Release of particles by abrasion of CNT composites using a belt sander

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Abstract. There have been many reports on the effect of exposure to nanomaterials such as titanium dioxide, silver, and carbon nanotube (CNT) on human health. Several experiments have examined the abrasion of CNT composites, in which CNT nanoparticles are embedded within a resin or rubber matrix, yielding varying results. Separate study of free CNTs and CNT nanoparticles in relation to health is important due to the different physicochemical characteristics of the two types of material. This study investigated the abrasion of CNT composites using a belt sander inside an enclosed chamber, with variation in the applied load and belt sander speed. At lower speeds, the population of particles with diameters of ~100 nm was observed to increase (cf. mode values of ~10 nm), and we found a relationship between the amount of the raising dust and the abrasion conditions. From these results, we propose a robust and widely applicable method to create particles of nanomaterial-containing composite materials of various types in order to conduct accelerated exposure assessment studies.

1. Introduction

Several experiments examining the wear of carbon nanotube (CNT) composites have been reported [1, 2]. For example, Wohlleben et al. performed a wear test using a Taber Abraser [1], finding that neither free nor overhanging CNT particles were generated from a CNT-containing composite material; meanwhile, Golanski et al. conducted Taber tests using similar apparatus [2] — in this report, we found that although CNTs were not discharged from the polymer, there was evidence of them collecting on the surface during the test.

There are several other types of equipment available for wear testing besides Taber Abrasers: one example is a DIN Abrasion Tester, in which a grinding cloth is wound around a revolving drum; samples are then pressed against the cloth to induce wear with a speed of 3.1 m/s [3]. In this study, we investigated the wear of CNT composites using a modified version of the DIN abrasion test involving a belt sander. While Taber and DIN abrasion testing is generally designed to quantify the wear depth of the materials, these methods are not usually used to analyse the dust generated by wear. First, the loading energy is usually too weak to detect nanomaterials in a resin or rubber. Second, the shaving speeds of these testing methods is quite slow, and thus little dust is generated (ultimately, this class of equipment was never designed with the intent of generating large amounts of dust).

To address the limitations of these testing methods, we used a commercial belt sander that allows the user to change the load and speed. This type of equipment is widely used for shaping processes in chemical factories, and thus the resulting base material wear will be somewhat similar to what would occur during industrial processing.

2. Methods
2.1. Test pieces
The samples used were composed of polycarbonate composite materials prepared without (PC0) and with 3% CNT (PC3). The CNTs distributed in the polycarbonate were multi-walled (CNano Flotube9000). The samples were manufactured by general distribution and kneading methods in a resin factory. All pieces were made from a commercial polycarbonate master batch and requested to Marubeni Information Systems to shape plate piece (10 cm²).

2.2. Exposure Assessment Chamber
The belt sander was located in a chamber used for exposure assessment experiments that possessed a depth of 1.08 m, width of 0.8 m, and height of 1.64 m (Figure 1) [4]. The air in the chamber was controlled and isolated from the outer environment by two different types of HEPA filters, and an electrically conductive film was used to seal the inner wall to prevent absorption of abrasion dust. A clean airflow was directed from the upper filtration box to filters on both sides of the bottom of the chamber. All manual operations inside the chamber were conducted using either the upper or lower pairs of rubber gloves shown in Figure 1, and a power adapter was located inside the box. Prior to abrasion testing, we ensured that the ambient particle concentration in the chamber was under 100 particles/cm³ by ventilating the chamber; once an acceptable background particulate level was reached, the ventilation system was switched off and the abrasion test initiated. Different particle detectors, FMPS (Fast Mobility Particle Sizer, Model 3091, TSI), SMPS (Scanning Mobility Particle Sizer, Model 3910, TSI), and OPC (Optical Particle Sizer, OPS 3330, TSI) were connected through holes in the ports in the chamber wall.

![Figure 1. Photograph of the exposure assessment chamber containing the belt sander.](image)

2.3. Belt sander abrasion testing
PC0 and PC3 samples were abraded using the belt sander; the sander was equipped with a 90 mm × 915 mm belt with a P80 grit size (aluminium oxide grains, average abrasive grain diameter of 177 μm), and the standard operating speed used in the experiments was 9.1 m/s (Figure 2). Each sample was attached to the bottom of a standard 500 g weight using double-sided tape. The weight was held
on a plate seat by a fixing handle specifically designed for this purpose by our group, as shown in the schematic diagrams in Figure 3 (top diagram, overhead view; bottom diagram, horizontal view). The design of the fixing stage allows for changes in the grid size, belt rotation velocity, and loading weight, depending on the type and dimensions of the material being tested.

Figure 2. Custom sample fixing stage installed on the belt sander.

Figure 3. Schematic diagram of the sample fixing stage designed for use with a belt sander.
Each sample was subjected to wear for 10 s, after which the particulate distribution within the enclosed chamber atmosphere was measured using the FMPS. Fine particles were collected from the chamber by a glass fibre filter using a mini pump (MP-Σ 300, Shibata Kagaku) with a flow rate of 0.3 L/min over a period of 13 min. Additionally, abraded powder stuck to the belt sander was collected using carbon tape in order to analyse the particle shapes, and to distinguish elemental CNT from embedded CNT with polycarbonate. Scanning electron microscopy (SEM) was used to observe the surfaces of the samples after testing, the fine particles from the filter, and the abraded powder on the carbon tape. Different abrasion conditions were used for each experiment, as shown in Table 1. In the second experiment, the weight used to apply the load to the sander was increased from 500 g to 1000 g, and in the third experiment, the belt speed was decreased from 9.1 m/s to 7.6 m/s (500 g load).

| Table 1. Experimental conditions. |
|-----------------------------|
| load (g) | grit size | speed (m/s) |
| Experiment 1 | 500 | P80 | 9.1 |
| Experiment 2 | 1000 | P80 | 9.1 |
| Experiment 3 | 500 | P80 | 7.6 |

3. Results

3.1. Chamber Atmosphere

Prior to measurement, the background air level in the chamber was measured. It was confirmed that an impurely particle in the chamber decreases by ventilating through the HEPA filter unit located at the top of the exposure chamber (Figure 1). The data in Figure 4 indicate the changes in the particle concentration in the chamber after the filter, showing that the concentration decreased dramatically over a period of 1,000 s, from 500 particles/cm³ to just several tens of particles/cm³. Prior to each abrasion test, we confirmed that the background level had been reduced to under 100 particles/cm³ by ventilation.

![Figure 4. Changes in chamber particle concentration on ventilation.](image)

3.2 Abrasion by belt sander
During the sanding experiments, the change in the total particle concentration was measured over time. Figure 5 shows the results of Experiments 1–3 (Table 1). In the period from 0 s to 60 s, the belt sander was switched on, but not placed in contact with the sample; after the initial 60 s period, abrasion was initiated. In addition to measuring the total particle concentration, the particle grade scale (grain size) was also measured during sanding, as shown in Figure 6. The results of Experiment 2 show that the concentration of sanded particles produced under these conditions was three times higher compared to that of Experiments 1 and 3. From these observations, we conclude that the load weight had a greater effect on particle generation than belt speed.

Figure 6 shows the average particle size distributions for Experiments 1–3 during the period from 60 s to 120 s. While the pattern of particle size distribution was found to be similar for each experimental dataset, the results from Experiment 2 (especially PC3) show a secondary concentration peak at around 30 nm diameter. In the case of Experiment 3, a slight uptick in the distribution was seen at around 100 nm diameter. In general, the composite samples that did not contain CNTs (PC0) were found to produce higher particulate concentrations soon after contact was made between the sample and the sander.
Figure 6. Size distribution of sanded particles. PC0 and PC3 = Experiment 1; 1.0kg suffix = Experiment 2; 7.6m/s suffix = Experiment 3.

SEM was used to analyse the abraded particles, and confirmed that the composite produced dust during the experimental. The difference in each experiment was not seen by particles. There were a lot of particles with size bigger than 10 μm. Elemental CNTs, CNTs overhanging from polymer, and embedded CNTs were not identified among the polycarbonate dust particles (Figure 7), and while several white dots were observed on the surface of the particles (as seen in the Figure 7, lower right), it was not confirmed that CNTs were present.

Figure 7. Scanning electron micrographs of the abraded composite powder.

4. Discussion
The results confirm that abrasion by the belt sander led to an increase in particulate concentration, with particles generated from both PC0 and PC3 composites increasing dramatically soon after contact between the belt and the sample. An increase in load led to an increase in the rate of particle generation, while the rate decreased with a decreased belt speed. A relation was found to exist between the amount of generated dust and the abrasion conditions. It was noted that a combination of increase weight loading and presence of CNTs (PC3) led to an increase in the population of particles with ~30 nm diameter; in contrast, high belt velocity increased the population of particles with ~100 nm particle diameter from both species of composite, PC0 and PC3. These results indicate that more high velocity effect a breaking of composites. To assess the exposure dose, we should find suitable load weight and velocity by using belt sander machine.
The presence of dust particles was confirmed by SEM observation. In future studies, after optimising the sanding parameters, we will measure the particle concentration and diameter distribution, followed by SEM analysis to distinguish between elemental, embedded, and overhanging CNTs.

5. Conclusions

When the belt speed was slowed, we observed that the population of particles with diameters of \(~100\) nm increased, the origins of which require further investigation. We will make a similar experiment by the other loads and the other speed from now on. A standard exposure assessment method for composite materials — including those containing nanomaterials — should be established using Taber Abraser, DIN Abrasion, belt sanders, or some other method. From this study, we also conclude that control of the background particle level is crucial for the chamber method.

From these experiments, it is clear that there are a number of challenges associated with CNT detection. There is some CNT including metal. We are trying the way to determine the quantity of the metal content which is included by wear in the trash which occurred and determine the quantity of CNT. This technique will support a SEM method to distinguish CNT variations after wearing.

References

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