Project 1 – **Piezoresistive effects in nanocrystalline carbonic films and their application as foil strain gauges** (**PIEZOCARB**)

Stage III-1: The fabrication and iterative optimization of piezoresistive tensometric sensors based on functional laboratory testing and industrial application's requirements. Continuous design, technological workflow and process optimizations.

1.1. The Fabrication of NCG-based foil strain gauge.

For this phase of the project, nanocrystalline graphite (NCG) was synthesized directly onto \sim 35 µm thick copper (Cu) foils. After the NCG growth, the carbonic films were transferred on flexible kapton foils of different thicknesses for the purpose of mechano-electrical testing. The nanocrystalline structure of the as-grown films was confirmed through Raman investigations.



Figure 1. Raman spectrum of a NCG thin film grown on a Cu foil for 2 hours. The characteristic bands in the spectrum correspond to the bands of NCG grown on SiO_2 .

The hydrocarbon radicals obtained from the dissociation of the CH_4 molecules inside the plasma, have a much higher diffusion speed on the Cu metallic surface than on SiO₂, thus increasing the NCG growth rate by 3 times. This is confirmed by scanning electron microscopy.

The adhesives and flexible foils used for the samples are presented in Table 1.

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Sample denomination	R_1, R_2	P_1, P_2	Ρ3, c, γ	a, I, II, α, β, M ₁ , M ₂	b
Adhesive	Epoxy resin	Cyanoacrylate	Epoxy resin	Epoxy resin	Cyanoacrylate
Substrate	Kapton (125 µm)	Kapton (25 μm)	Kapton (50 μm); Tombak (100 μm)	Kapton (50 μm); Kapton (125 μm)	Kapton (50 μm); Kapton (125 μm)

Table 1. Sample denomination with respect to the used adhesives and flexible foils.

In Figure 2, three of the samples prepared for the mechano-electrical characterizations can be observed.



Figure 2. Photographs of the sensitive structures prepared for piezoresistive testing: (a) R1, (b) P1 and (c) β

The NCG-based piezoresistive samples where each electrically characterized during a 0.5 mm elongation of the flexible foil. All samples were initially investigated during one elongation-relaxation cycle, after which they were subjected to 200 more cycles and investigated again during a final additional cycle.



Sample: M1 NCG thickness: 5 nm Substrate: Kapton (50 μm) + Kapton (125 μm) **GF = 2.11**

Sample: II NCG thickness: 10 nm Substrate: Kapton (50 μm) + Kapton (125 μm) **GF** = **1.84**

Sample: α NCG thickness: 17 nm Substrate: Kapton (50 µm) + Kapton (125 µm) **GF = 2.33**

Sample: a NCG thickness: 52 nm Substrate: Kapton (50 μm) + Kapton (125 μm) **GF = 15**

Sample: P2 NCG thickness: 156 nm Substrate: Kapton (25 μm) **GF = 19**

Sample: R2 NCG thickness: 1250 nm Substrate: Kapton (125 μm) GF = 236

1.2. The fabrication of piezoresistive tensometric sensors based on carbonic aerogel (CAG).

The graphene-based nanocomposite aerogels were synthesized from graphene oxide (GO) and polymer solutions by mixing specific precursors. Two types of aerogels were prepared: (1) Graphene/polyurethane (G/PU) nanocomposite aerogel, by using GO and an aqueous polyurethane dispersion; (2) Graphene/hydroxyethyl methyl cellulose (G/HC) nanocomposite aerogel, by using GO and an aqueous solution of hydroxyethyl methyl cellulose.



Figure 3. Lyophilized and stripped G/PU and G/HC aerogel samples.

The resulting aerogels were diced in 2 cm x 2 cm pieces and electrically contacted with 2 coppered sticlotextolite plates. On the plate's surface, a conductive layer of silver (Ag) paste was applied as shown in Figure 4. The Ag paste was left to polymerize at room temperature for 24 hours.



Figure 4. CAG-based piezoresistive device.

The fabricated CAG devices were each subjected to electrical investigations during 2 mm mechanical compression tests. Firstly, the samples were characterized during one compression-relaxation cycle, after which they all underwent 200 more cycles and characterized again during one final additional cycle. The experimental data show that the G/PU CAG samples prepared with different GO:PU mass ratios are either not conductive or their electrical resistance increases exponentially after 200 compression-relaxation cycles, thus being impractical for the studied application. Of the G/HC CAG samples, only the ones with a mass ratio of GO:HC=5:2 allow for reproducible results and a compression of up to 50 % of the initial volume. The piezoresistive investigations for the G/HC samples with a mass ratio of 5:2 are presented in Figure 5. The calculated gauge factor in this particular case was GF=214.



Figure 5. Electrical characterization of a G/HC CAG sample during a compression-relaxation cycle over 2 mm: before (bottom) and after (top) 200 cycles.