APPLIED PHYSICS LETTERS **108**, 153108 (2016)

Precise measurement of single carbon nanocoils using focused ion beam technique

Yasushi Nakamura,[1] Yoshiyuki Suda,[1,a)] Ryuji Kunimoto,[1] Tamio Iida,[2] Hirofumi Takikawa,[1] Hitoshi Ue,[3] and Hiroyuki Shima[4]

[1]*Department of Electrical and Electronic Information Engineering, Toyohashi University of Technology, Toyohashi, Aichi 441-8580, Japan*
[2]*Department of Electrical and Computer Engineering, National Institute of Technology, Gifu College, Motosu, Gifu 501-0495, Japan*
[3]*Fuji Research Laboratory, Tokai Carbon Co., Ltd., Oyama, Shizuoka 410-1431, Japan*
[4]*Department of Environmental Sciences, University of Yamanashi, Kofu, Yamanashi 400-8510, Japan*

(Received 17 August 2015; accepted 25 March 2016; published online 12 April 2016)

We have developed a precise resistivity measurement system for quasi-one-dimensional nanomaterials using a focused ion beam. The system enables the resistivity of carbon nanocoils (CNCs) to be measured and its dependence on coil geometry to be elucidated. At room temperature, the resistivity of CNCs tended to increase with coil diameter, while that of artificially graphitized CNCs remained constant. These contrasting behaviors indicate coil-diameter-induced enhancement in structural disorder internal to CNCs. Low-temperature resistivity measurements performed on the CNCs revealed that electron transport through the helical axis is governed by the variable range hopping mechanism. The characteristic temperature in variable range hopping theory was found to systematically increase with coil diameter, which supports our theory that the population of sp^2-domains in CNCs decreases considerably with coil diameter.
[http://dx.doi.org/10.1063/1.4945724]

Carbon nanocoils (CNCs) are an exotic class of low-dimensional nanocarbons with helical shape.[1] Typical thicknesses and coil diameters of CNCs fall within the ranges of 100–400 nm and 400–1000 nm, respectively,[2,3] and their full lengths are on the order of several tens of micrometers. Electron microscopy measurements[2–6] have revealed that single CNCs are bundles of helically twisted carbon nanofibers (CNFs) that encapsulate a long, thin, coaxial hollow core.[3] The helical structure of CNCs originates from spatial inhomogeneity in the rate of carbon precipitation over the active surfaces of catalyst particles.[4,6] Possibly owing to this inhomogeneity, the inside of a CNC is filled with amorphous carbon, i.e., a mixture of many small sp^2-domains with disordered sp^3-bonded carbon.

The helical geometry of CNCs implies versatile applications such as microwave absorbers[7] and in energy devices.[8,9] To exploit their potential utility, the mechanical and electrical properties of CNCs should be clarified. Against this backdrop, efforts have been made to obtain the spring constant and shear stress of CNCs through stress-strain measurements.[10–12] On the issue of electrical properties, several research groups have addressed the measurement of the resistivity of CNCs using different approaches.[13–16] Hayashida *et al*. developed a measurement system by connecting both ends of a CNC to a carbon electrode using an electron beam deposition technique. They suggested that the room-temperature resistivity of CNCs ranged from $7.1 \times 10^{-3}\,\Omega\,\mathrm{cm}$ to $9.3 \times 10^{-3}\,\Omega\,\mathrm{cm}$,[13] the measurement samples of which were selected from a large amount of CNCs dispersed on a substrate. Low-temperature resistivity measurements were also performed by Chiu *et al*., who used a four-terminal method[14] to show that the temperature-dependence of the CNC resistivity below 280 K is largely attributable to the variable range hopping (VRH) mechanism.[17]

Despite the pioneering work described above, the relationship between the pristine (i.e., undeformed) shape of CNCs and their electrical resistivity remains to be addressed.[18] During CNC synthesis, spatial inhomogeneity in the precipitation of carbon on catalytic particles governs both the coil geometry and the internal structure of the constituent fibers. This implies a certain correlation between coil geometry and the degree of structural disorder inside CNCs. Therefore, it is inferred that variation in the coil geometry induces changes in electrical resistivity. To verify this conjecture, the development of a high-precision measurement system into which an artificially selected CNC with desired coil geometry can be integrated is indispensable.

In this study, we established a precise measurement system using a focused ion beam (FIB) technique that enables precise evaluation of the electrical resistivity of CNCs. Room-temperature resistivity measurements of many CNCs and artificially graphitized CNCs (G-CNCs) with various shapes unveiled a clear relationship between coil diameter and electrical resistivity. It was further revealed that the temperature-dependence of CNC resistivity between 4 and 280 K is well described by the VRH theory.

CNCs and G-CNCs samples were synthesized by catalytic chemical vapor deposition[3] (see supplementary materials, S1).[19] After the synthesis, a portion of the CNCs was graphitized to obtain G-CNCs by heat treatment in Ar atmosphere at 2873 K for 30 min using a Tammann oven. Figure 1 displays transmission electron microscopy (TEM; JEM-2100F, JEOL Ltd., Tokyo, Japan) images of an obtained CNC and G-CNC; electron diffraction patterns of the same

[a)]Author to whom correspondence should be addressed. Electronic mail: suda@ee.tut.ac.jp

