

Graphite Piezoresistive Sensors in Polymeric Substrates

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Abstract— In this work the results of the characterization by digital microscopy, scanning electron microscopy and EDX of the mechanical properties of the graphite 2B deposited by the GoP - Graphite on Paper process on A4 paper substrate are presented. The different properties and network structure of the graphite film and paper chosen for the development of sensing devices are aimed at practical applications in MEMS - Micro electromechanical devices.

Keywords—MEMS, Sensors, Piezoresistive Effect, Polymers, Graphite.

I. INTRODUCTION

In recent years a wide range of devices and sensors using graphite films and paper substrates is being developed, including strain gauges, super capacitors, energy storage and electromagnetic radiation detectors in the mid-infrared range [1]. The literature shows that most of the work in the area focuses on sensor production methods and the exploration of new applications of cellulose as substrate for different types of devices [2]. In this work the graphite is characterized by depositing itself on the paper, aiming to obtain a piezoresistive sensor. The results will be compared, in the future, with the results obtained by a numerical computational simulation [3].

II. PIEZORESISTIVE SENSOR ELEMENT ON POLYMERIC SUBSTRATE

2.1. Piezoresistive sensor element

A sensor has as main purpose the conversion of energy between the different domains, that is, to react to a signal and convert it to another type of signal. Piezoresistive sensors are an example of passive transducers because they require an external excitation that can be originated by a crimped beam or a source of electric current [3]. According to the literature [4], there are several methods to measure forces considering the changes of the dimensions of the materials due to the applied mechanical stresses. The strain gauge type piezoresistive sensors are

widely used for this purpose. The piezoresistivity consists in the reversible change of the resistivity that a material presents due to a mechanical effort [5]. The piezoresistive sensor has a direct dependence on the property of the material from which it is made. In electrical and thermal terms, the most significant properties for the manufacture of the sensors are those related to the resistivity and mobility of the load carriers. In mechanical terms, density, thermal expansion, modulus of elasticity and Poisson's coefficient [6] are considered.

In order to generalize the properties of the material to be used as sensor element, the piezoresistive coefficient, π_{ij} , was defined as the entity that considers all the measurable properties of the material. These coefficients are related to the concentration levels of dopant impurities, crystallographic orientation of the material, temperature and, consequently, the conductivity type according to [7]. The piezoresistive coefficient is an intrinsic property of the material considered and chosen as a sensor element, and it is possible to adjust its magnitude in the laboratory, that is, to improve the physical properties of the material [6].

These parameters are important for the technological processes of manufacturing the sensing devices and actuators since they are related to the sensitivity of the devices [3, 8, 9]. The piezoresistive effect is defined by equation (1),

$$\frac{\Delta\rho_{ij}}{\rho} = \pi_{ijkl} T_{kl} \quad (1)$$

Where, π_{ijkl} , is the piezoresistive coefficient tensor, an intrinsic property of the material chosen as sensor elements and which can be adjusted by specific doping techniques. The mechanical stress in the structure of the material is given by T_{kl} , where $\Delta\rho_{ij} / \rho$ is the ratio of the electric resistivity when the material is subjected to external tensile forces or mechanical deformations.

In general, the structures of the films deposited on the substrates have a thickness much smaller than the

thickness of the substrate itself, therefore, the mechanical stresses are transmitted integrally from the substrate to the film according to the theory of the small deflections. Surface and substrate roughness can also produce thermal and mechanical hysteresis effects through the creation of concentrated mechanical stresses which produce loss of sensitivity and adhesion of deposited films.

A deformation in a given material, exerted along a given direction, always causes changes in all dimensions. The piezoresistive effect can be described by expressing the change in electrical resistance, ΔR , in a macroscopic manner as a function of the mechanical stress, mechanical strain, ϵ , and the sensitivity factor or gauge factor, GF given by equation (2).

$$GF = \frac{\Delta R}{R\epsilon} \quad (2)$$

The sensitivity factor is dependent on the crystallographic orientation of the material and is related to the piezoresistive coefficient through the Young's modulus, E , given by equation (3),

$$GF = \pi_{ij} E \quad (3)$$

The relationship between the change in sensor dimensions and sensitivity is given by the Poisson coefficient through equation (4),

$$GF = 1 + 2\theta + \frac{\Delta\rho}{\rho\epsilon} \quad (4)$$

Where θ is the Poisson's coefficient and the term $1 + 2\theta$ represents the change in the dimensions of the material and $\epsilon = \Delta L / L_0$ is the mechanical deformation.

2.2. Graphite Characteristics

Graphite is formed of carbon atoms that bond together by covalent bonds. Each carbon atom is attached to three other carbon atoms in the same plane. An atom is arranged in such a way that its bonds are the vertices of a triangle, leading to the formation of hexagonal rings. Thus sheets of carbon atoms bonded together by covalent bonds are formed. The layers of atoms forming the graphite are bonded together by very fragile bonds. In general graphite is a soft mineral, being an electric conductor, has applications in electronics, as in electrodes and batteries. Graphite is the most stable crystalline form of carbon. It consists of infinite layers of carbon atoms hybridized in sp^2 bonds. In each layer, called a graphene sheet, one carbon atom binds to three other atoms, forming a planar arrangement of fused hexagons [10].

Hybridization of the sp^2 type occurs with carbon atoms forming a double bond. Carbon hybridization results from a double bond and two single bonds. Graphite is a good conductor of electric current, however it does not allow thermal conduction.

The density of the graphite turns around 2.26 g / cm^3 . Graphite can be found in natural or synthetic form and comes in three distinct forms: amorphous, crystalline and flakes. Natural graphite is one of the allotropic forms of

carbon found in nature, while synthetic is produced industrially with the use of high temperatures and pressure.

2.3. Process of deposition of the graphite film

Figure 1 shows the process steps of deposition of the piezoresistive sensor elements on the substrate by mechanical exfoliation of graphite 2B, a process known as GoP-Graphite on Paper [11,12] produced by traces of pencils.

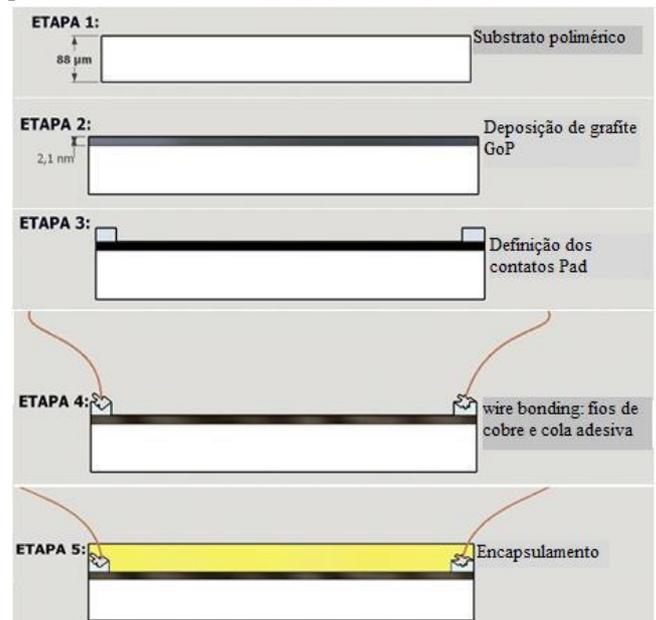


Fig. 1: Stages of piezoresistive sensor element processing of graphite.

Figure 2 shows a photograph of the piezoresistor obtained through a digital microscope Dino-lite model AM-313T. To finalize the sensors the encapsulation was done with epoxy glue.

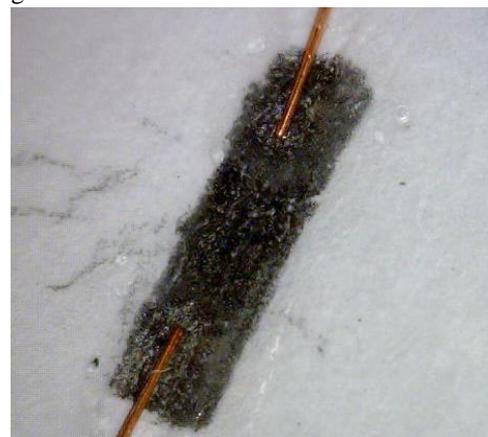


Fig. 2: Digital photograph of piezoresistor graphite on paper.

In order to determine the chemical composition, the X-ray fluorescence analysis was performed by dispersive energy

(see Table I) in a SPECTRO spectrometer model MIDEX.

Table.I: Chemical composition of the graphite deposited on paper

Element	Concentration (%)	Symbol
Silicon	2,37	Si
Sulfur	54,91	S
Calcium	14,21	Ca
Titanium	8,76	Ti
Rhodium	2,21	Rh
Paladin	5,76	Pd
Silver	3,79	Ag
Cadmium	3,60	Cd

The X-ray diffraction technique was used for the determination of the atomic structure in equipment brand Philips model X'Pert -MPD. The resulting Diffractogram can be seen in Figure 3.

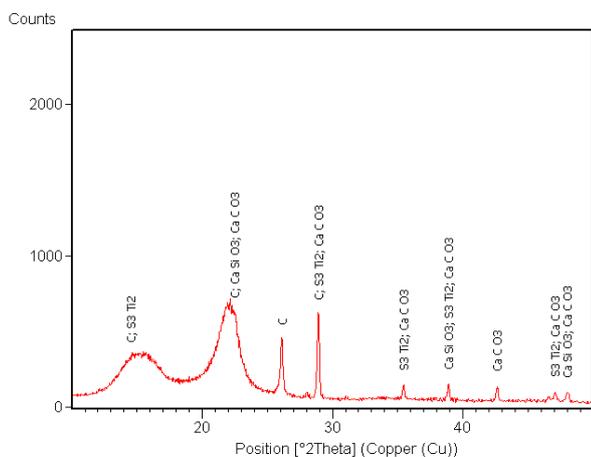


Fig. 3: X-ray diffraction for sample of graphite deposited on paper.

The microscopy was used to observe how the deposition of the graphite occurs in the polymeric substrate. Fig. 4 shows an optical microscopy of the graphite film on paper substrate.

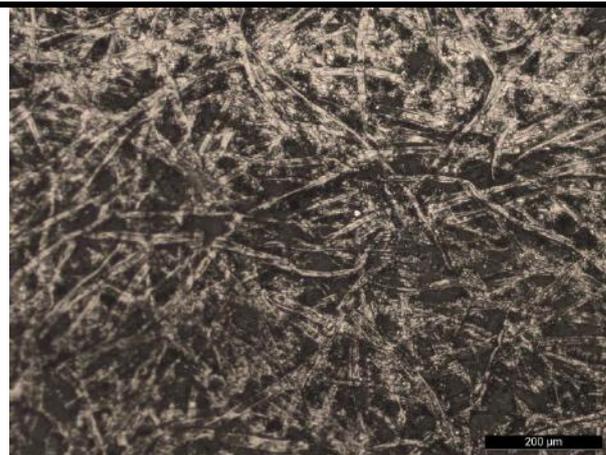


Fig. 4: Optical microscopy for graphite on paper. Increase by 10x.

Figure 5 shows in more detail aspects of the graphite matrix and cellulose fibers of the substrate when analyzed by digital microscopy.



Fig. 5: Digital microscopy for graphite on paper. 150x magnification.

Figure 6 obtained with SEM reveals the graphite flakes deposited on the substrate through the technique of mechanical exfoliation of graphite - GoP.

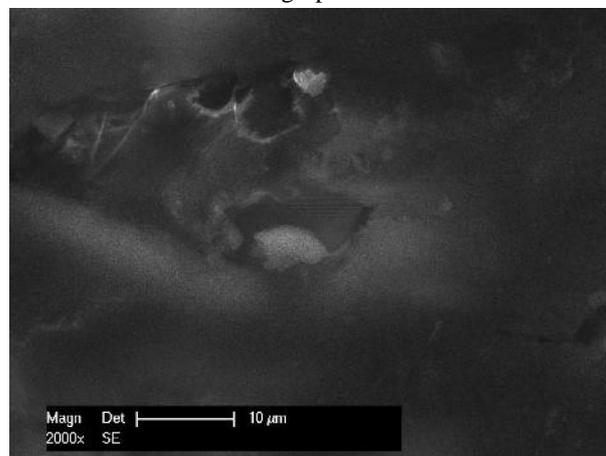


Fig. 6: SEM for graphite on paper. Increase of 2000x.

Figure 7 shows the dimensions of the graphite flakes constituting the deposited film in this piezoresistor manufacturing process.

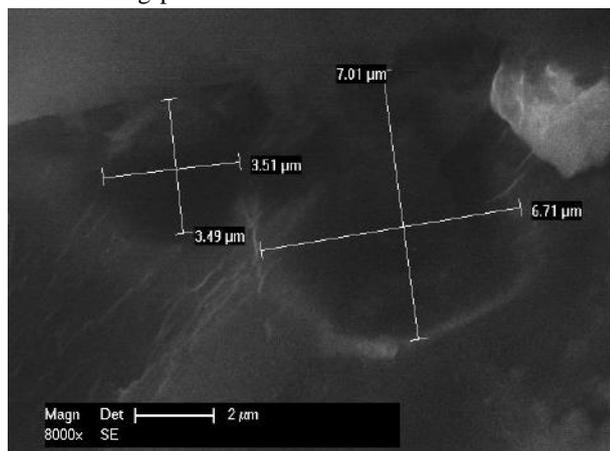


Fig. 7: Scanning electron microscopy – SEM for graphite on paper, determination of the size of the flakes. Increase of 8000x.

Figure 8 shows the EDX analysis for the graphite slides. The images were obtained with a Keygen VK-X digital microscope and Philips scanning electron microscope model XL 30 ESEM. This analysis shows the composition of the graphite used to manufacture the sensor elements.

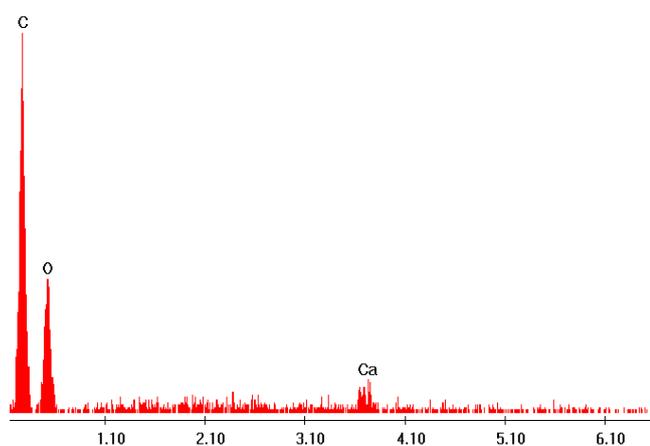


Fig. 8: EDX analysis for graphite flakes

III. RESULTS AND DISCUSSIONS

The characterizations made for the graphite deposited on a polymeric substrate, A4 paper, made it possible to better understand and know the conductive material and, mainly, how the substrate / conductive material interface is given. X-ray fluorescence (XRF) made it possible to identify several chemical elements, and Sulfur and Calcium are present in greater amounts. The other identified chemical elements are residues from the substrate manufacturing processes. X-ray diffraction analysis allowed to identify a series of compounds mainly hexagonal carbon. Through optical and digital

microscopy it was possible to visualize and identify the graphite sheets adhered between the fibers of the paper as well as other particles considered undesirable impurities. An EDX analysis of these impurities identified them as being calcium particles, a result consistent with the other analyzes performed (DRF and XRD). In addition, the scanning electron microscopy allowed measuring the size of the graphite slides, which vary between 3.5 μm and 7 μm . EDX analysis can confirm the composition of the constituent particles of the film, carbon being the main component found. The results are consistent with the literature review, allowing reliable and reliable data for the production of piezoresistive graphite sensors.

IV. CONCLUSION

The results found in the performed analyzes are consistent with the literature review, allowing reliable data for the production of piezoresistive graphite sensors using a low cost process when compared to silicon technology.

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