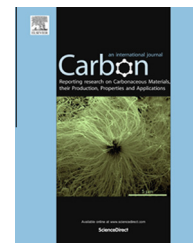


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Carbon periodic cellular architectures



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ABSTRACT

The first carbon periodic cellular architectures derived from 3D printing, in the form of new tetrakaidecahedra meshes, are reported and investigated in this paper. They were prepared in hydrothermal conditions by a template method based on polymer periodic structures of the same geometry, and fabricated by a 3D printer using photocurable resin. Several formulations based on resorcinol–formaldehyde were tested, and the best ones were those using low concentrations of resorcinol at 150 °C in a pressurised solution of nickel nitrate. After pyrolysis at 1000 °C, catalytic graphitisation was demonstrated by TEM, XRD and Raman studies. The higher was the amount of nickel, the higher was the resultant graphitisation level. Mechanical tests were also carried out on such extremely lightweight periodic carbon structures, showing that these new materials present a much higher modulus than carbon foams of similar bulk densities.

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1. Introduction

Highly porous, cellular, carbon materials can be prepared by different techniques, e.g. by foaming [1–8], leaching of sacrificial template particles [9], emulsion- or bubble-templating [10–17] of carbon precursors, among others, and hard-templating using polymer preforms [18–23]. In most cases, a polymer is used as the carbon source, and once the cellular structure of the material is stabilised, a pyrolysis step has to be carried out. The polymer can be pyrolysed directly or not, depending on its chemical nature. Thermoplastics indeed lose their shape when heated, therefore need to be stabilised beforehand, and most of them have a nearly zero carbon yield. In contrast, thermoset resins, especially phenolic ones, can be directly converted into carbon with a very satisfactory

yield, close to 50% [24]. When the polymer has a too low carbon yield, whether thermoset or not, an impregnation is required for getting enough material after pyrolysis. This impregnation allows coating the cellular structure of the polymer used as template, thereby stabilising its shape, as well as providing the necessary source of non-volatile carbon.

As far as the present authors know, 3D printing has never been used so far for obtaining new carbon structures whereas it has been already shown to be an excellent method for preparing new, ordered, ceramics [25–27]. Only one rapid prototyping method has been used so far for preparing 3D scaffolds based on the extrusion of a paste composed of graphite with a polymer binder [28], which is a completely different method than that reported here. Additive fabrication is indeed not directly applicable to carbon, as pure carbon can

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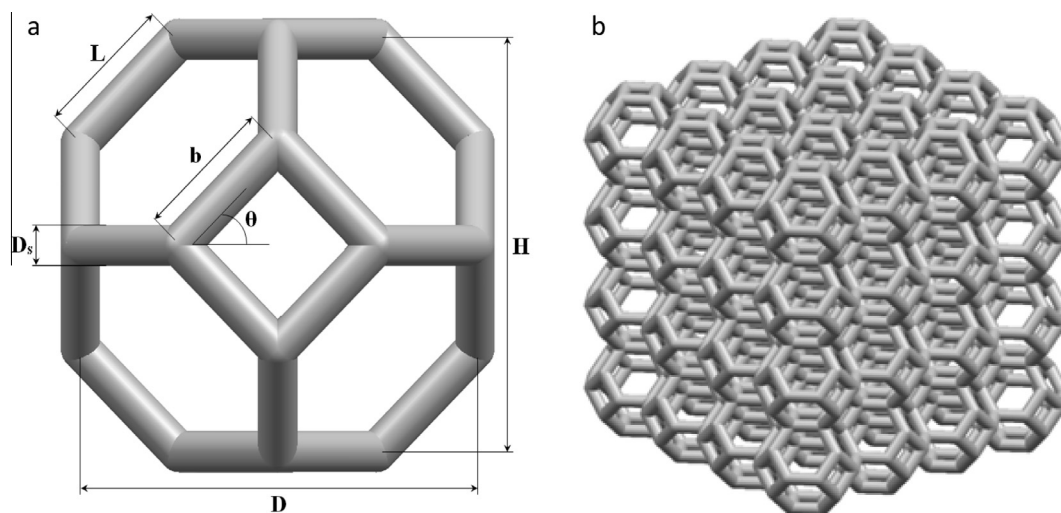


Fig. 1 – (a) Tetrakaidecahedron cell parameters: strut diameter D_s , edge length L , height H , cell width D , and strut inclination $\theta = 45^\circ$ for a Kelvin cell. (b) Schematisation of the $4 \times 4 \times 4$ cells numerical model.

obviously neither been molten, crosslinked nor polymerised after layer-by-layer deposition by a 3D printer. Therefore, either the 3D structure has to be pyrolysed or, better, used as a template for another carbon source of higher yield. The polymer used in the present work was indeed a photocurable resin which almost completely evolved into gases by direct pyrolysis. In order to prepare the carbon replica of printed 3D structures, a template synthesis using a hydrothermal route has been used. We show here how such a process was optimised, especially through the use of catalytic graphitisation, and we present a few properties of these new materials. Tetrakaidecahedra meshes have been chosen for the present studies, but any other complex structure, periodic or not, might be treated in the same conditions.

2. Experimental

2.1. Materials

2.1.1. Polymer tetrakaidecahedra periodic cellular structures
The lattice structures were generated by a MATLAB code (MathWorks, Natick, MA, USA). Each model consisted of a 3D distribution of points (nodes) knowingly connected. This tool can generate any cell type with desired cell sizes depending on the nodes distribution and their connection. Each connection was modelled by two vertices and a line between them (strut axis). Different parameters such as strut's diameter and elongation can be chosen to design a numerical model.

3D-printed polymer templates were designed by computer-aid design (UGS NX 8.5, Siemens, Germany). The numerical model consists of a $4 \times 4 \times 4$ array of tetrakaidecahedra as represented in Fig. 1. The tetrakaidecahedron structure is especially interesting as it is widely accepted to represent the random foam structure while it is parametric. On the other side, it is an engineered structure, therefore it can be placed as the border between random foams and designed lattices.

Templates with $D = H = 3, 4, 5$ and 7.5 mm were 3D-printed (Eden 260 V 3D, Objet, Rehovot, Israel) with photosensitive resins (Fullcure 705 and 850 VeroGray, Object Ltd.), which were composed of acrylics, urethanes and epoxies. Resins were compounded with a photo-initiator that triggered the polymerisation under UV light. The 3D printing was performed with the maximum resolutions of $16 \mu\text{m}$ and $42 \mu\text{m}$ for vertical and planar directions, respectively. Fig. 2 shows the corresponding periodic tetrakaidecahedra architectures as directly obtained from the 3D printer. The cell size and the struts thickness were modified according to the values gathered in Table 1.

In the present work, only carbon materials based on mesh A (see again Table 1) are described, but experiments showed that the same kind of structures can be easily prepared in the same conditions from the three others structures reported in Table 1 (see below). Not only changing the strut thickness at constant cell size and geometry (and therefore changing the porosity, all other things being equal) was possible, see Fig. 3(a), but totally different periodic structures were also available for preparing highly ordered and porous carbons, see Fig. 3(b). However, as the present paper is a first report of carbon architectures prepared by 3D printing, only carbons prepared from lattice A (see again Table 1) were described here.

2.1.2. Carbon tetrakaidecahedra periodic cellular structures
Different tests were carried out for determining the method leading to the most regular and flawless carbon structures. The first trial consisted in doing a simple and direct pyrolysis of the polymer mesh in a flow of very pure nitrogen up to 1000°C at a heating rate of 1°C min^{-1} . As expected from the nature of the photocurable resin, principally based on various acrylates and on acrylic monomers and oligomers¹, the structure was completely lost and only a very low amount of carbon, around 1% of the initial mass, remained.

¹ Fullcure 850 VeroGray, Safety data sheet, version 2. Object Geometries Ltd., 2008.

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