Unidirectional alignment of Carbon nano-sized fiber using drawing process

YOUNGMIN BAIK, SANGKWAN LEE
Materials Engineering Department, Korea Institute of Machinery and Materials, Changwon 641-010, Korea

YOUNGHWAN JANG, SANGSHIK KIM*
Division of Materials Science and Engineering, Engineering Research Institute, Gyeongsang National University, Chinju 660-701, Korea
E-mail: sang@gsnu.ac.kr

Published online: 16 September 2005

Recently, there is great interest in the metal-matrix composites (MMC) reinforced with the carbon nano-sized fiber (CNF) due to their superior mechanical, thermal and electrical properties [1, 2]. At present, the fabrication of MMC reinforced with the CNF is considered to be practically impossible using the liquid infiltration process, since most of the composite manufacturers and researchers believe that the applied pressure should be extremely high [3]. Previously, however, the authors conducted a theoretical calculation of the required pressure necessary for the Cu melt to infiltrate the CNF bundles, and reported that the fabrication of CNF reinforced Cu composite was possible by the liquid infiltration process at a pressure as low as 20 MPa by employing a simple hydrostatic pressurization principle [4]. In order to minimize the hydrostatic pressure loss during melt flow through the CNF bundles, the Cu tubing compacted with CNFs were used as a perform material for the present composite fabrication, as schematically demonstrated in Fig. 1. For the successful application of CNFs for the MMC fabrication as a reinforcing phase, however, the directional control of the fiber is of great importance. Unfortunately, at present, the CNFs are supplied in the form of highly entangled bundles as a result of their strong attractive forces between the fiber surfaces, as shown in Fig. 2. Several methods, including centrifugal force method and electrical field method, have been endeavored for the unidirectional alignment of the fiber [5]. However, little success has been made. In the course of fabricating Cu wire compacted with CNFs, the authors found that the CNFs could be aligned unidirectionally by utilizing a simple multi-drawing process, as schematically illustrated in Fig. 3. First of all, the CNFs, produced at Showa Denko (Japan) with an average diameter of 150 nm and an average length of 15 μm, were mechanically compacted into the 8 mm diameter oxygen-free Cu tube with a length of 300 mm. The Cu tubing was then continuously drawn down to the final diameter of 0.2 mm. To prevent the CNF extruding from the tubing during the multi-drawing process, both ends of the tubing were tightened by swaging. The drawing speed was maintained at 1.6 m/min. The drawing force of 5 kN was used in the present study, which was obtained by using the upper bound solution [6] as follows:

$$\begin{aligned}\sigma_d \cong\ & 2\left(\frac{R_i}{R_o}\right)^2 f(\alpha_i)\mathrm{In}\frac{R_o}{R_{fo}} + \frac{2}{\sqrt{3}}\left(\frac{R_i}{R_o}\right)^2 \\ & \times\left(\frac{\alpha_i}{\sin^2\alpha_i} - \cot\alpha_i\right) + \frac{2}{\sqrt{3}} m\left(\frac{\sigma^o_{Cu}}{\sigma^o_{fiber}}\right)\left(\frac{L}{R_f}\right) \\ & + 2\left(\frac{\sigma^o_{Cu}}{\sigma^o_{fiber}}\right)\left[f(\alpha) - f(\alpha_i)\left(\frac{R_i}{R_o}\right)^2\right]\ell n\frac{R_o}{R_{fo}} \\ & + \frac{2}{\sqrt{3}} m\left(\frac{\sigma^o_{Cu}}{\sigma^o_{fiber}}\right)\cot\alpha \times \ell n\frac{R_o}{R_{fo}} \\ & + \frac{2}{\sqrt{3}}\left(\frac{\sigma^o_{Cu}}{\sigma^o_{fiber}}\right)\left\{\frac{\alpha}{\sin^2\alpha} - \cot\alpha - \left(\frac{R_i}{R_o}\right)^2 \right. \\ & \left. \times\left(\frac{\alpha_i}{\sin^2\alpha_i} - \cot\alpha_i\right)\right\} \end{aligned} \tag{1}$$

where σ_d is the theoretically calculated drawing force, $f(\alpha)$ is the function of the semi-die angle, σ^o_{Cu} is the flow stress of Cu ($\approx$ the average of 0.2% yield strength), σ^o_{fiber} is the flow stress of CNFs. The R_o, R_i, R_{fo}, R_f, α, α_i, L and m value, respectively, represents the outer diameter of tube, the inner diameter of tube, the final outer diameter of tube, the final inner diameter of tube, the die angle, the internal die angle, the bearing length and the friction coefficient between tube and die, respectively.

The optimum die angle was calculated by using the following equation:

$$\alpha_{opt} \cong \sqrt{\frac{3}{2} m \frac{\ell n(R_o/R_{fo})}{1-(R_i/R_o)^3}} \tag{2}$$

where m, R_o, R_i and R_{fo}, respectively, represents the friction coefficient between tube and die, the outer diameter of tube, the inner diameter of tube and the final

* Author to whom all correspondence should be addressed.

0022-2461
DOI: 10.1007/s10853-005-4557-0

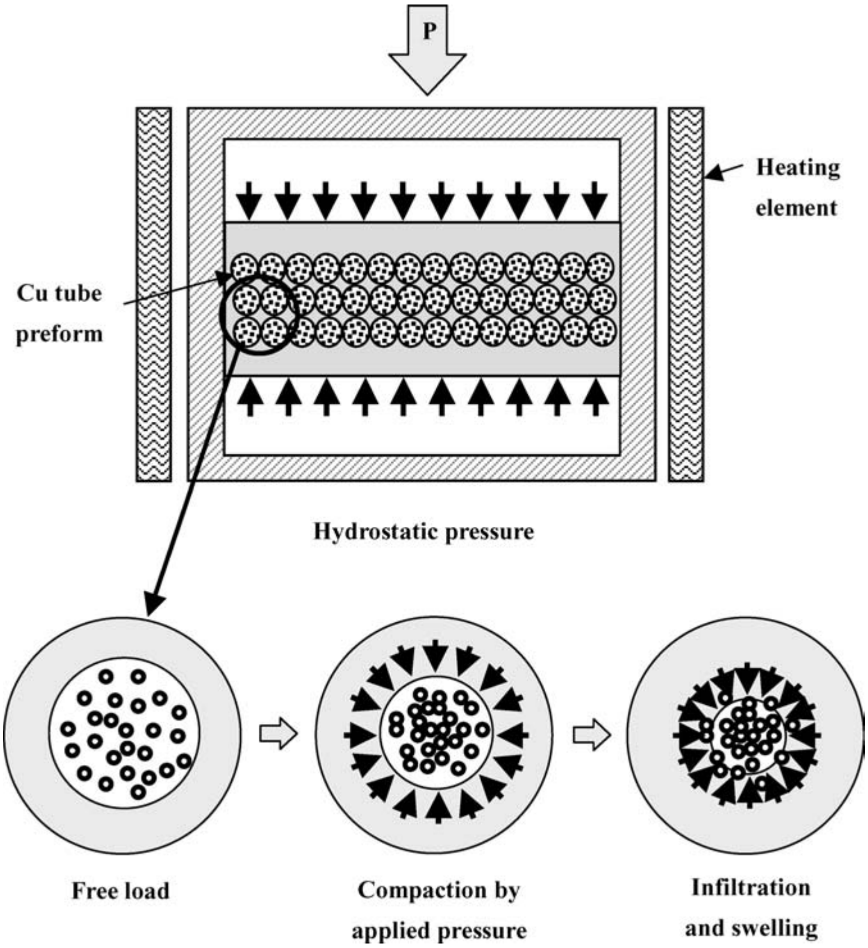

Figure 1 Schematic illustration of liquid pressing process for the fabrication of MMC reinforced with CNF.

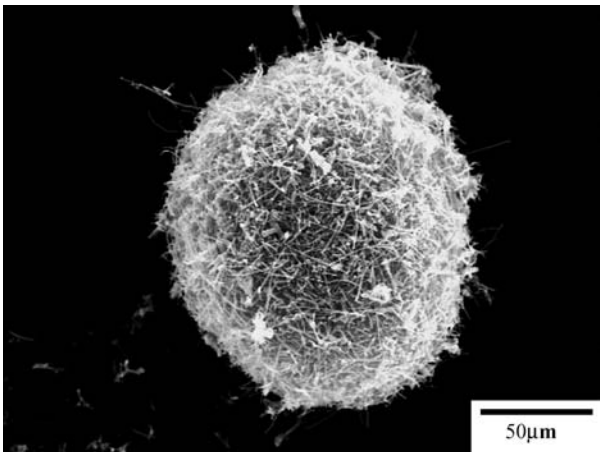

Figure 2 The SEM micrograph of the as-received CNF agglomerates, showing a severely entangled appearance.

outer diameter of tube, respectively. After several drawing process, the Cu tubing was annealed at 440 °C for 5 hr for the subsequent drawing process. Fig. 4 shows SEM micrographs of the (a) drawn Cu tube packed with CNFs and (b) cross-section of the tube, respectively. Fig. 4b shows that the CNFs were almost perfectly aligned along the drawing direction. Some CNFs appeared to lose the perfect alignment possibly during cutting and polishing the surface. Conclusively, the perfect alignment of the CNF was obtained by mechanical drawing process. The Cu tubing compacted with the unidirectionally aligned CNF was successfully used for the fabrication of CNF reinforced Cu composite by liquid infiltration process, as shown in Fig. 5. Table I shows the tensile property of the CNF-reinforced pure Cu composite fabricated in the present study. Considering that the tensile strength of rolled, pure Cu is approximately 220 MPa, the tensile strength of the Cu composite reinforced with aligned CNFs was improved by approximately 2 times.

From the present study, it was demonstrated that the severely entangled CNF could be aligned unidirectionally by using mechanical drawing process. The tube packed with CNFs or other nano-sized fibers could be used as a raw material for composite fabrication. There is also strong possibility to use this metal tubing as a filter material with nano-sized porosity.

TABLE I Tensile properties of CNF reinforced Cu composite

Materials	YS (MPa)	UTS (MPa)	Tensile elongation (%)
CNF reinforced Cu composite	452	476	6.5

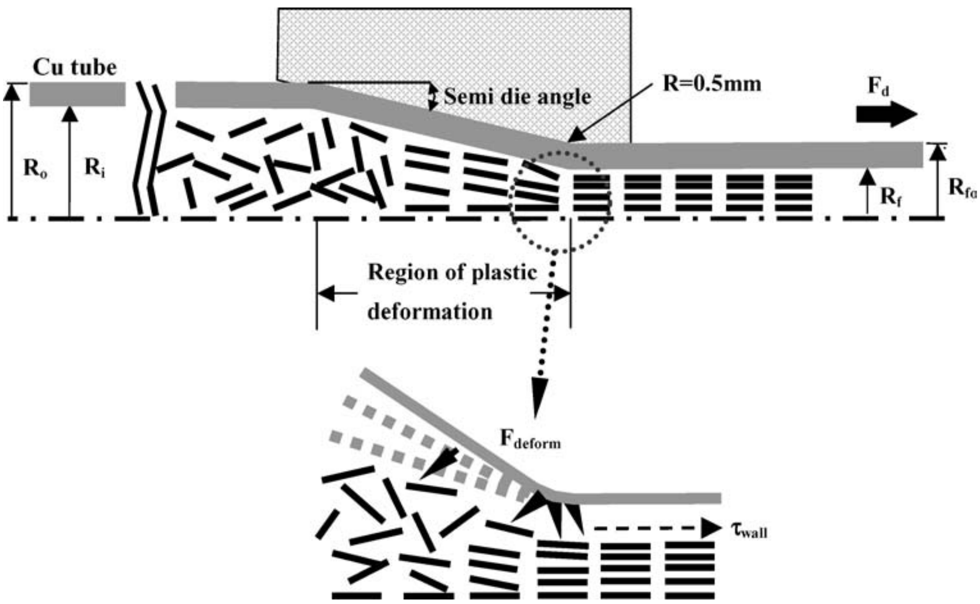

Figure 3 The schematic diagram of the drawing process of the Cu tubing packed with CNFs.

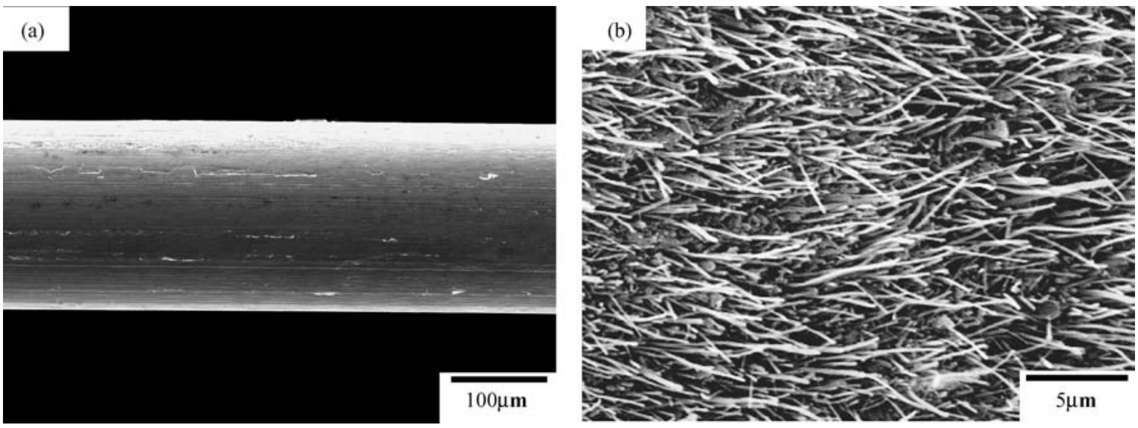

Figure 4 SEM micrographs of the (a) drawn Cu tube packed with CNFs and (b) cross-section of the tube, respectively.

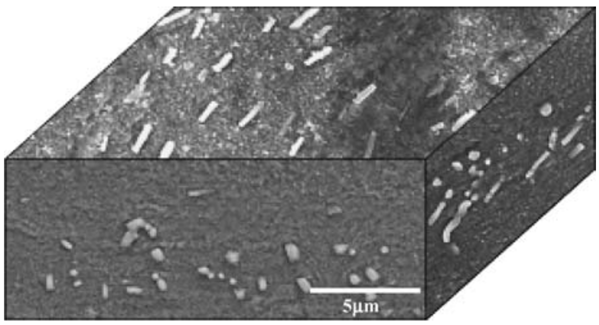

Figure 5 SEM micrographs of the cold-rolled CNF reinforced Cu composite fabricated by liquid infiltration process, showing the unidirectionally aligned CNFs.

Acknowledgement

This research was supported by a grant (code #: 03K1501-00710) from 'Center for Nanostructured Materials Technology' under "21st Century Frontier R&D Programs" of the Ministry of Science and Technology, Korea.

References

1. S. ARAI and M. ENDO, *Electrochem. Commun.* **5** (2003) 797.
2. E. HAMMEL, X. TANG, M. TRAMPERT, T. SCHMITT, K. MAUTHNER, A. EDER and P. PÖTSCHKE, "Carbon Nanofibers for Composite Applications" *Carbon* (2004), in press.
3. A. N. ATTIA, *Mater. Design* **22** (2001) 451.
4. S. S. KIM, D. H. KIM, S. Z. HAN, S. K. LEE and D. H. HA, "Development of Carbon Nanofiber Reinforced Cu Matrix Composites using Liquid Pressing Process," 2003 Annual Meeting, The Korean Society for Composite Materials, Daejeon, Korea (2003).
5. P. POULIN, B. VIGOLO and P. LAUNOIS, *Carbon* **40** (2002) 1741.
6. B. AVIZUR, R. WU, S. TALBERT and Y. T. CHOU, *J. Eng. Ind.* **104** (1982) 293.

Received 14 April 2004
and accepted 4 April 2005