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Nanocomposite Sensors of Indium Tin Oxide Nanowires in a PMMA Matrix for Structural Health Monitoring

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Abstract. *This paper proposes a new sensor that uses a nanocomposite composed of Indium Tin Oxide (ITO) nanowires embedded in a Poly(methyl methacrylate) (PMMA) matrix to detect the presence of damage in a structure. The nanocomposite has high transparency and conductivity. The proposed analysis measures the resistivity between terminals because the fiber network can change when damage modifies the nanowire's position. An electric circuit converts the voltage applied to the sensor into resistivity. However, some weaknesses still need to be studied to enable a broader sensor application. This project addresses two key points: defining the damage and location and assessing the temperature and humidity influence on the sensor. Experimental tests are conducted on an aluminum beam representing a structure, with damage-emulating cracks introduced on the sensor. The results demonstrate that the sensor's resistivity varies with the introduction of damage, and the resistivity changes with the damage size. The nanowires dispersed in the matrix undergo reorganization due to their difficulty in breaking, which changes the electric current path and may increase or decrease the resistivity. The new arrangement of the nanowires affects a large part of the sensor, and the change in the intensity of the resistivity does not indicate the location of the damage. All tests are conducted in a climatic chamber to ensure constant temperature and humidity. By varying the temperature, it is observed that the increase in temperature changes the resistivity due to the material characteristics. The same occurs with humidity, but the conductivity variation is smaller. These initial tests need more reproducibility to ensure the broader use of this new sensor and to develop methods that standardize and facilitate their installation in structures.*

Keywords: *Nanocomposite sensors, ITO, PMMA matrix, resistivity, Structural Health Monitoring*

1. INTRODUCTION

Structural systems are present in various applications in modern society, such as aeronautical structures, civil infrastructure, and industrial equipment (Baptista *et al.*, 2010; Wang *et al.*, 2020; Farrar and Worden, 2007). Structural health monitoring (SHM) techniques focus on identifying damage that can propagate and cause structural failure. This field has been the subject of numerous studies in industry and academia, resulting in a substantial body of literature on techniques and methodologies that have economic interests and improve safety (Lopes *et al.*, 2023; Baptista *et al.*, 2010, 2014; Cortez *et al.*, 2013).

SHM involves monitoring and identifying damage through non-destructive testing, which requires using sensors, typically permanently installed on the structure under analysis (Cortez *et al.*, 2013). By employing this approach, acquired signals can be analyzed within the context of a healthy system, detecting damage initiation and providing guidance on appropriate actions based on the findings (Baptista *et al.*, 2014).

High-cost commercial systems are commonly used in SHM applications that require high sampling frequencies to ensure reliable results, with the analysis focused on the impedance's frequency response function (FRF). In this context, SHM methods based on electromechanical impedance (EMI) offer a promising alternative due to their utilization of low-cost acquisition and sensing systems, as well as the simplicity of analysis and data acquisition (Baptista *et al.*, 2010, 2014; Cortez *et al.*, 2013).

The EMI technique is based on the piezoelectric effect. Piezoelectric materials, such as piezoelectric transducers (PZTs), can convert electrical energy into mechanical energy and vice versa. This property allows PZTs to function as both actuators and receivers. By interacting with the mechanical impedance of the system, PZTs facilitate damage identification by measuring the electrical impedance of the transducer (Cortez *et al.*, 2013).

Implementing the EMI technique requires specific signal transmission, acquisition, and analysis equipment. Furthermore, correctly interpreting the acquired data requires operators with prior knowledge and expertise. To address these challenges, this study aims to develop a solution that utilizes static measurements and parameter comparison by applying nanocomposite sensors. Existing literature presents various applications of nanocomposites for structural monitoring using static measurements. For instance, Thostenson and Chou (2008) employed carbon nanotubes to monitor bolted joints in composite materials. The study involved conducting tensile tests on single-lap and double-lap bolted joints and successfully establishing a relationship between resistance variation and shear-out failure. Additionally, they observed that resistance variation occurred when the bolts were not adequately tightened.

In another study, Loh *et al.* (2009) conducted damage identification on plates subjected to impact using multi-layer composites of carbon polyelectrolyte nanotubes. The researchers applied three different impact intensities to the system and measured conductivity at the front and back of the plate. They found a linear relationship between impact energy and conductivity, with increased conductivity variation for high-energy impacts.

Similarly, Gao *et al.* (2009) developed a monitoring system for composite structures using a network of carbon nanotubes and acoustic emission, comparing the two methods. By measuring the electric resistance of the nanotubes, they successfully identified damage to the composite under quasi-static and cyclic conditions, as evidenced by changes in the measured resistance. Both analyses demonstrated a bilinear relationship in damage detection for the tested structures, providing valuable information on the damage evolution. In metallic structures, Ashrafi *et al.* (2012) monitored cracks using carbon nanotubes embedded in an epoxy matrix. Although the sensor could not precisely determine when the crack reached the film, it could detect it after less than 5 mm propagation. As the damage propagated throughout the nanocomposite, the resistance tended toward zero. Applying normalization to the collected data proved more accurate in identifying crack propagation. The sensor exhibited a current drop at the crack entrance, followed by linear decay with crack propagation, allowing estimation of crack size through static measurements.

Furthermore, Takiuti *et al.* (2014) and Takiuti *et al.* (2016) utilized ITO (indium tin oxide) nanocomposite films in a PMMA (Polymethyl Methacrylate) matrix. The former study focused on damage identification and observed variations in electric resistance caused by damage. The latter study investigated the influence of different temperature and humidity conditions on the measurements of the nanocomposite and found minimal impact on the sensor's performance.

This paper builds upon the work of Arlindo *et al.* (2012) and Orlandi (2005), who introduced nanocomposite films consisting of indium tin oxide (ITO) nanowires embedded in a PMMA matrix. In this study, we aim to detect damage in an aluminum plate and analyze the behavior of the sensor under various humidity and temperature conditions. Measurements will be conducted over multiple days using a simple circuit that provides the material resistivity, indicating the presence of damage in the analyzed system.

2. ITO/PMMA NANOCOMPOSITE SENSOR

Nanomaterials exist on the nanometer scale, with materials like carbon nanotubes and indium tin oxide (ITO) nanowires having two nanometric dimensions (Arlindo *et al.*, 2012). These materials exhibit unique properties that make them suitable for various applications. ITO, for example, is widely used in optical-electronic devices due to its high electrical conductivity and good optical transmittance. It also possesses desirable characteristics such as hardness, substrate adherence, and chemical inertness (Farahmandjou, 2013).

The ITO used in this study was obtained through the carbothermal reduction method proposed by Orlandi (2005). In this method, oxides are mixed with carbon black and evaporated in a tube furnace. The crucible containing the materials is placed in the region with the highest temperature and vaporizes when reaching a temperature of 1260°C. The growth of nanowires follows the vapor-liquid-solid (VLS) mechanism, where metallic drops act as catalysts by absorbing the vaporized reagents. Once the drops become saturated, nucleation occurs, guiding the growth of monocrystalline structures (Arlindo *et al.*, 2012). Figure 1 schematically illustrates the production of ITO, with nanowires formed at the opposite end of the N2 flow to create an inert atmosphere.

The formation of a sphere at one end of the nanowire is characteristic of this process, having a higher concentration of tin than the rest of the single crystal. The formed nanowire has a square cross-section due to the formation of monocrystals in the cubic phase of In_2O_3 (Arlindo, 2014). These characteristics are evidenced by the scanning electron microscope (SEM), as shown in Fig. 1.

The ITO can be used together with other materials to form a nanocomposite. The most common matrix used for ITO is the Poly(methyl methacrylate) (PMMA) because it has high transparency, good mechanical properties, and is insulating. Thus, the nanocomposite can conciliate good transparency and high conductivity (Arlindo *et al.*, 2012).

According to tests conducted by Arlindo *et al.* (2012), using 10% wt of ITO about the amount of PMMA guarantees the percolation limit. In this way, this forms a path to the electric current and ensures the film's conductivity. This parameter is important because it will be responsible for the sensor sensitivity. For a sample with a higher density of wires, changing the paths would generate low variation in the conductivity measurements. On the other hand, the percolation limit guarantees fewer paths traversed by the electric current due to the smaller number of possible directions.

The percolation limit, represented in Fig. 2, is reached in item 2, as it presents limited possibilities of paths for the course of the electric current. In the first condition, the nanocomposite does not reach percolation, resulting in isolated

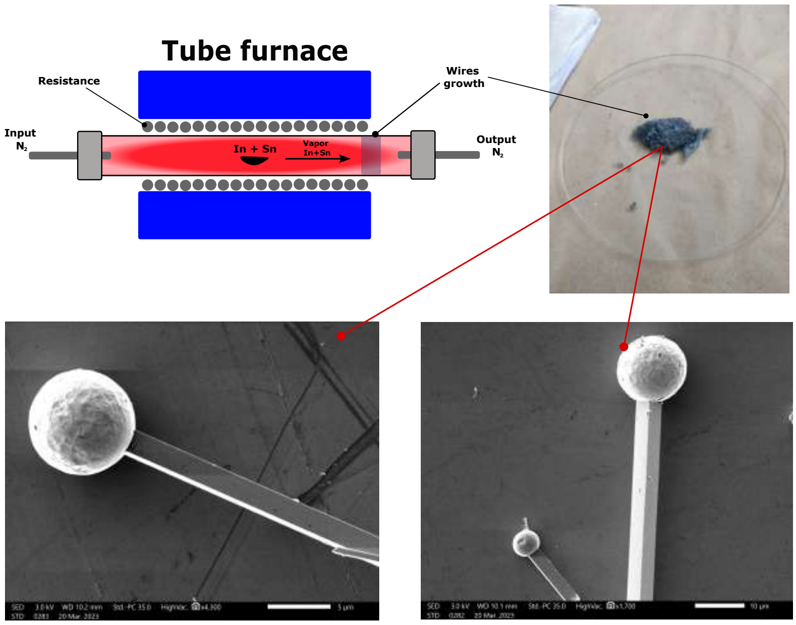

Figure 1. ITO nanowire growth scheme and SEM images.

behavior with no conductivity between the terminals. In the third case, there are multiple paths for the current flow. When damage is introduced, causing a change in the current path, the resistance shows minimal variation, unlike the second scenario.

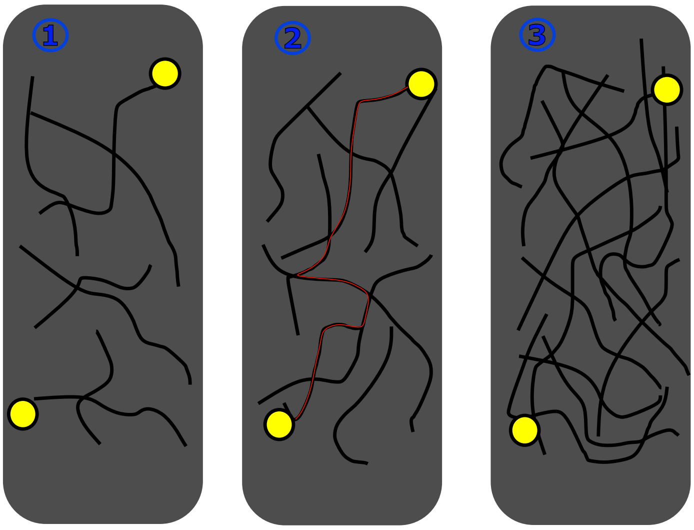

Figure 2. Scheme indicating the arrangement of wires and percolation.

The crucial aspect is to verify if the nanocomposite has ohmic resistance. When the voltage is adjusted, the resulting electric current follows a linear curve, with the slope representing the electric resistance. The linearity of the curve ensures that the resistor is ohmic, meaning that its resistance remains constant regardless of changes in the power supply. Arlindo (2014) conducted this test and proved that the nanowires have an ohmic resistance. This material was produced at the Chemistry Institute in Araraquara - São Paulo.

3. METHODOLOGY TO ESTIMATION NANOCOMPOSITE RESISTANCE

The terminals were introduced into the sample using carbon paint to fix and connect the wire at a specific point in the nanocomposite. Silicone resin was applied to ensure contact between the wire and ITO and to protect the region. The terminal positions were introduced at the ends of the sample only to characterize the material. As the arrangement of the wires is unknown, and thus it is impossible to optimize the positions of the terminals. Measurements are conducted by pairing two terminals simultaneously, resulting in various combinations. The samples used in the experiment have five terminals, resulting in ten resistance measurements.

The resistivity measurements at each terminal are performed using an electronic circuit comprising a known resistor (R_{ref}) and a voltage source (V_1). As shown in Figure 3, the circuit setup was constructed using the National Instruments ELVIS II system. The circuit is supplied with a constant voltage of 5V, and the voltage across the nanocomposite terminals (V_2) is measured. By applying Ohm's Law and Kirchhoff's Laws for the mesh, the resistance of the sensor can be determined using the following equation:

$$R_{\text{ITO}} = \frac{V_2 \, R_{\text{ref}}}{(V_1 - V_2)} \tag{1}$$

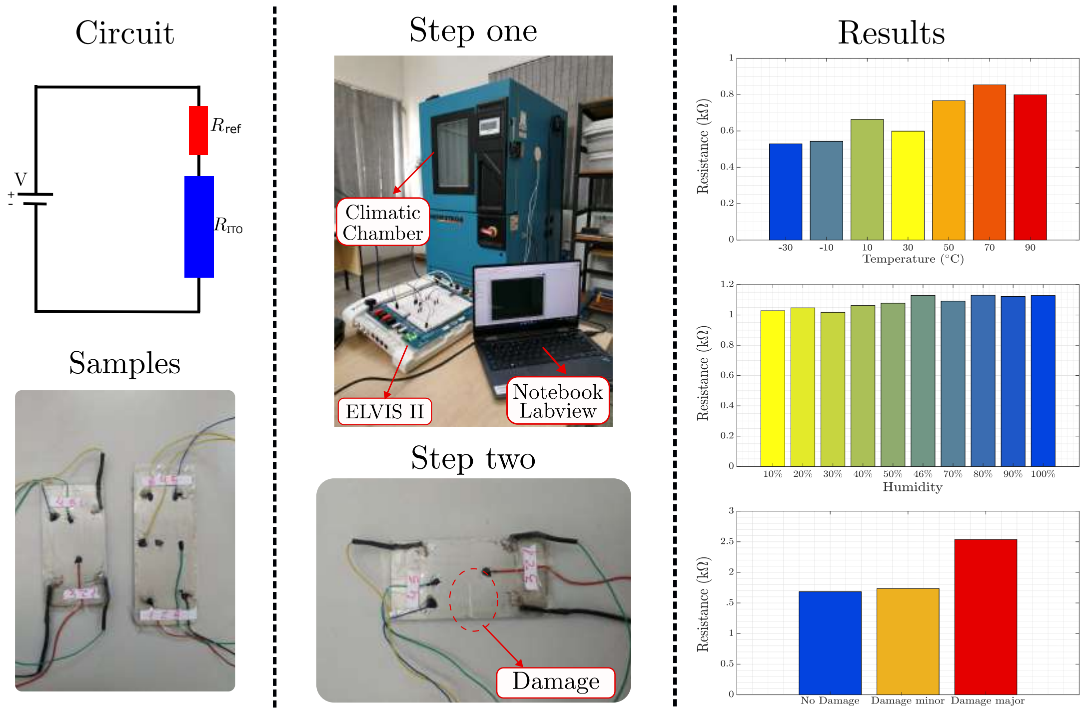

Figure 3. Graphical abstract indicating the circuit, samples, experiment steps and results.

Figure 3 presents the abstract of the steps to be applied to promote the tests and obtain the results. The first stage of the tests consists of introducing the sample in the Thermotron SM-8-8200 climatic chamber. It will vary the temperature from $-30°$C to $90°$C and the humidity from 10% to 100%. The resistance measurements are collected for each condition and then analyzed to identify the nanocomposite behavior.

In the second stage, the damage is introduced in the nanocomposite to verify the behavior of the static measurements. Initially, the test is performed for the sample without damage. Two damage are introduced in the nanocomposite, a with 10 mm and then increased to 20 mm. Stage two in Fig. 3 shows the damage, and in both situations, resistances are measured. In this way, it will be possible to verify if the sensor can identify and locate the damage generated in it.

In both stages, the power supply signals and the voltage on the sample channels are acquired for 30 seconds 3 times for each situation. These two voltage data are used to find the resistance through equation 1. Finally, the average of the three acquired resistances in each channel is analyzed for damage and environmental conditions. Therefore, a resistance value and standard deviation will be obtained for each analysis condition.

4. RESULTS

The first step is to verify the resistance behavior of nanocomposite when varying the temperature in the climatic chamber. The results of the channels at a set temperature are shown in Fig. 4. The electric resistance measured in each channel shows variation for the same temperature. This behavior is due to the non-uniformity in the deposition of the nanowires. The temperature variations observed in each channel exhibit an upward trend, which is typical for good conductors, although some points within the channel do not conform.

The temperature variation impacts the measured nanocomposite resistivity due to its influence on the conductive properties of the nanowires. As the temperature increases, the conductivity of the nanocomposite decreases, increasing the resistance.

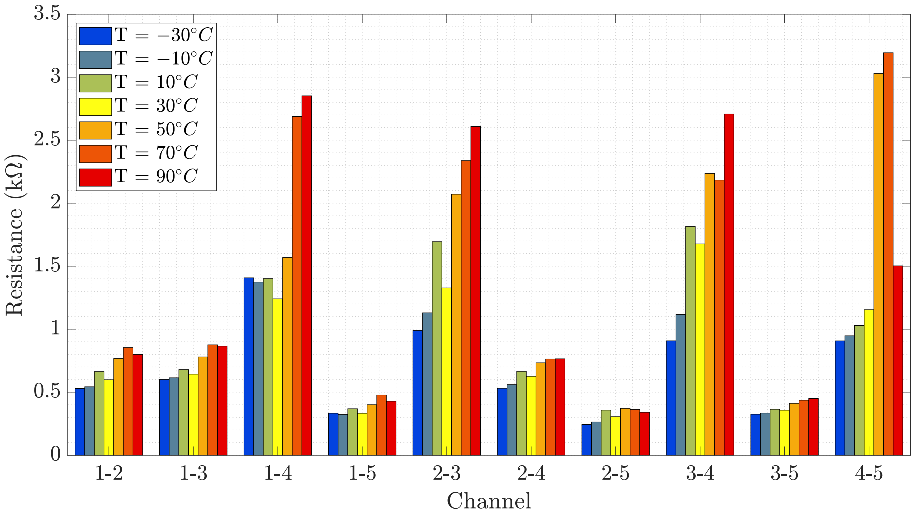

Figure 4. Resistance behavior due to temperature variation in each channel.

The temperature was set at 40°C, and the humidity ranged from 10% to 100% in the climatic chamber. As seen in Fig. 5, the variation in measurements tends to be smaller than the temperature, and in some channels, the variation can be considered negligible. Thus, the influence of humidity on the sensor is minimal.

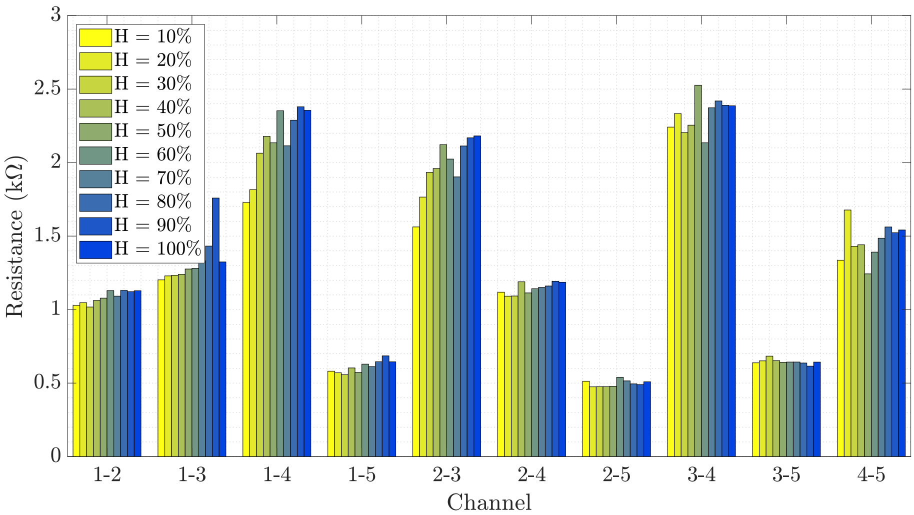

Figure 5. Resistance behavior due to humidity variation in each channel

The second step was performed to measure the resistivity of the undamaged and two damaged conditions in the nanocomposite. Figure 6 shows the resistance behavior for three situations. The increase in resistance on some channels was relatively low, as is the case with channels 1-2, 1-3, 2-4, 2-5, and 3-5. In these channels, the damage has a low influence on the passage of electric current since the nanowires connection is established around the damage region, in addition to allowing a new arrangement of the nanowires. In the last channel, however, there was a significant increase, indicating that the arrangement hinders the passage of electrons and thus reduces the conductivity.

The other channels showed a decrease in resistance when subjected to damage. This fact may result from the new combination of nanowires and facilitated the passage of electrons, providing increased conductivity. In the case of channels 1-4 and 3-4, the increase in damage modified the resistance due to the increase in damage that interfered with the nanowires to reduce the conductivity between the two points.

The results were inefficient in locating the damage since it interfered in several channels. The random dispersion of nanowires within the sample, along with variations in wire dimensions, poses challenges in pinpointing the exact location of the damage. This occurs because the failure in one channel can potentially impact neighboring channels, making it challenging to identify the damaged location.

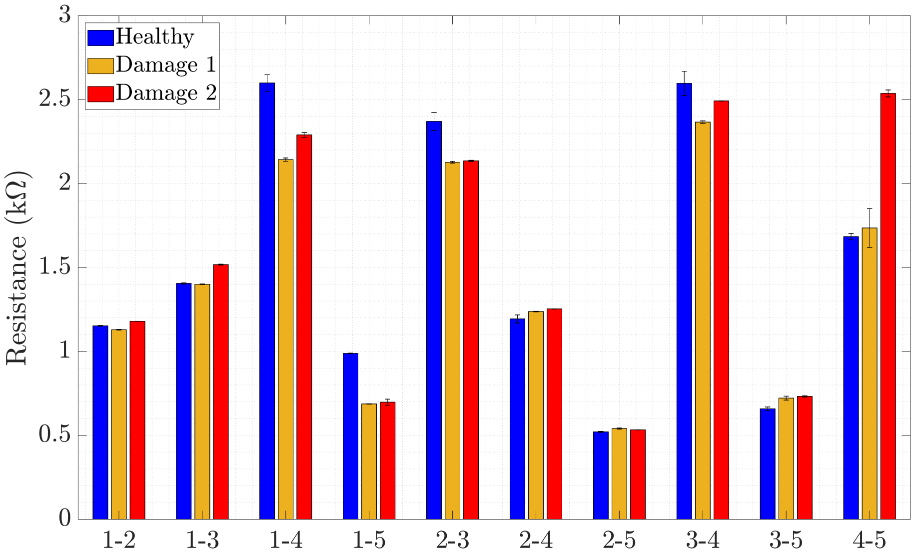

Figure 6. Resistance behavior for the undamaged (Healthy) nanocomposite, damage of 10 mm (Damage 1) and 20 mm (Damage 2)

5. CONCLUSION

This work presented the initial results for the characterization of the nanocomposite to identify its behavior when subjected to varied temperature and humidity conditions, in addition to verifying the sensitivity to damage and the ability to locate this failure in the structure.

The results showed that the temperature influences the material resistivity since it is a conductive material and therefore tends to decrease the conductivity with the increase in temperature. Humidity showed low variation, but it is possible to identify an upward trend in the measurements, but this variation is more minor compared to temperature.

When introducing a failure in the nanocomposite can be identified by the increase or reduction of conductivity, since when damaging it, the rearrangement of ITO nanowires is altered and modifies the electric current path and hinders or facilitates the passage of electrons. The results obtained do not allow identifying the precise location of the damage. The analyzed channels exhibited variations in measurements, making it challenging to estimate the specific region affected by the failure.

The sensor has great potential due to its simplicity in collecting and analyzing results since low-cost and compact systems would be sufficient for the analysis. In addition, it is necessary to implement methodologies to achieve a uniform dispersion of nanowires, which would facilitate the identification, estimation of the damage location, and enable optimization of analysis points. Lastly, for future studies, there is a need to investigate the material's behavior in dynamic systems.

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