SYNTHESIS OF GRAPHITE-GRAPHENE AEROGELS FOR THERMAL ENERGY STORAGE APPLICATIONS AND SENSORS

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The use of graphite and graphene oxide in combination with phase change materials is a viable choice for enhancing the thermal conductivity of latent heat storage materials [1, 2]. Graphene-based aerogels attracted a lot of interest due to their fascinating properties, such as high mechanical strength, electrical conductivity, adsorption capacity and thermal resistance [3], which allow them to be used in a variety of applications like energy storage, supercapacitors and gas sensing [4].

The present study includes the preliminary results regarding the functionalization of graphite, and the chemical synthesis of graphite-graphene aerogel. The obtained carbon materials were studied using X-ray diffraction and scanning electron microscopy.

Graphite-graphene aerogel synthesis

The incorporation of surface oxygen into graphite can be achieved through a variety of methods. The *functionalization of the graphite* surface by acidic oxidation is the starting point for the vast majority of reports [5].

H_2SO_4 and HNO_3 were mixed in a 3:1 ratio to create a solution with a final volume of 160 mL. Graphite powder (2 g) was added to this solution and the mixture was heated to 70 ^{o}C with stirring for 8 h. After the oxidation process, the excess of the oxidizing agent was removed by centrifugation, followed by repeated washing, decanting and further centrifugation of the functionalized graphite in bidistilled water until the pH of the supernatant exceeded 5 (the concentration of suspension was 73.4 mg/mL).

The graphene oxide (prepared by the modified Hummers' method) was mixed with the functionalized graphite, in a 1:100 ratio, after which the suspension was immersed in liquid nitrogen for 2 minutes. The sample lyophilization occurred at a gas pressure of 2 Torr, and after 20 hours the gas pressure dropped to about 960 mTorr. At this point the graphite-graphene aerogel can be clearly seen inside the quartz tube (Figure 1). Finally, the aerogel was heat-treated under vacuum (GSL-1500X Vacuum furnace, MTI Corporation) at 800 ^{o}C (2 ^{o}C/min) for 60 minutes, during which the aerogel fragmented.

To also study the thermal reduction of the graphene oxide, this was heated under the same conditions. The results are compared in Figures 2 and 3.

The crystalline structure of the samples was characterized by X-ray diffraction measurements (XRD, PW 3040/60 X'Pert PRO, Cu-Kα radiation), the results being presented in Figure 2.

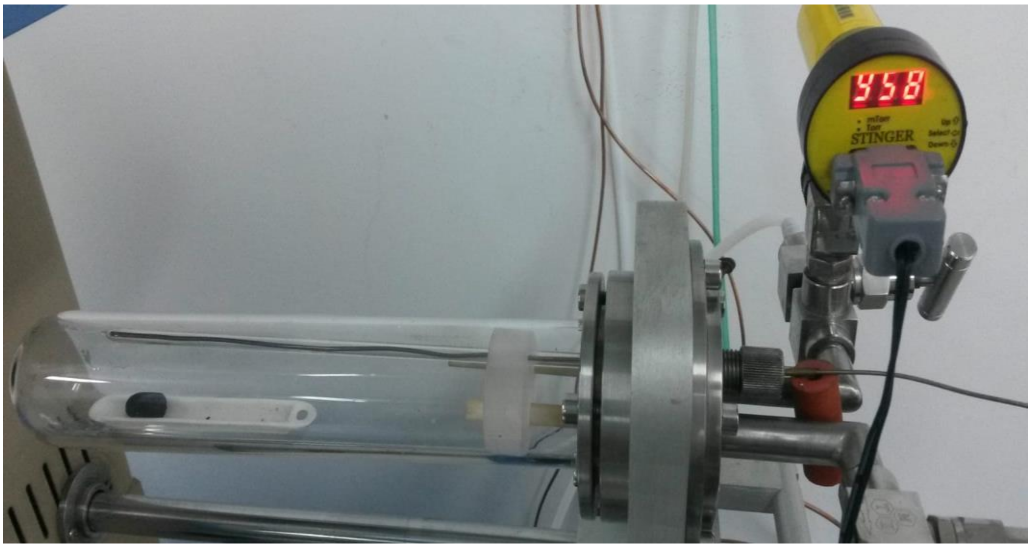

Figure 1. The graphite-graphene aerogel during its synthesis.

The crystallite size was determined using the Scherrer equation, by considering the diffraction peak located at a 2θ of 26.6 degrees, having the Miller indices (002). The obtained values were 103 nm for graphite and, respectively, 49.5 nm for the graphite in aerogel.

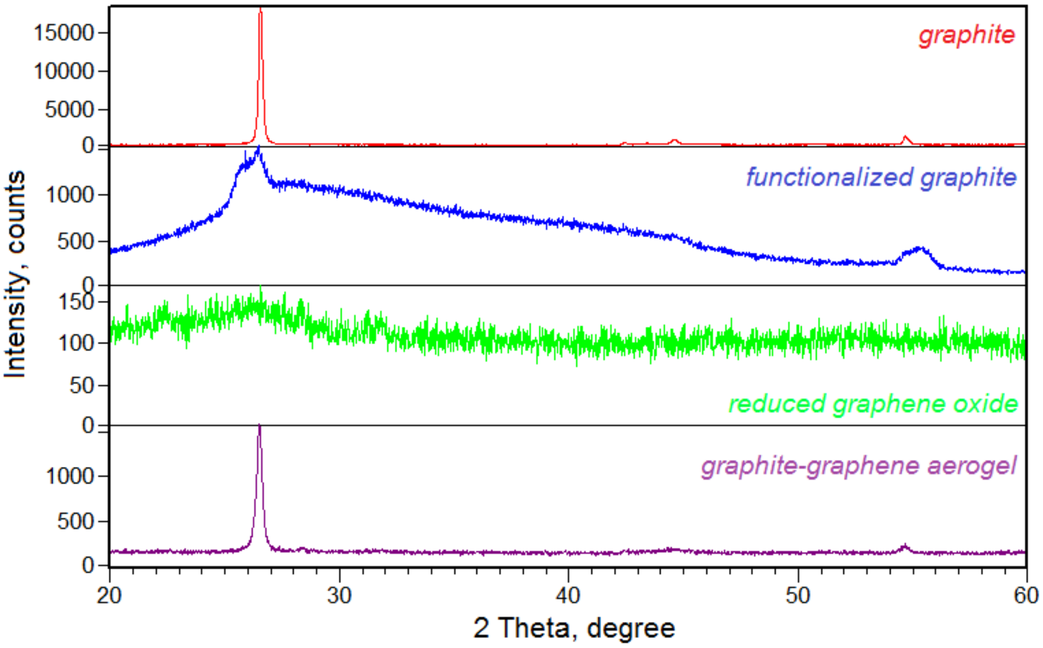

Figure 2. XRD patterns of graphite, functionalized graphite, reduced graphene oxide, and graphite-graphene aerogel.

Thus, the oxidizing mixture leads, on one hand, to the fragmentation of graphene sheets from the graphite network, and on the other hand to the intercalation of functional groups across the carbon atom layers, which is followed by the separation of sheets. The functionalized graphite is completely reduced at 800 °C.

The morphology of the samples, investigated using the scanning electron microscope (FEI Inspect S), is presented in Figure 3.

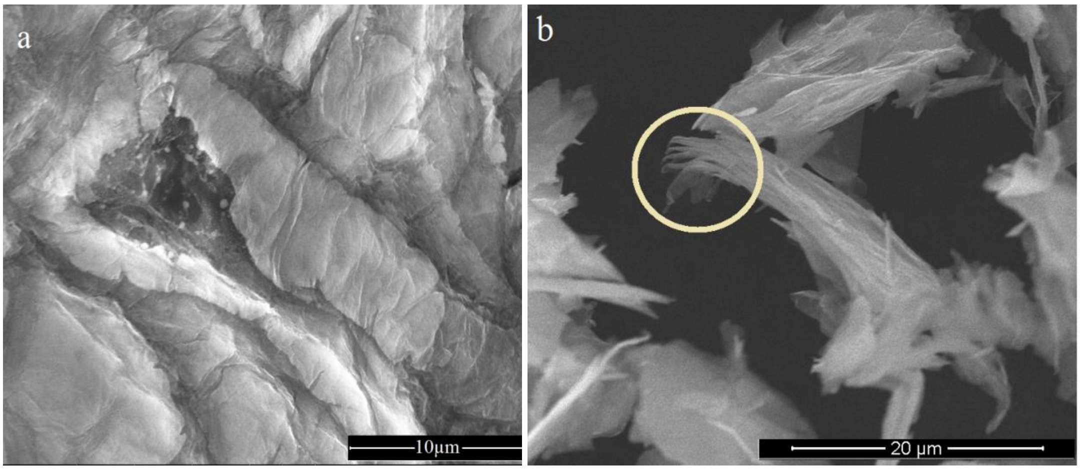

Figure 3. SEM images of (a) reduced graphene oxide and (b) graphite-graphene aerogel.

The aerogel has an interconnected porous network, the pore walls consisting of layers of stacked graphene and graphite sheets. In the SEM images (Figure 3b) we can observe the existence of individual graphite sheets, produced during the loss of oxygen through high-temperature reduction, which confirms the XRD data.

Acknowledgments

This work was supported by a grant of the Romanian Ministry of Research and Innovation, CCCDI - UEFISCDI, project number PN-III-P1-1.2-PCCDI-2017-0619 / research contract 42/2018 “Nanostructured carbon materials for advanced industrial applications”, within PNCDI III.

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