



# Electro-deposition of graphene on aluminium open cell metal foams



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## ABSTRACT

This manuscript deals with the electro-deposition of graphene on open cell aluminium foams. It was tried to combine the superior features of stiffness and conduction of the graphene to aluminium foam. Metallic foams are highly porous materials, which present complex structure of three-dimensional open cells. This aspect causes strong limitations in mass transport due to electro-deposition technology. A novel method of electro-deposition at low temperature was developed. First, experimental tests were performed to study the influence of the operational parameters on the overall performance of the coated aluminium foams. Each sample was electro-deposited in three steps, or phases: first copper, then graphene and copper and finally copper again. Second, a mechanical and thermal characterization of the produced samples was led through a mechanical test machine and conductivity tests. Finally, on the basis of the experimental results, a semi experimental model of the evolution of deposition process of graphene was proposed. The experimental findings revealed that the manufactured metal foams were characterized by a higher mechanical resistance and thermal conductivity and low process costs, making these materials very promising in many technological fields.

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## 1. Introduction

Since it was discovered in 2004, graphene was considered an exceptional material and has been attracting extensive scientific interest from both experimental and theoretical communities [1,2]. Thanks to its particular planar structure, graphene is characterized by unique properties, such as excellent chemical inactivity, high electrical conductivity, high optical transparency, good thermal stability, and extraordinary flexibility [3–6]. Since it significantly improves thermal and electrical conductivity [7–9], the range of applications where it can be exploited is very wide [10]. Graphene can substitute silicon in processors and solar panels [11,12] or, combined in a sandwich structure, it can work like a transistor. Furthermore, graphene can be exploited as electrodes for ultra capacitors, anode material for lithium batteries, thermally conductive films and coatings, films or coatings for EMI shielding, additive for composites of metal origin [10,13].

Several deposition methods have been proposed to fabricate two-dimensional graphene sheets on conductive substrates, such as chemical vapour deposition and thermal decomposition [14–18]. However, most of these are burdensome for the assembly of graphene layers because they employ high temperatures and complex instrumentation [14]. In this work a novel method of

electro-deposition at low temperature was developed. It was tried to combine the superior features of stiffness and conduction of the graphene to aluminium foam electrodeposited with copper. Metal foams, in fact, combine good mechanical characteristics with low weight. Furthermore, their open cell structure has interesting functional properties that can be exploited in many technologies such as heat exchangers, water cleaning and orthopaedic applications. Moreover, a semi experimental model of deposition of graphene was proposed, based on the experimental results and theoretical models.

Before electro-deposition, all the base aluminium foams were treated by means of sandblasting, in order to eliminate the oxide layer that covers the surfaces. Graphene has been co-electro-deposited together with copper exploiting a particular solution that will be described in the following paragraphs and of course copper anodes. While the values of current exploited for the electro-deposition of the cupric layer only follows what is indicated in [19,20], the deposition of graphene was carried out using 0.01 and 0.02 A, for at least  $3.6 \times 10^3$  s; in fact higher values of this parameter could activate the carbon in graphene.

The mechanical and thermal characterization of the produced samples was led through compression and conductivity tests, in the same way as [20]. Unfortunately, the volatility of graphene made impossible to execute the thermal conductivity test immediately once it was deposited, but it was carried out after the third phase.

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## 2. Experimental procedure

Metal foams of aluminium alloy 6101 T6, produced by “NANESA s.r.l.”, was used in the experimental tests. The employed foams were produced through the method of replication of polymeric path. All the samples were manufactured with a parallelepiped shape with dimensions  $20 \times 20 \times 30 \text{ mm}^3$ . The density of the specimens is in the range from  $160 \text{ kg/m}^3$  to  $220 \text{ kg/m}^3$ . The average pore size is  $10 \text{ ppm}$  ( $2.237 \times 10^3 \text{ }\mu\text{m}$ ).

Since graphene is very volatile, occurred the necessity to electrodeposit twice aluminium samples with copper: a cupric layer covers the base foam, and a second cupric layer prevents graphene to leave the specimen. So each sample was electro-deposited in three steps, or phases: first copper, than graphene and copper and finally copper again. Two different kinds of solution were exploited in the three different phases of electrodeposition. In the first and last ones, an acid solution based on copper sulphate was chosen as electrolytic bath. Its composition consisted of  $1.25 \text{ M CuSO}_4$ ,  $0.61 \text{ M H}_2\text{SO}_4$  and  $\text{Cl-50 ppm}$  [20]. The second phase instead was characterized by a basic solution of  $1.25 \text{ M CuSO}_4$ ,  $\text{Cl-50 ppm}$  and GNP, provided by NANESA s.r.l. (Italy). GNP consists of small stacks of graphene obtained by exfoliation of expanded graphite. It is characterized by a carbon content of 98% and less than 1% of residual acid. Its average flake thickness is  $10 \text{ nm}$  (30 layers), with an average particle size of  $5\text{--}50 \text{ }\mu\text{m}$  and specific surface area of  $30 \text{ m}^2/\text{g}$ . In Fig. 1 are shown SEM and TEM images of graphene.

Both the baths were kept in agitation by mean of a magnetic agitator, located inside the electrolytic cell. The agitation was set at  $3.5 \text{ rpm}$ . For the acidic bath a current of  $2.3 \text{ A}$  was exploited, while a current of  $0.01 \text{ A}$  or  $0.02 \text{ A}$  was used in the graphene bath.

The consumable anode was the used for all three phases. Its shape is a hollow tubular, completely made of copper. The specimens were placed in the center of the anode and completely immersed in the electrolytic bath [20].

A surface preparation was planned for the substrate material, in order to eliminate the oxides that normally cover the surfaces of aluminium. This procedure was demonstrated that speeds up the process of electrodeposition and enhances the quality of the coverings [19,20]. It was carried out using a sandblasting machine with tiny glass spheres, at the pressure of  $3 \text{ bar}$ . It was paid attention to not let pass to much time between the end of a phase of the electrodeposition process and the begin of the following one. In this way the integrity of the surface of the coating was prevented by possible alterations caused by the interaction of the copper with the atmosphere. When this was not possible, the samples were put under vacuum. In fact, the presence of these alterations, for

example the oxidation of the copper surface or the formation of any other possible thermal barriers, could have modified the results of the thermal tests.

Compression tests were leaded on each samples in a MTS Alliance RT50 testing machine with a  $10 \text{ kN}$  load cell, setting a constant deformation rate of  $5 \text{ mm/min}$ .

Conductivity tests were leaded on each sample after each phase, because of the volatility of the graphene. In the experimental device used the sample is tightly held between two Peltier cells that impose a thermal flux through the foams. Two thermocouples evaluate the gap of temperature between the bases of the foam [20]. In this way is possible to calculate  $\lambda * S$ , where  $\lambda$  is the thermal conductivity of the foam and  $S$  is the surface exposed. The enhancement of this parameter was evaluated relatively to the phases of the same sample.

## 3. Theoretical model

The semi-experimental models that will be presented in this work have their fundamentals in a model that is fully explained in a previous work [20], and it is based on Faraday law.

$P\%$ , is a parameter chosen to assess the standard of the electrodeposition. It is a mass ratio: the amount of substance electrodeposited in a specific phase divided by mass of the previous phase. Exploiting the Faraday law,  $P\%$  can be expressed as:

$$P\% = \frac{(MM_{\text{Cu}} * n_e / r * V_i * 2) * (N_a * i * t / N_a * i * t)}{(MM_{\text{Cu}} * 60 / N_a * 2e) * (i * t / \rho * V_i)} \quad (1)$$

where  $MM_{\text{Cu}}$  is the molar mass of copper,  $n_{\text{Cu}}$  is the number of moles of copper electrodeposited,  $\rho$  is the density of the sample,  $e$  is the electric charge of an electron,  $n_e$  is the number of moles of electrons exchanged,  $t$  is the time (in minutes),  $i$  is the electric current and  $N_a$  the Avogadro's number. This expression can be used only for copper electrodeposition, as it can be seen observing the quantity displayed. Moreover it is noted that the first part of the second member of this equation is constant and assumes value  $1.978 \times 10^{-5} \text{ kg/A min}$ .

Thermal conductivity ( $\lambda$ ) was established exploiting the expression for heat exchange by conduction:

$$P = (\lambda / \delta) * S * \Delta T \quad (2)$$

where  $P$  is the thermal power exchanged,  $\delta$  is the distance between the thermocouples,  $S$  is the exchange surface and  $\Delta T$  is the gap of temperature between the bases.

Since it was impossible to correctly quantify  $S$ , due to the complex nature of the foam surfaces, the information on thermal

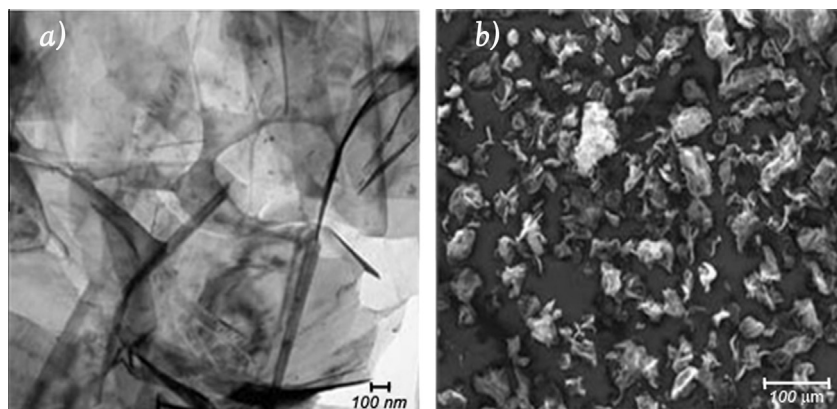


Fig. 1. (a) TEM and (b) SEM micrographs of graphite nanoplates.

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