Carbon MEMS

Synonyms
C-MEMS, Carbon MicroElectroMechanical Systems

Definition
A process used to fabricate miniaturized glass-like carbon structures through the carbonization of previously shaped organic precursors.

Overview
Carbon MEMS combines different patterning techniques with carbonization to derive miniaturized glass-like carbon features from an organic precursor. Since glass-like carbon is a brittle material, the carbonization of an already patterned precursor compares advantageously against subtractive manufacturing of glass-like carbon bulk. To date, the most common precursor patterning technique used in C-MEMS has been conventional photolithography, but many other techniques could be used. These cover a wide range: from relatively inexpensive like electrospinning, stamping, molding, polymer machining and casting; to Next-Generation Lithography (NGL) techniques such as NanoImprint Lithography (NIL) and electron beam lithography. The choice of precursor patterning technique is dictated by the quality, complexity and final dimensions of the desired carbon part. Shrinkage of the precursor structure occurs during carbonization and must be accounted for. However, the existence of commercial high-quality precursors, like the negative photoresist SU-8, and availability of standardized fabrication tools make the fabrication process and the dimensional control highly reproducible.

Historical Background
Carbon can be found as a number of allotropes including diamond, lonsdaleite, buckminsterfullerenes, graphene, carbyne, graphite, carbon nanofoams, diamond-like carbon, amorphous carbon and those carbons derived from the pyrolysis of organic materials, better known as glass-like carbons. Different precursors to obtain glass-like carbon may be used, including phenolic resins, polyfurfuryl alcohols, cellulose, polyvinyl chloride and polyimides.

The first documented modern derivation of glass-like carbon from an organic resin (phenolic in this case) appears to be from 1915, when Weintraub and Miller in Massachusetts, USA derived disks of a "very bright, shiny looking carbon with hardness equal or greater than 6 on the Mohs mineral scale". These disks were then used for better microphones in telephone transmitters. Their fabrication protocol featured slow heating of a hardened resin to a temperature close to 700 °C in about one week, followed by firing at temperatures from 800 to 1100 °C in just a few hours. The advantage of patterning an organic precursor, rather than carbon bulk, was realized since then [1]. A sustained flow of publications on glass-like carbon did not begin until 1962, when Davidson at the General Electric Co. in Kent, England derived glass-like carbon from cellulose [2] and Yamada & Sato, at the Tokai Electrode Manufacturing Co. in Nagoya, Japan, published preliminary characterization results of carbon derived from organic

polymers [3]. They named such carbon "glassy carbon", a term that was later registered as a trademark. Around the same time, in 1963, Lewis, Redfern and Cowlard at the Plessey Company in the United Kingdom postulated the use of glass-like carbon, named "vitreous carbon" by the authors and later registered as trademark as well, as an ideal crucible material for semiconductors. Later that year, Redfern and Greens disclosed several production processes to derive this "vitreous carbon" in a patent [4]. In 1965, the advantages of glass-like carbon electrodes for voltammetry and analytical chemistry were characterized by Zittel and Miller, from Oak Ridge National Laboratory in the US, using "glassy carbon" from the Tokai Electrode Manufacturing Co. [5]. In 1967, Cowlard and Lewis published a detailed description of the properties of "vitreous carbon", the fabrication process and its potential applications [6]. The decade of 1970 brought a significant interest on the use of glassy carbon as a material for different implants and biomedical instrumentation, and also witnessed an explosion of the interest on glassy carbon by the analytical and electrochemistry communities, which still remains strong. In 1971, a structural model for glass-like carbon was postulated by Jenkins and Kawamura [7]. This model is up to this date the only one capable of explaining most of the experimental results obtained with glass-like carbon.

Carbon derived from organic polymers by pyrolysis in inert atmosphere has been historically known by three different names: "vitreous carbon", "glassy carbon" and glass-like carbon. Although highly referenced in implant-related publications during the 1970s, the term "vitreous carbon" started to fall in disuse by the end of that decade. "Vitreous carbon" is now better identified with Reticulated Vitreous Carbon (RVC), a material introduced in the late 70s by Chemotronics International Inc. from Ann Arbor, Michigan. The commercialization of Tokai's "glassy carbon" electrodes targeting the electrochemistry market made "glassy carbon" the term of preference for the electroanalytical chemistry community to refer to glass-like carbon. In 1995, the IUPAC (International Union of Pure and Applied Chemistry) defined glass-like carbon as the material derived by the pyrolysis of organic polymers and recommended that the terms "Glassy carbon" and "Vitreous carbon", which had been introduced as trademarks, should not be used as synonymous for glass-like carbon.

From the microfabrication standpoint, in 1983 Lyons et al. at AT&T Bell Laboratories published their work on the use of photodefined novolac resist patterns as precursors for carbon microstructures [8]. The drive behind this effort was finding an alternative to carbon films deposited by chemical vapor deposition. Miniaturized glass-like carbon 3D structures were not reported until the late 1990s by Schueller and co-workers at Harvard University. In their process, polydimethylsiloxane (PDMS) molds were fabricated using soft lithography and then used to pattern furfuryl alcohol-modified phenolic resins and phenol-formaldehyde resins, which were subsequently carbonized [9]. By 2000, Kostecki and colleagues were obtaining further results on the fabrication of planar carbon microelectrodes from positive photoresists to study the influence of their geometry in their electrochemical response [10]. Such work ignited the use of Pyrolyzed Photoresist Films, or PPF, in fields such as electrochemistry. In 2002, the derivation of carbon from negative photoresists was reported by Singh et al. [11]. The obtained carbon showed higher electrical resistivity and vertical shrinkage than the one synthesized from positive resists. In 2004, structures with aspect ratios higher than 10 were reported by Wang et al. at the University of California, Irvine (UCI) [12, 13]. These authors coined the term Carbon MEMS. Since

then, most of the work identified as Carbon MEMS has used SU-8 as carbon precursors and photolithography to shape 3D structures. Other precursors include resorcinol-formaldehyde gels [14] and cellulose. Starting in 2009, electrospinning of SU-8 and PAN (polyacrylonitride) has been developed towards obtaining carbon fibers in the nanoscale [15, 16].

Material Properties of Carbon MEMS

The carbon obtained in a Carbon MEMS process resembles glass-like carbon. Although the choice of precursor and patterning technique has an impact on the properties of the resultant carbon, the following properties can serve as a starting reference. Although a consensus on the crystalline structure of glass-like carbon has not been reached to this date, the most widely accepted model [7] is the one that considers this type of carbon as made up of tangled and wrinkled aromatic ribbon molecules that are randomly cross-linked by carbon-carbon covalent bonds. The ribbon molecules form a networked structure, the unit of which is a stack of high strained aromatic ribbon molecules. Such structure of crystallites reflects the features of thermosetting resins structure which are commonly used as precursors for glass-like carbons. Glass-like carbon is widely considered as impermeable to gases and extremely inert, with a remarkable resistance to chemical attack from strong acids and other corrosive agents such as bromine. The material will react with oxygen at temperatures above few hundred degrees centigrade, making oxygen plasma a favored etching process. Glass-like carbon has a hardness of 6 to 7 on Mohs' scale, a value comparable to that of quartz. Its density ranges from 1.4 to about 1.5 g·cm^{-3}, compared to 2.3 g·cm^{-3} for graphite. X-ray diffraction studies have shown that glass-like carbon presents an extremely small pore size, ~50 Å, of a closed nature, and that has an amorphous structure [31-38]. Glass-like carbon features a coefficient of thermal expansion of 2.2-3.2 X 10^{-6}·K^{-1} which is similar to some borosilicate glasses. Its Young Modulus can vary between 10 and 40 GPa. An indicative electrical resistivity of glass-like carbon is 1 X 10^{-4} Ω·m when the carbonization temperature is as high as 900 °C. The electrical resistivity drops further as the temperature increases over this value or the dwell time at temperatures below it increase [17, 18]. One of the reasons glass-like carbon is preferred by electrochemists is the fact that it has a wider electrochemical stability window than platinum and gold.

Photolithography as toolbox for Carbon MEMS

The embracing of photolithography to fabricate the precursor photoresist structure has brought significant advantages and enabled rapid and notable developments. Photolithography refers to patterning with light, since the chemical composition of the photoresist changes upon being exposed to light of specific wavelengths, usually 365 nm for SU-8. In SU-8, light starts a cross-linking reaction in the matrix that makes the exposed section less soluble in a developer. The photolithography mask, a patterned stencil that enables the selective pass of light, must then be designed accordingly to the desired final topography: transparent areas in the mask must correspond to the topography to be fabricated. Traditional photolithography of a negative-tone photoresist involves a set of basic processing steps: spin coating a photoresist on a substrate, soft bake to evaporate the casting solvent, exposure through the mask to initiate cross-linking, post-exposure bake to finalize cross-linking and developing to obtain the exposed topography. Optimization of specific steps in traditional photolithography protocols have enabled the fabrication of a number of structures detailed below. The use of electrospinning has

also emerged as an alternative to spin coating to deposit nano-scaled polymer fibers on a substrate, either as bulk or as single fibers.
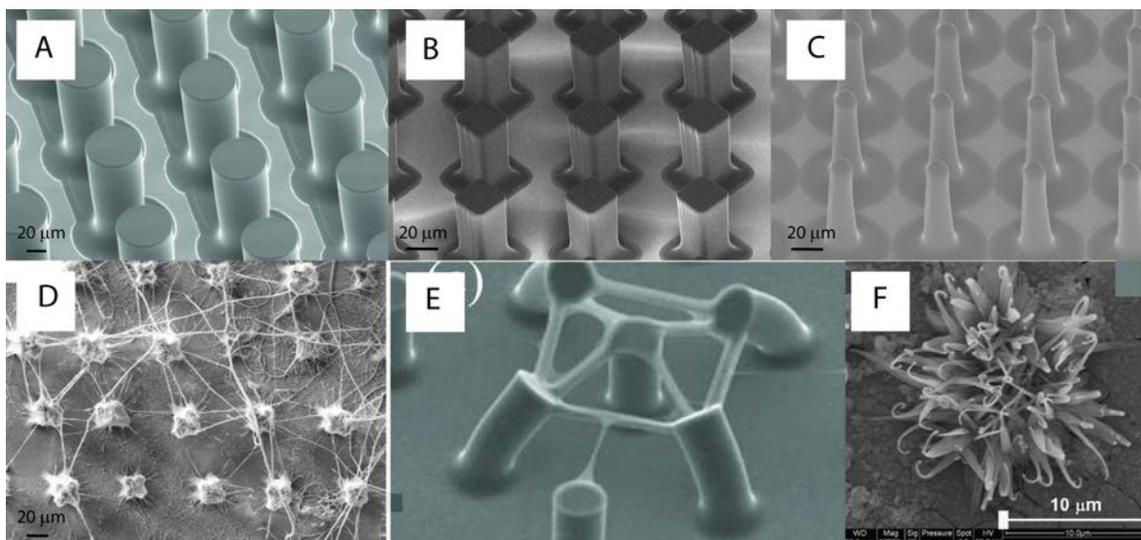

Figure 1. A) High aspect ratio carbon electrodes on planar connecting leads. SU-8 precursor structure was fabricated using 2-layer photolithography. B) Further example of high aspect ratio carbon structures. C) Tapered carbon structures fabricated using backexposure during photolithography. D) Example of SU-8 wires suspended between carbon posts. The fibers were made with electrospinning and later carbonized. Reprinted from [16] with permission from Elsevier. E) Wire arrays made using electron beam lithography. Reprinted from [19] with permission from Elsevier. F) Example of a fractal structure after carbonizing a sol-gel polycondensation of resorcinol and formaldehyde. Reprinted from [14], with permission from Elsevier.

The fabrication of high-aspect ratio carbon structures, like those shown in figure 1A and B, was the first milestone of Carbon MEMS technology. SU-8 was an ideal choice of precursor to achieve this, given its low absorbance of light at wavelengths above 360 nm and the possibility to implement very thick layers in a single spin coating step. Multi-step photolithography has been used to fabricate dense arrays of carbon electrodes of different shapes to be used for capacitors [20, 21], heaters [22], glucose sensors [23], batteries [24–27], bioparticle manipulation using electrokinetics [28–36] and cell scaffolding [37]. By varying the exposure setup and dose, wires and suspended plates can also be manufactured. The implementation of a photolithography process on a releasable substrate or on a sacrificial layer leads to free standing SU-8 structures which were used as micromolds after carbonization [38]. Only the carbonization of free-standing SU-8 structures leads to a true isometric shrinkage. Carbonization of precursor structures anchored to a substrate yields carbon structures with a slight distortion on their base. UV backexposure of an SU-8 layer through a transparent substrate, fused silica for example, leads to the development of tapered structures resembling cones (Figure 1C). Exposure dose can be tailored to specify the taper of the angle [39]. SU-8 wires suspended between two posts were made in 2006 using electron beam lithography. After carbonization, a number of carbon wire arrays were obtained

(Fig. 1E) [19]. Suspended carbon wires like those shown in figure 1D have also been made using electrospinning in recent years [16, 40–42]. Electrospinning was also used to deposit a fiber mat on a substrate which was then patterned using photolithography. The fibrous structures were then carbonized to obtain highly porous carbon structures [15].

Other methods to shape the precursor exist. For example in 2009, carbon fractals and microspheres were obtained by pyrolyzing a sol-gel polycondensation of resorcinol with formaldehyde [14]. The amount of surfactant during sol-gel synthesis determined the size of the spheres and the eventual switch to fabricating fractal structures (Fig. 1F). Micromolding of parylene has also been used to fabricate high aspect ratio carbon posts [43].

Shrinkage

Carbonization is the process by which solid residues with a high content of carbon are obtained from organic materials, usually by pyrolyzing them in an inert atmosphere. The degree of shrinkage and carbon yield, the ratio of the weight of carbon to the weight of the original polymer sample, varies depending on the choice of carbon precursor, its degree of cross-linkage and its shape. For example, SU-8 features a carbon yield of 35-60% [39]. The variability seems to depend on the shape of the precursor micro- or nano-structure.

In the case of Carbon MEMS, carbonization usually takes place in a furnace under a nitrogen, vacuum of forming gas atmosphere under a flow around 2000 ml/minute. The carbonization protocol features 3 stages: 1) a temperature ramp from room temperature to 200-300 °C at 10 °C/min, followed by a 30 minute dwell. This step is to completely eliminate the solvent and allow for any residual oxygen to be evacuated from the chamber and prevent combustion of the polymer as the temperature is raised further. 2) A temperature ramp to 900-1000 °C at 10 °C/min with a one to four hour dwell; and 3) a natural cool down to room temperature by turning the furnace off. Carbonization is a complex process with many reactions taking place concurrently, including dehydrogenation, condensation, hydrogen transfer and isomerization. The pyrolysis process of organic compounds can be divided into three major steps: 1) pre-carbonization, 2) carbonization and 3) annealing. During pre-carbonization (T < 300 °C) molecules of solvent and unreacted monomer are eliminated from the polymeric precursor. The next step, carbonization, can be further divided into two sub-stages: a) from 300 to 500 °C, when heteroatoms such as oxygen and halogens are eliminated causing a rapid loss of mass while a network of conjugated carbon systems is formed and hydrogen atoms start being eliminated; and b) from 500 to 1200 °C, where hydrogen, oxygen and nitrogen atoms are almost completely eliminated and the aromatic network is forced to become interconnected. At this point, permeability decreases and density, hardness, Young's modulus and electrical conductivity increase. The carbon content of the structures carbonized at 900 °C is expected to exceed a mass fraction of 90% in weight. At T ~ 1300 °C more than 99% carbon can be found. Annealing is usually carried out at temperatures above 1200 °C to allow for the gradual elimination of any structural defects and evolution of any further impurities.

Applications

Several applications have been demonstrated using Carbon MEMS. Some examples include the development of 3D architectures for lithium ion batteries [24, 25]; carbon plates in PEM fuel cells derived from machined polyimide [44, 45]; post arrays which were electrochemically activated or decorated with carbon nanotubes to achieve supercapacitors [20, 21]; and the development of carbon microspheres and fractal like structures for sensors and batteries [14]. Other sensor applications include post arrays for rapid quantification of glucose in low concentrations [23], and interdigitated arrays with very narrow gaps to implement redox-based detection of dopamine with an amplification factor up to 25 [46]. C-MEMS devices have also been used as substratum for cell growth [47] and as carbon scaffolds to induce stem cell differentiation [37]. Extended arrays of carbon posts have been integrated in flow-through microfluidics devices to implement high throughput cell separation and manipulation using electric fields in a technique now known as carbon-electrode dielectrophoresis [28, 29, 48]. This technique has been used to purify viable bacteria from an antibiotic-treated sample [31], to implement a sample preparation step that increases the sensitivity of traditional PCR protocols [34], and to extract DNA from a sample [32]. The combination between centrifugal microfluidics and carbon-electrode DEP marked an important step towards a sample-to-answer diagnostics platform [30]. Particle transport using electric fields has also been implemented [36], as well as high throughput electrical lysis of different cells [33]. Other applications include the use of carbon MEMS techniques to fabricate robust, inexpensive carbon shapes for the micromolding of bulk metallic glasses [38, 49].

Future directions of the field

Although most of the Carbon MEMS work has been done using SU-8 photoresist and photolithography, there is a strong interest on using different precursors, including biopolymers [50]. Development of techniques for shaping these precursors in the micro- and nano-scale will be needed, *i.e.* extrusion-based additive manufacturing. Electrospinning has been gaining importance as a technique to fabricate precursor nanostructures and is expected to be developed further. A broader use of composites is also expected: the patterning of SU-8-CNTs composites has been published [21] and preliminary work has also been disclosed using SU-8- silver and SU-8-silica composites. The use of catalysts to lower the energy required for carbonization is also of high interest; as well as understanding the shrinking process.

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