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Fabrication and Mechanical Properties of a Micro/Nanoscale Hybrid Composite

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Abstract. Microscale SiC particles with multiwalled carbon nanotubes grow on were filled in epoxy resin to construct multiscale hybrid composite. Static and dynamic compressive responses of composites were investigated. A dynamic experimental technique, split Hopkinson press bar (SHPB), was introduced to study the constitutive laws of materials at high strain rates. SEM observations show good dispersion of carbon nanotubes and SiC particles, but bad link between SiC particles and carbon nanotubes exists. Experimental results of static and dynamic tests verify that increase of strength happens with SiC/CNT as reinforcement Increment of strength under dynamic loading is bigger than under static loading.

Keywords. Carbon nanotube, hybrid composite, fabrication, compressive test, dynamic test.

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1 Introduction

The discovery of carbon nanotubes [1] (CNTs) has provoked enormous interests in their fundamental behavior and potential applications due to their exceptional properties. Researchers have envisioned CNTs as the most viable candidates to dominate the coming 21st century revolution in nanotechnology. Carbon nanotubes can be thought of as rolled up sheets of graphite that are sometimes capped on each end, with structures that vary depending on the conditions under which they are synthesized. In the last decade, CNTs reinforced nanocomposites have attracted numerous attentions.

CNTs' extraordinary properties, high strength and ductility, low density, huge aspect ratio, electrical conductivity of copper and silicon, thermal conductivity of diamond, the chemistry of carbon, size of DNA, make them attractive candidates as a reinforcement filler material. CNTs offer an appealing mechanism to dramatically improve both strength and stiffness characteristics, as well as add multifunctionality to polymer based composite systems. A lot of carbon naotube based composite films [2, 3], fibers [4–6] were made. However, fabrication of CNTs reinforced composites is still very complicated. There are several critic issues need to be overcome. The CNTs are most always organized to aggregates, which make it difficult to disperse them homogeneously into a matrix. Alignment of CNTs is also a problem, although it is not always beneficial. Alignment is a good way to maximize reinforcement in fibers, but it need to be avoided in bulk materials because of its very anisotropic mechanical properties. Perfect CNTs are atomically smooth and the surfaces are inert. There is not enough bonding between CNT walls and matrixes.to transfer forces. So there exist weak interfaces. But as fabricated CNTs, especially multiwalled carbon nanotubes (MWNTs) usually are not perfect. Function groups of polymer can graft to the defects of CNTs to form strong interface. Surface chemical modification is an active approach to enrich interfacial strength [7]. The extremely high production costs obstruct CNTs based skyscraper and aircraft being true. Recently research on multiscale hybrid materials with CNTs included has arisen [8–11]. It has been proved that the existence of carbon nanotubes could greatly improve the interfacial strength between fiber or particle and matrix by local stiffening near the interfaces of fiber/matrix or particle/matrix [8, 9]. Most of them focus on fabrication of materials and static tests. To my knowledge, dynamic compressive experimental results for this kind of composite are rarely.

Herein, we report a fabrication process of micro/nanoscale hybrid composite, in which SiC particle of average 4 micron with multiwalled carbon nanotubes grow on were mechanically blent with epoxy resin. Static and dynamic compressive tests were made to study compressive response of both specimens with and without SiC/CNT.

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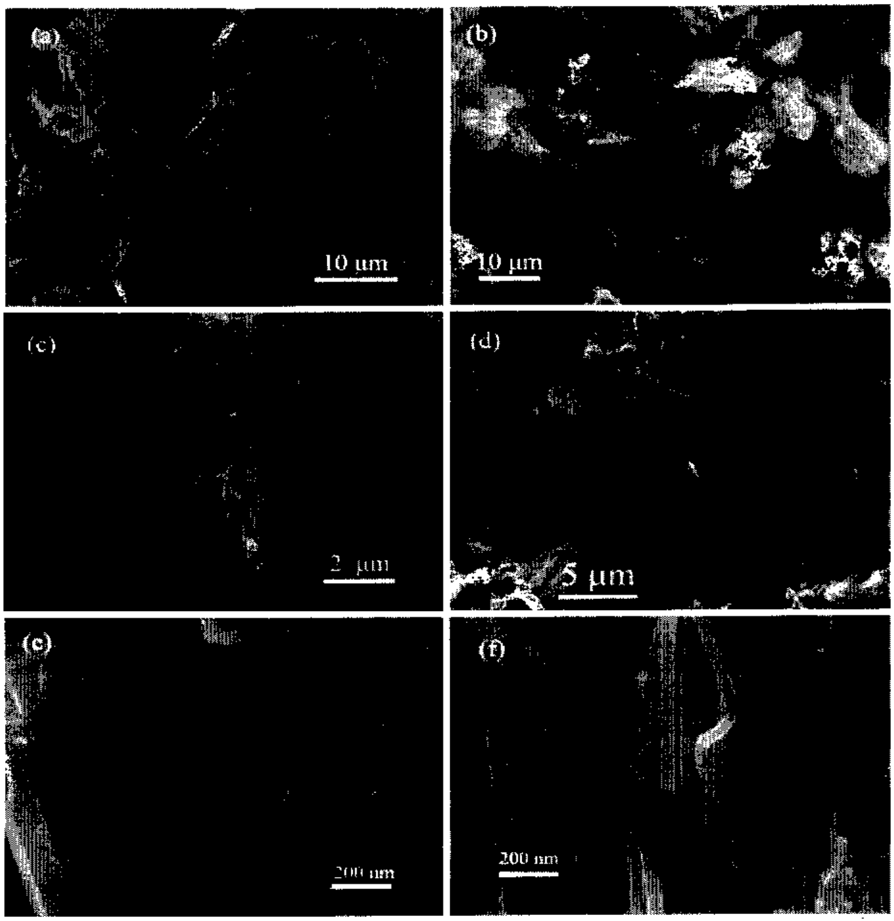

Figure 1. SEM images of SiC particle (a) before and (b–e) after carbon nanotubes growth, and (f) enlarged SEM image of carbon nanotubes on SiC particle.

2 Fabrication

Silicon carbide has high strength, low density (40% the density of steel), excellent corrosion resistance in most chemical environments, very good thermal shock resistance, low thermal expansion, high current density and very high hardness. It is widely used in wide bandgap power electronics and ceramics. A typical floating-catalyst chemical vapor deposition (CVD) method was used to grow aligned multi-walled carbon nanotubes on the surface of SiC particles at about 750°C in a quartz tube reactor. Compared with arc-discharge and laser methods, CVD is a simple and economical technique for mass synthesizing CNTs at low temperature and ambient pressure. Figure 1 (a) shows a scanning electronic microscope (SEM) image of as-received SiC particles with an average size of 4 μm. These particles are irregular in shape and have several flat surfaces. Figure 1 (b) displays a low magnification SEM image of typical product. Carbon nanotubes are aligned and perpendicular to some of flat surfaces. Weight proportion of SiC particle and carbon nanotube is about 3 ± 0.2 : 1. They are 10–20 μm long. Figure 1 (c) and (d) are enlarged images of carbon nanotubes grown on SiC particles. Figure 1 (c) shows carbon nanotubes grow on only one side of penny shaped SiC particle. Figure 1 (e) shows enlarged image of interfacial zone of SiC particle and carbon nanotubes. Figure 1 (f) is enlarged image of carbon nanotubes on SiC particles. They are 10–50 nm in diameter.

For effective load transfer, the length of CNTs, L_c, has to meet demand of critical aspect ratio, $L_c/\sigma_{\max}$, which is defined by shear lag theory:

$$L_c/D = \sigma_{\max}/2\tau_c, \tag{1}$$

where $\sigma_{\max}$ is the maximum tensile strength of the CNTs, D the diameter of CNTs, and τ_c the interfacial shear strength or maximum matrix shear strength. Taking the classic polymer value 50 MPa as interfacial strength, experimental tensile strength value 50GPa as maximum tensile strength, the critical aspect ratio is 500 : 1. The CNTs used in this experiment basically meet the requirement of aspect ratio.

3 wt% as grown SiC/CNTs were first dispersed in the liquid epoxy (R1080, Resoltech). The suspension was mechanically stirred for 40 minutes and degassed for 10 min-

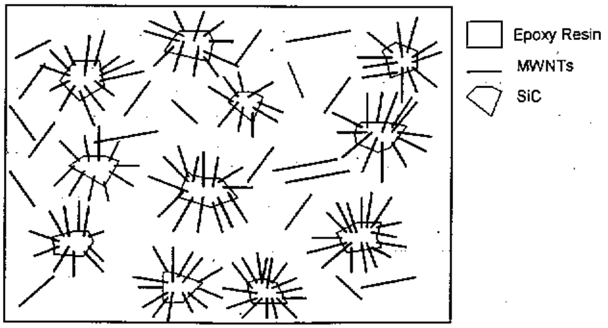

Figure 2. Illustration of reinforcement from MWNTs and SiC particles.

utes. After adding hardener (R1083, Resoltech), the suspension was stirred for 10 minutes and degassed for 10 minutes again. At last the solution was fallen over the cylindrical mold, vacuumed for 3 minutes. The specimens were first solidified at room temperature for 24 hours, then increased temperature to 60°C and held for 12 hours. At last temperature was increased to 90°C and held for 24 hours. After curing, the specimens were cut and polished. The dimensions of cylindrical specimens are 10 mm in length and 10 mm in diameter. Pure epoxy specimens were made for comparison. Figure 2 shows the schematic diagram of SiC/CNT reinforced hybrid composite.

3 Mechanical Experiment

Both static and dynamic compressive tests were made to study compressive response of both specimens with and without SiC/CNTs reinforced. Static compressive tests were conducted on material testing machine (MTS 810) at a loading rate of 0.01 mm/s. Split Hopkinson press bar (SHPB) is a commonly used experimental technique to study constitutive laws of materials at high strain rates [12]. It is shown in Figure 3. The forces and the velocities at both faces of the specimen are given by

$$\begin{aligned} F_i(t) &= S_b E(\varepsilon_i(t) + \varepsilon_r(t)), \\ F_o(t) &= S_b E \varepsilon_t(t), \\ V_i(t) &= c(\varepsilon_i(t) - \varepsilon_r(t)), \\ V_o(t) &= c\varepsilon_t(t), \end{aligned} \tag{2}$$

where F_i, F_o, V_i, V_o are forces and particle velocities at the interfaces, S_b, E and c the cross sectional area of the bars, Young's modulus, and the longitudinal wave speed, and ε_i, ε_r, ε_t the incident, reflected, transmitted waves respectively at the bar-specimen interface. 10 m/s was chosen as impact speed for dynamic compressive tests.

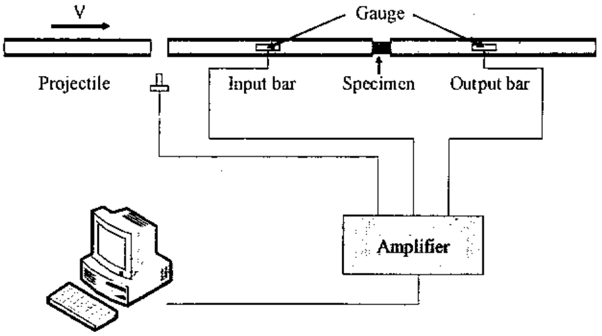

Figure 3. Schematic of split Hopkinson press bar (SHPB) setup.

Figure 4 shows the typical static and dynamic compressive stress – strain curves of the specimens with and without SiC/CNT. The upper two curves represent dynamics test

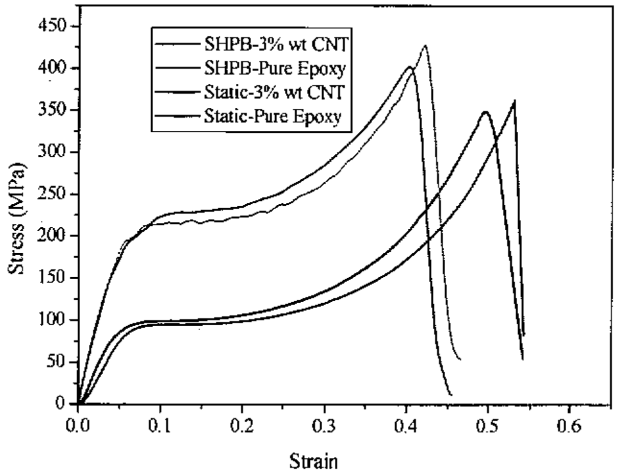

Figure 4. Typical experimental results of static and dynamic compressive tests for both specimens with and without SiC/CNTs reinforced.

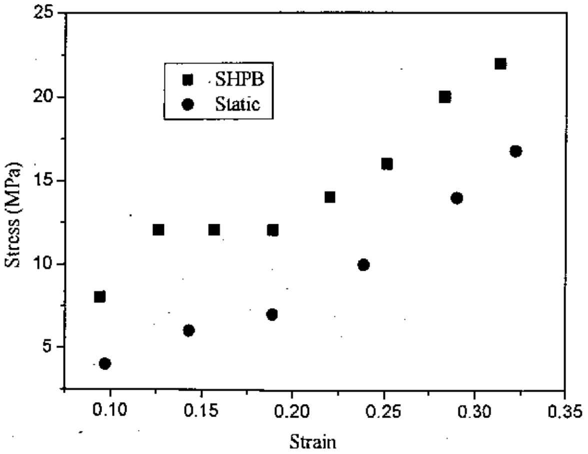

Figure 5. Increments of strength of both static (round) and dynamic (square) tests.

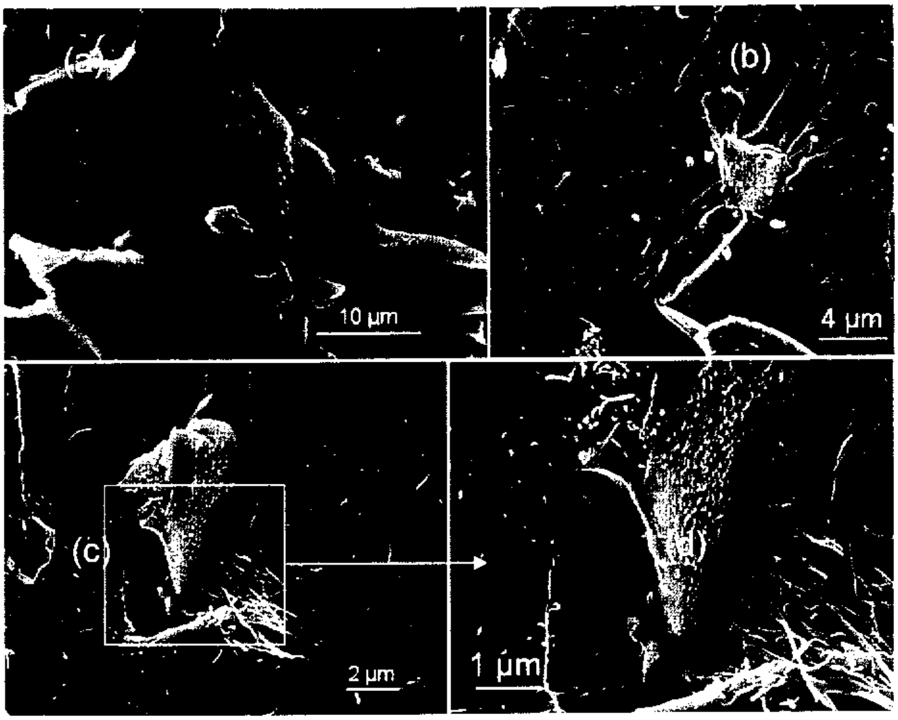

Figure 6. SEM micrograph of fracture surfaces after dynamic compression; (a), (b) dispersion of carbon nanotubes, (c) carbon nanotubes were token from SiC, (d) enlarged image of (c).

results, the lower two curves represent static test results. Young's modulus increases (from 1708 MPa to 2202 MPa) by 30% with 3 wt% SiC/CNT from the static test. But for dynamic test, the equivalent Young's modulus hardly changes. The experimental results also show that ultimate stress and ultimate strain under dynamic test are smaller than static test. For the same type of loading, although yield strength of composite with reinforced is bigger than without, the case of ultimate stress and ultimate strain are opposite. Increments of strength of both static and dynamic compressive test are shown in Figure 5. It is obvious that increment of strength under dynamic loading is bigger than under static loading.

Dynamic fracture surfaces of 3 wt% SiC/CNT specimen are shown in Figure 6. It can be seen that good dispersion of SiC/CNTs in matrix is achieved. SiC particle is used for carrying carbon nanotube in experiments. Figure 5 (c) shows that part of carbon nanotubes grown on SiC particle were taken off, which means bad link between SiC particle and carbon nanotubes exists. Figure 5 (d) is enlarged image of square area in Figure 5 (c).

4 Conclusion and Discussion

A micro/nanoscale hybrid composite was made by mechanically stirred and degassing. Good dispersion of CNTs is achieved due to the existence of SiC particle, which acts as carrier. Static and dynamic compressive tests were made to study compressive response of both specimens with and without reinforcement. The slight increment of yield strength can come down to lower weight fraction of reinforcement weak link between SiC particle and CNTs and compressive loading. Carbon nanotube can take much less compression than tension. The joint strength of particle and carbon naontube needs to be improved. More experiments of composite with higher weight fraction of reinforcement should be further made.

Acknowledgments

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