Carbon Nanotube Nano-Composite Fibers and Carbon Nanotube Fibers by Wet Spinning Process

P. Poulin, B. Vigolo, P. Bernier, M. Paillet, P. Launois, F. Soutric,
M. Dieudonne, M. Grac, and K. Gedda

Nanoledge Inc. and Associated Labs,
Montpellier 34940, France

ABSTRACT

Single wall carbon nanotubes are known for their unique and very promising properties at the molecular level [1]. The challenge is to transfer these properties to structural materials that can be used for diverse applications, space and aeronautic indutries will be eager for. The most exciting of all being the combination light-weight, and anisotropic shape with mechanical and/or electromechanical properties. The wet spinning process developed by Vigolo and Poulin, et al [2] and the treatments associated [3] are designed to be a perfect mean to develop nano-fibrils and nano composite fibers. The approach allows to introduce the nanotubes in a polymer matrix, and to align them in the axis of the nano-fibril. Although mechanical properties of the fibrils are still far from the ones of the nanotube itself, other properties are interesting. The processes developed allow the realization of endless fibrils in composites where CNT play the role of the matrix and lead us to think realization of a close to 100% nanotube fibril is at hand.

Keywords: carbon nanotube, nano-composite fibers, carbon nanotube fibers, wet spinning process

1 INTRODUCTION

Single wall carbon nanotubes are known for their unique and very promising properties at the molecular level [1]. The challenge is to transfer these properties to structural materials that can be used for diverse applications, space and aeronautic indutries will be eager for. The most exciting of all being the combination light-weight, and anisotropic shape with mechanical and/or electromechanical properties. The wet spinning process developed by Vigolo and Poulin, et al [2] and the treatments associated [3] are designed to be a perfect mean to develop nano-fibrils and nano composite fibers. The approach allows to introduce the nanotubes in a polymer matrix, and to align them in the axis of the nano-fibril. Although mechanical properties of the fibrils are still far from the ones of the nanotube itself, other properties are interesting. The processes developed allow the realization of endless fibrils in composites where CNT play the role of the matrix and lead us to think realization of a close to 100% nanotube fibril is at hand.

2 WET SPINNING PROCESSES

The original process consists of injecting a solution of dispersed nanotubes in a flow of a polymer solution in order to form a ribbon which then is collated into a nanotube nanofibril. The nanofibril is obtained thanks to the coagulation reaction of the polymer with the nanotubes. Alignment and orientation of the tubes is improved through variation of the speed flow (shear forces) and geometry.

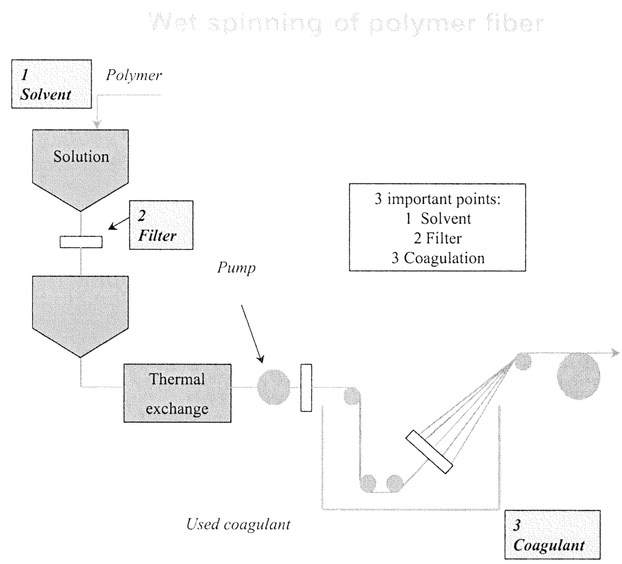

Fig 1: Wet spinning of nanocarbone fibril.

The raw material used is produced with the arc-discharge process, through a specific Nanoledge method ,. The material thus obtain is composed of bundles of nanotubes interlaced with carbon impurities and catalysts. The solution is obtained by sonicating raw material in a mixture of water (or solvent) and surfactant such as sodium dodecyl sulfate. Preparing the solution is primordial to the success of the process. Low or high concentration of the surfactant prevent the birth of a regular and steady nanofibrils.

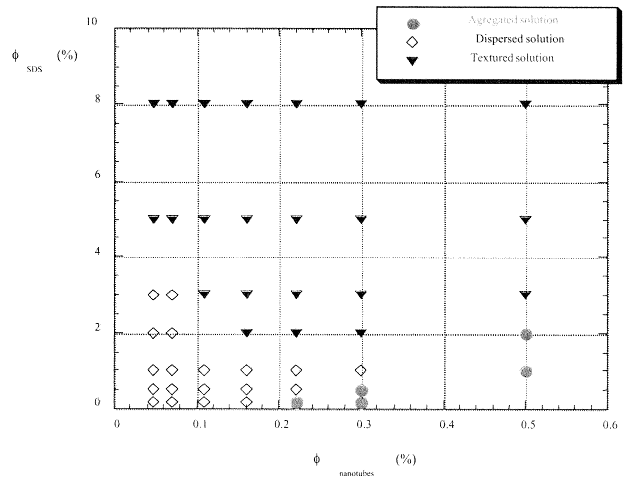

Fig 2 : Phase diagram of SDS/SWCNT/H2O.

The intermixing of the flows results in the birth of the nanotubes fibrils through the diffusion of the polymer inside the nanotube solution. Shear forces and chemisty interact to shape the fibrils by coagulation and alignment of the nanotubes.

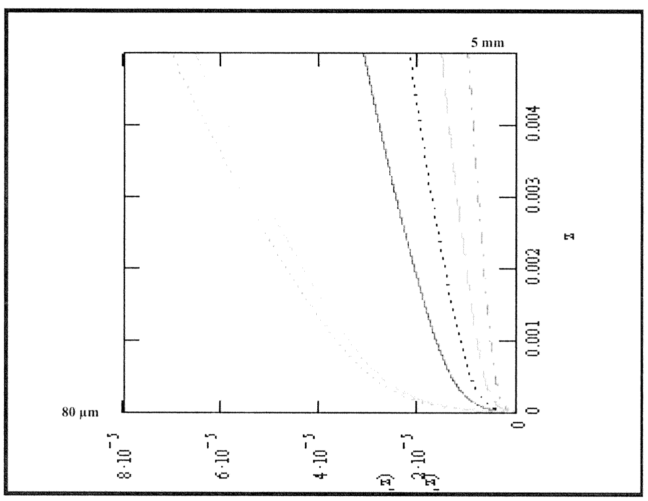

Fig 3 : Diffusion of polymer in the nanotube dispersion – simplified process.

The nanofibril thus formed is bathing in a rincing solution. In most of the cases, its shape is more the one of a ruban until drying is completed. Drying process and further treatments are playing on the elasticity of the fiber thanks to the nanotubes. Pulling the nanofibril out of the rincing solution, brings elongation and hence alignment of the nanotubes.

Repeating this process: getting the new fibril rewetted, swollen and dried vertically [3] improve alignment of the nanotubes. (by 40%) [3].

3 PROPERTIES OF THE NANOFIBRIL

Control of the diameter can be controlled by varying the parameters (flow conditions and dimensions of the capillary tube). Diameters can thus vary from a few micrometers to 100 µm. [4] Density of the fibers is estimated by directly weighing them : 1.3 to 1.5 g/cm3.

Alignment in the key to unlock nanotubes molecular properties on a macroscopic level [5]. Different analysis have shown the impact of alignment on mechanical and electro-mechanical properties – cf extract herafter. Raman spectroscopy and XR scattering experiments have shown a preferential alignment of the nanotubes in the fibers. As previously mentionned mechanical properties targetted may be at hand but are still far from industrial needs. However other properties can be reached in the meanwhile. The high aspect ratio of the tubes explains partly this phenomenon. Thus stretched fibers with more aligned nanotubes have a Young Modulus increased by a factor of 4.

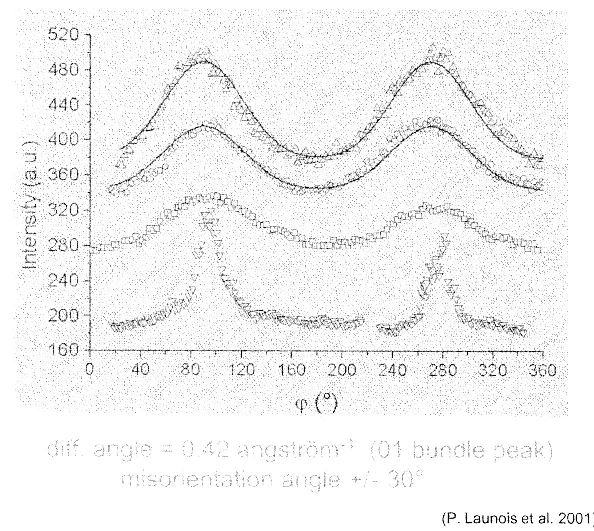

Fig 4 : X ray diffraction of nanotubes fibers.

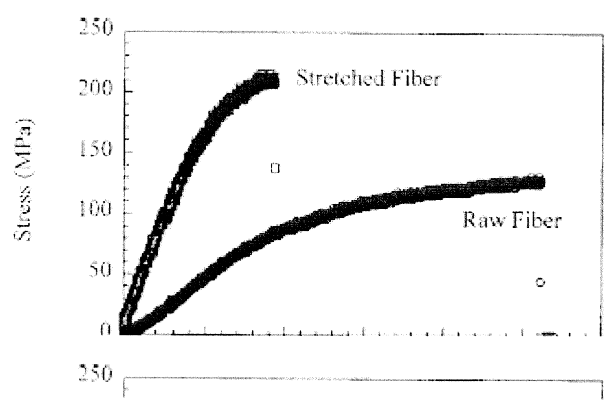

Fig 5 : Comparison of an initial vs stretched nanofibril.

Similarly to bucky papers [6], the nanofibrils can be used for their electromechanical properties, as actuator per say. [4] Therefore for a fibril coated with a polymer gel, and bathed in a ionic solution, contraction and expansion can be seen for a voltage of 1V with a frequency of 1Hz.

4 CONCLUSIONS

According to the properties we focus on, different paths are open to improve the properties of the nanofibrils / nanofibers made through electro-spinning process. Alignment has mentionned is the key and can be improved either directly at birth or through rewetting post-treatments. Functionnlaization of the tubs and choices of polymer solutions can also improve the alignment ratio. Thus we can hope to get a fiber that will answer "soon" the needs of the space industry.

5 REFERENCES

[1] M.M. J. Treacy, T.W. Ebbesen, J.M. Gibson, Nature 381, 678 (1996)

[2] B. Vigolo, A. Pénicaud, C. Coulon, C. Sauder, R. Paillet, C. Journet, P. Bernier & P. Poulin – Macroscopic Fibers and Ribbons of Oriented Carbon Nanotubes, Science 17 Nov 2000, V 290, pp1331-1334

[3] B. Vigolo, P. Poulin, M. Lucas, P. Launois, P. Bernier Improved structure and properties of single wall carbon nanotube spun fibers Applied Physics Letters 29 July 2002

[4] B. Vigolo, P. Launois, M. Lucas, S. Badaire, P. Bernier, P. Poulin Fibers of carbon nanotubes, MRS

[5] F. K. Ko, W. B. Han, S. Khan, A. Rahman, & O Zhou Fibrous Materials Research Center, Department of Materials Engineering, Drexel University, Proceedings of the ASC 16th Annual technical conference.

[6] R.H. Baughman et al, Science 1999, V 284 pp1340.