

## WHITEPAPER

INTRODUCING SMOLTEK CARBON NANOFIBER TECHNOLOGY

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Vincent Desmaris, Muhammad Amin Saleem, and Sareh Shafiee Digital Object Identifier 10.1109/MNANO.2015.2409394 Original date of publication: 7 April 2015

## **Examining carbon nanofibers** – Properties, growth and applications

One dimensional carbon nanostructures have been known and fabricated for more than a hundred years [1] and were originally referred to as "filamentous carbon," "carbon filaments," or "carbon whiskers" [2]. However, it was only during the 1990s and 2000s with the demonstration of carbon nanotubes (CNTs) [3], [4] and the introduction of catalytic plasma-enhanced chemical vapor deposition (PECVD) [5], [6] that one-dimensional carbon nanostructures emerged as a promising solution for a large variety of applications. In this whitepaper, carbon nanofibers (CNFs), a particular type of one-dimensional carbon with unique properties, are described, their growth is discussed, and their main applications are presented.

## SUMMARY

Even though CNFs do not fully meet the electrical and thermal performance of CNTs, the reliability and deterministic nature of their low-temperature growth and properties makes them extremely attractive for various applications. They have been shown to provide alternative and potentially disruptive solutions for high-reliability lowpitch interconnects or flip–chip and die-attach when employed intrinsically (i.e., as grown) or in combination with solders and polymers, respectively. Additionally, their thermal properties and possibility to grow them on chip can provide new thermal managements solutions for packaged components. Furthermore, CNF-based materials for the enhancement of supercapacitors as replacement for lithium battery or on-chip decoupling capacitors widen the prospective application area of CNFs.



Vertically aligned carbon nanofibers, grown by Smoltek



### **CNFs INTRODUCTION**

CNFs are allotropes of carbon having sizes of about 50–100 nm diameter with length ranging from one to hundreds of micrometers (Figure 1).

Unlike CNTs, which are hollow cylindrical structures made of one or several concentric graphene rolls (single-wall CNT and multiwall CNT, respectively), CNFs consist of a stack of graphene cones. CNFs and CNTs are distinguished by measuring the angle a between the one-dimensional carbon nanostructure axis and the graphene layers forming it. single- or multiwall CNTs exhibit an angle of zero, whereas CNFs reveal a nonzero angle (Figure 2).

The electronic bindings between carbon atoms in a CNF are twofold: inplane covalent bonds within each cone of graphene and interplane van der Waals bindings between the cones. as a result, the a angle not only determines the nature of the carbon nanostructures but also its electrical and mechanical properties.

Unlike CNTs, whose conductivity depends on their chirality and usually present a ratio of metallic/ semiconducting CNTs of 1/3 [7], CNFs have the advantageous properties of being 100% metallic and possibly individually grown at low temperatures.

The typical properties of a CNF are summarized in Table 1. The variations depend on their processing conditions and, hence, their structural configuration, diameter, length, interface, and contacting schemes.



Figure 1: (a) A scanning electron microscope (SEM) micrograph of CNFs and (b) a transmission electron microscope (TEM) micrograph of CNFs.



Figure 2: The schematics of the internal structure: (a) a single-wall CNT, (b) a multiwall CNT, and (c) a CNF.

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## **CNF GROWTH**

The properties of CNFs are largely dependent on the growth conditions. arc discharge and its modern variant electrospinning can provide pure material; however, the control and reproducibility of the length, shape, and alignment of CNFs produced

using these techniques are inherently limited and consequently restrain their applications fields.

It is the development of catalytic PECVD during the late 1990s [5] that made possible a more deterministic method to produce CNFs on substrates. The positioning of the fibers on a substrate would depend on the presence of a catalyst. Therefore, the CNF alignment is controlled by the electric field generated by the plasma, whereas the length of the CNF is simply determined by the growth time.

Even though the full growth process of the CNF is not completely under- stood, it is generally accepted [8] that the perpendicular alignment of the CNF to the substrate results from uniform stress at the catalyst–CNF interface due to electrostatic forces, which compensate for any non-verticallity during the growth process.

In addition, many PECVD systems using different radiofrequency and microwave sources were developed [9]–[11]. However, so far, dc PECVD has produced the best results for

TABLE 1 Typical properties of CNFs.	
PARAMETER	TYPICAL VALUES
Diameter	1–100 nm
Length	0.1–100 µm
Fill factor when grown as films	5-80%
Density	<2 g/cm <sup>3</sup>
Thermal expansion coefficient (CTE)	~10-6/K-10-7/K
Young's modulus	80800 Gpa
Poisson's ratio	0,2-0,25
Tensile strength	30 Gpa
Electrical resistivity	0.1 μΩ·m–2mΩ·m
Thermal conductivity	20-3,000 W/m·K
Temperature tolerance	>1,000 °C without oxygen, >400 °C with oxygen
Vertical pressure strength as grown	>50 Bar
Horizon shear strength as grown	Weak
Shear strength of bonded nanostructures	1.5x-3x MIL

the fabrication of CNFs at a low complementary metal–oxide semiconductor (CMOS)-compatible temperature using different pre- and postprocessing techniques to cope with the inherent discharge problems that arise when using insulating substrates [12].

The most accepted description of the growth mechanism of CNFs originates from the pioneering work of baker et al. in the 1970s. it decomposes the growth process into four steps, which are shown in Figure 3. First, a molecule of gas precursor is adsorbed at the surface of the catalyst. second, the gas molecule is dissociated at the surface of the catalyst. Third, the dissociated carbon atoms diffuse through [1] or at the surface [13] of the catalyst toward the CNF, depending on the propensity of the catalyst to form carbide or enable low-energy diffusion of carbon at its surface. Fourth, the carbon atoms are incorporated into the CNF. as a result, the PECVD growth of the CNF requires three components: gases, an underlayer, and a catalyst.



Figure 3: CNF growth mechanisms: (a) catalyst nanoparticle formation, (b) carbon adsorption at the catalyst site,

(c) carbon through the bulk and/or surface diffusion of the catalyst, and(d) nucleation.

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The catalytic PECVD growth process generally involves two gases. The first acts as a carbon precursor (usually acetylene or methane) and reacts with the catalyst to build the CNF. The second gas is a carbon etchant (often ammonia or hydrogen) and prevents parasitic growth [14].

The role of the underlayer, located between the catalyst and the substrate, is to act as a diffusion barrier between the catalyst and the substrates. The underlayer prevents the catalyst from being poisoned by a reaction with the substrate or diffusion in the substrate as a result of the elevated temperature during the growth process. Various metal underlayers based on refractory metals or alloys have been used depending on the choice of substrates and catalysts. a comprehensive review can be found in [15].

The activity of the catalyst particle is crucial for the growth process [16]. It consists of one or few elements [15]; however, the vast majority of the reports regarding the growth of CNFs deals with catalysts made from transition metals such as iron, cobalt, and nickel [15]. In addition, it is currently believed that the catalyst should form nanoparticles of a favorable size to initiate the growth of a CNF. Therefore, the catalyst can be deposited as a film using physical vapor deposition techniques or directly as nanoparticles using spray coating [17] or spinning [18], provided that the growth temperature will lead to a dissociation of the film into droplet/nanoparticles of appropriate size.



Figure 4: An SEM micrograph of individual fibers.

### **APPLICATIONS**

Their remarkable properties, combined with the unique lowtemperature deterministic growth process, as seen in Figure 4, make CNFs suitable for semiconductor packaging applications and building advanced capacitors.

## **CNFs AS INTERCONNECTS**

Since the CNFs grown by catalytic PECVD are vertical dimensional material with large aspect ratios and are inherently rooted and attached to the underlying substrates, which can be grown individually (Figure 4) with controllable pitch down to single-digit microns on substrates, CNFs are alternatives to micro- and nano-copper pillars for packaging chips with high-density inputs/outputs [19]. In fact, their resilience to electrical [20] and chemical corrosion makes them highly relevant when electromigration is limiting the reliability of copper interconnects for small pitch applications.



Figure 5: (1.) An SEM micrograph of carbon bumps grown (hence rooted) on solder pads and (2.) an array of carbon bumps.

Clusters of thousands of CNFs result in the formation of a forest, which have been considered interconnects. such bump inter- connects (Figure 5), made solely of CNFs rooted on the underlying bonding pads, used as flip–chip or integrated, have been demonstrated to form robust interconnects supporting current densities of several tens of ma/cm2. in addition, such bumps made solely out of CNFs have been demonstrated to provide a microwave link suitable for high-frequency operations to about 20 GHz [21] and would be perfectly suited for the packaging of high-power microwave devices such as gallium nitride field-effect transistors or silicon carbide transistors or diodes.



## **CNFs AS SOLDER JOINT ENHANCERS**

A number of studies found that CNFs can reduce the CTE of solder when embedded in the solder [22]–[25]. commonly used tin–silver–copper (SnAgCu, SAC) solders can have CTEs ranging from 16.7 to 26 ppm/°C, whereas the CTE of the adjoining silicon can range from 3 to 8 ppm/°C, creating a mismatch that can cause solder joint cracking and thermal cycling over time. The effect of this mis- match as the solder volume is reduced raises the probability of joint failure significantly. CNFs have the ability to bond with solder while still remaining rooted on the integrated circuit (IC) pads, enabling them build composite materials that take advantage of their high (terapascal) Young's modulus to provide robust joints between two chips or chips to package [25], [26].

## The PECVD growth of the CNF requires three components: gases, an underlayer, and a catalyst (figure 6).

Studies show that CNFs using flash metal layers wet many of the commonly used IC assembly solders, such as indium in 290, In-Sn [19], or SAC305 [4], remarkably well leaving few voids at the fiber-CNF interface and forming a composite joint with lower CTE than the solder itself, since vertically aligned CNFs (VACNF) have hardly any CTE. Furthermore, the wettability of the CNF to the solder could prevent the solder from wicking out and away from the pads, thus reducing the number of solder shorts that occur as the solder pad-pad pitch gets smaller. The length of the rooted CNF is solely determined by the CND growth process. This length makes it possible to enhance the reliability of the fiber to solder joint since fibers could be longer than the intermetallic region on the solder to pad interface. all of the previously outlined features of the CNF/solder composites make them promising for nanoscale interconnect with improved reliability. The thermal fatigue life was demonstrated to be increased by 87% by creating a 29% (by volume) mixture of CNFs and solder [22].

The considerable Young's modulus of the CNFs along their growth axis confer them remarkable piercing properties,

e.g., for nanoimprinting [27]. Therefore, beside the rebar of the intermetallic compound layer in the bump formation, the CNF's remarkable compressive strength would imply that they could pro- vide considerable reinforcement of a solder bump, preventing it from deforming and dimpling during testing [28].

## **CNF-BASED ADHESIVE INTERCONNECTS**

CNF films can also be used as performance enhancers of flip–chip bonding when soldering is not possible. Using a standard thermal compression flip–chip technique, composites made of CNF grown directly on chips, such as SiC power transistors or Si laterally diffused metal–oxide– semiconductor and embedded into a polymer, have shown to provide good ohmic contacts (specific contact in the order of  $105 \ \Omega/cm^2$ ) on direct-copper-bonding or lead frames [19]. after assembly, the embedding of CNFs in polymers forms a type of anisotropic conducting film, where the polymer provides the necessary adhesion and compensates for the relatively weak shear stress resilience of the CNFs. such adhesive interconnects meet the MILx1 to MILx3 standards.

### **CNFs AS ELECTRODE MATERIAL FOR CAPACITORS**

Because of their metallic nature, CNFs, when grown on metallic surfaces, provide a large increase in effective surface area per footprint. as a result, CNFs have also been implemented as electrode material for two types of capacitors, namely, decoupling capacitors and supercapacitors.



Figure 6: Schematic depiction of PECVD reactor.

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CNF-based capacitor electrodes can be used in two relatively large decoupling capacitors directly on-chip, having a large advantage over the embedded film capacitors [29]–[30] surface mount and land-side decoupling capacitors can be realized by growing the CNF directly on the connecting pads of an IC chip, provided that the growth process is compatible with the IC's technological node [31]. Different prototypes of CNF-based decoupling capacitors have shown an enhancement by a factor of 10–100 in capacitance density compared to current technology [31].



Figure 7: An SEM micrograph of an ultra-thin carbon nanofiber (CNF-MIM) capacitor made by Smoltek – total

height is just under 40 micrometers, including substrate.

Supercapacitors are electrochemical capacitors [32] with high power density. Their lifetime is 100 times larger than the lifetime of lithium batteries and their performance is largely dictated by the effective area of the electrode materials and its structure. Historically, active carbon has been used as electrode material; however, more recently, flexible sheets of electro-spun CNFs are considered prospective electrode material for supercapacitors because of their interconnected pores and high mechanical and electrochemical stabilities [33], [34].

The main drawback of low specific surface area has been circumvented by their nitrogen doping and functionalization with CNTs, resulting in three-dimensional CNT–CNF composites suitable for supercapacitors with high power

density and long-life-cycle performance [35] for nextgeneration supercapacitors.

## **CNFs AS THERMAL INTERFACE MATERIAL**

CNFs exhibit large thermal conductivity but mainly in the direction of their growth axis. as a result, early studies [36] have emphasized the need to combine CNFs as a filler into a matrix of another material such as solders [37] or copper [36]. This intrinsic isotropic heat conduction in the CNF limited the prospect of using them in a thermal interface mate- rial. in fact, much focus on the packaging industry has been on heat spreading using materials such as graphite. despite the successful implementation of heat spreaders, removing heat from the chips to the package and CNF is expected to play a key role in the vertical extraction of heat from the chip to the package [38].

## CNFs FOR NANO-ELECTROMECHANICAL SYSTEMS

VACNFs also have potential in nano-electromechanical systems because a single CNF can be grown up to a few tens of microns long while standing alone vertically aligned at a controlled position, which can be addressed easily. The long single CNF can be easily bent by applying a low electric field. The nano-electromechanical systems based on CNF grown using the PECVD technique at CMOS-compatible temperature can be embedded in CMOS chips. Cruden and Cassell [39] made the nano-electromechanical switch by bending VACNFs using an electric field and actuated the fiber by applying 10 V potential across a 5-µm distance. The fiber could bear the 900 bend and returned to its original position when the electric field was switched off.

## The positioning of the fibers on a substrate would depend on the presence of a catalyst.



#### **ABOUT THE AUTHORS**

Vincent Desmaris earned his M.Sc. degree in material science from the institute of Applied Science, Lyon, France, in 1999 and his Ph.d. degree in electrical engineering from Chalmers University of Technology, Gothenburg, Sweden, in 2006. In 2007, he joined Smoltek AB, where he has been the Chief Technology Officer. His main interests are the development and modeling of devices, components, and prospective technological solutions for themicroelectronics industry, based on carbon nanofibers. Since 2013, he has also been an associate professor at Chalmers University of Technology in the field of terahertz technology and microwave electronics. His research deals with radioastronomy instrumentation and superconducting electronics. He is the author and inventor of more than 70 articles and patents.

Muhammad Amin Saleem is currently an industrial Ph.d. student at Chalmers University of Technology and employed by Smoltek AB. His main research interests are the different applications of carbon nanostructures. He received his M.Sc. degree in nanoscale science and technology in 2006 from Chalmers University of Technology, after which he worked as a research assistant with the atomic Physics Department, Gothenburg University, until 2007. From 2007 to 2012, he worked as a processing engineer at Smoltek AB, and he is also the inventor of two patents filed by smoltek.

Sareh Shafiee earned her B.S. degree in solid-state physics from IA University, Tehran, Iran, in 2008 and her M.Sc. degree in science from University of Umeå, Sweden, in 2014. she then joined Smoltek AB as a process engineer, working with carbon nanomaterials fabrication. Her main research interests are investigating the physical properties of vapor-grown carbon nanofibers as well as the development and electrical property measurements of carbon nanofibers.

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