50/50, the sample capacity decreases;
c. the smallest capacity is 1% 70/30 MUNT and graphene (Figure 6 green), 3% 70/30 MUNT (Figure 6 blue);
d. the largest capacity is 3% 50/50 MUNT and graphene (Figure 6 black).

The calculation of the capacities for these samples was carried out according to the equivalent scheme shown in Figure11.

The maximum radius of the circle in the green graph in Figure 6 is Z'=-31.6 kOhm at a frequency of 500 kHz, the active resistance is 67.9 kOhm.

The minimum radius of the circle in the black graph in Figure 6 is Z'= -1.39 kOhm at a frequency of 1 MHz, the active resistance is 7.2 kOhm. According to calculations, 20.08 pF is obtained for the green graph. For black – 691.5 pF. The size of the capacity indicates its nature. According to the table.1 it may be due to the capacitance between the grain boundaries formed by carbon nanoparticles [6].

e) inclined lines above the abscissa axis to the right of the semicircle in samples of 1% 70/30 MUNT and graphene, 3% 70/30 MUNT (Figure 6 - green and blue) indicate the presence of diffusion phenomena between the composite material and the substrate, therefore, the model of such samples should be supplemented with a Warburg element;

f) the second semicircle on the left of samples 3 and 5% 70/30 MUNT and graphene (Fig. 6, blue and red graphs) shows the presence of another RC pair in the substitution circuit with lower resistance and higher capacitance (about 50 pF);

g) artifacts of measurements at low frequencies (Figure 6, green graph - 1% 70/30 MUNT and graphene) indicate an imperfection of the method of variation in the low frequency region, especially in the presence of a larger capacity of sensitive elements.

Figure 7 shows the results of the study of samples of 1%, 3% and 5% MUNT without other impurities (the far axis is the percentage of samples: 0 - 1%, 1 - 3% and 2 - 5%; the near axis is the active power in KOM; the vertical axis is the reactive power in kOm) [7-9]. A 3-fold change in the sample capacity is clearly shown with an increase in the content of MUNT from 1% to 5%. The capacity is due to the intergrain interaction.

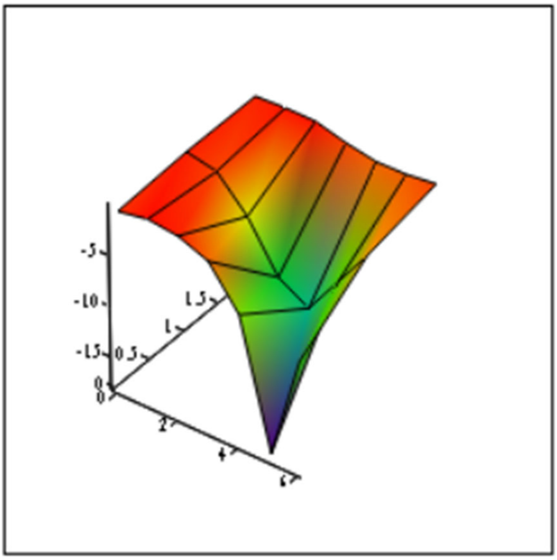


Fig. 7. Comparison of electrical properties of samples with substrate No. 4 (1%, 3% and 5% MUNT), far axis – percentage of samples: 0 - 1%, 1 - 3% and 2 - 5%; the near axis is the active power in KOM; the vertical axis is the reactive power in kOm.

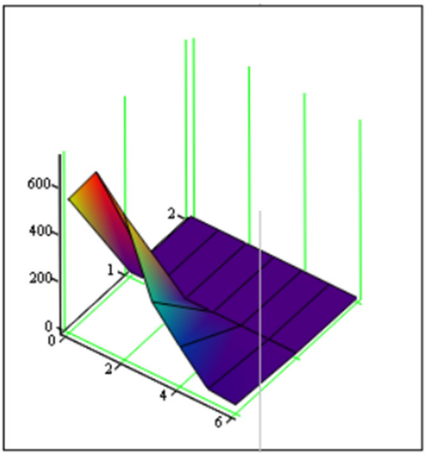


Fig. 8. Comparison of electrical properties of samples with substrate No. 4 (1%, 3% and 5% graphene), far axis – percentage of samples: 0 - 1%, 1 - 3% and 2 - 5%; the near axis is the active power in KOM; the vertical axis is the reactive power in kOm.

A separate study of the electrical characteristics at 1%, 3% and 5% graphene content without MUNT was carried out (Figure 8). The change in the sample capacity is more than 100 times with an increase in the graphene content from 1% to 5%. The capacity is due to the intergrain interaction [10].

3) Determination of the drift of the characteristics of the presented samples under short-term high-voltage exposure.

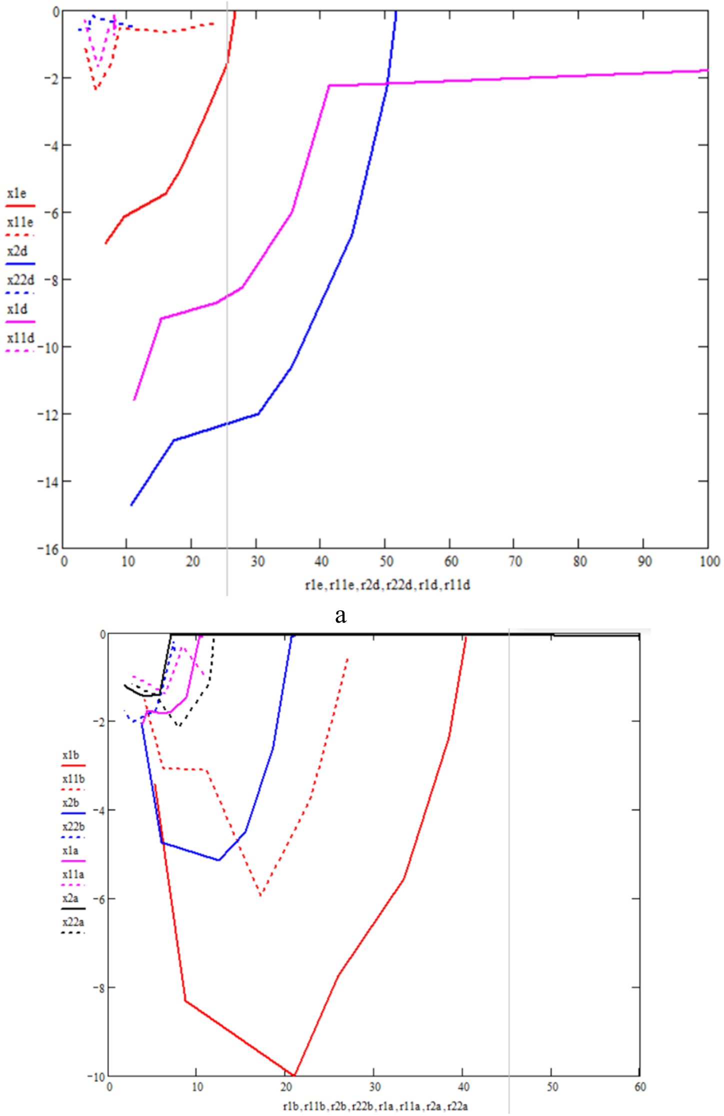


Fig. 9. Comparison of the electrical properties of samples with substrate No. 4 before and after exposure to 2.5 kV voltage (solid line – before exposure, dotted line – after) a: red - 5% 70/30 MUNT and graphite, blue and pink - 3% 70/30 MUNT and graphene; b: red and blue - 5% 50/50 MUNT and graphene, pink and black - 3% 50/50 MUNT and graphene.

Studies of the characteristics of samples under short-term exposure to a voltage of 2.5 kV. The results are shown in Figure 9. The solid line shows diagrams of samples before exposure, dotted – after. As follows from the obtained graphs, the effect caused a significant decrease in the active and reactive components in all samples. In addition, this decrease occurred to the same value [11]. The final capacity after exposure was about 0.59 nF. The reason for this change may be the breakdown and burnout of a part of the dielectric filler of the composite material, followed by the formation of conductive layers. Nevertheless, obtaining similar characteristics after high-voltage exposure for different materials may be the basis for introducing such effects into the manufacturing technology of sensitive elements to unify samples during their mass production. Additionally, this information may be useful in organizing stable operation of sensors based on these elements in conditions where the latter are fixed to conductive parts of technological equipment (crane tracks, rails, etc.), where sensitive elements may be subjected to similar influences during operation [12-13]. A significant point in this case remains the protection of microcontroller devices from leakage currents, which can damage the system through the sensitive elements. In order to avoid it, it is proposed to install TVS diodes in parallel with the sensitive elements.

3 Conclusion

Based on the comprehensive research conducted, several key conclusions can be drawn. Among the five investigated substrate designs for sensing elements, type 3 and 4 configurations demonstrated the best noise immunity characteristics, showing predominantly resistive-capacitive components in their impedance spectra. Samples with 1% content of MWCNT/graphene mixture in a 70/30 ratio exhibited the most stable characteristics across the temperature range from -40°C to +40°C, excellent reproducibility in manufacturing, and stable performance in frequencies up to 50 kHz.

Increasing MWCNT/graphene content from 1% to 5% in 70/30 ratio leads to increased capacitance, while increasing content from 3% to 5% in 50/50 ratio results in decreased capacitance. The lowest capacitance was observed in 1% 70/30 MWCNT/graphene samples.

High-voltage exposure testing (2.5 kV) revealed significant reduction in both active and reactive components for all samples, with convergence of electrical characteristics to similar values after exposure, suggesting potential application of high-voltage treatment for standardizing sensor elements during mass production. Temperature stability analysis showed minimal frequency response variation in the low-frequency range (up to 10 kHz) and significant changes in high-frequency characteristics (500 kHz-1 MHz) for 3% and 5% samples, confirming superior temperature stability of 1% samples in the operating frequency range.

The results indicate that 1% 70/30 MWCNT/graphene composite material is optimal for manufacturing sensing elements with stable characteristics and good reproducibility. Further improvement in manufacturing technology, particularly in mixing procedures, could enhance the uniformity of nanotubes distribution and further improve performance consistency.

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