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# Particle release from single-wall and multiwall carbon nanotubes in polystyrene-based composites during grinding

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Abstract. The aerosol particles released during the grinding of polystyrene (PS)-based composites with well- and poorly dispersed single-wall carbon nanotubes (CNTs) and multiwall CNTs were measured using real-time aerosol measuring instruments. Increases in the concentration of aerosol particles were recorded during the grinding of the samples. However, similar increases were observed even when CNT-free polystyrene was ground. Electron microscopic analysis of the released particles revealed that particles with protruding CNTs were observed for the well-dispersed CNT-PS composites, but free-standing CNTs were not found. On the other hand, particles like agglomerated CNTs were found for the poorly dispersed CNT-PS composites.

#### 1. Introduction

Carbon nanotubes (CNTs) exhibit unique properties, and are promising candidates as filler materials in composites. Although composites based on CNTs are expected to be used in a wide range of industrial applications and consumer products, the potential impact of CNTs on human health remains a concern. Therefore, in order to control exposure to CNTs, an awareness of the potential release of CNTs throughout their life cycle is important.

In our previous study [1], we investigated the particle release during the grinding of polystyrene (PS)-based composites with well-dispersed single-wall CNTs (SWCNTs). In this study, we have broadened the range of sample types to include PS-based composites with both well- and poorly dispersed SWCNTs and multiwall CNTs (MWCNTs).

#### 2. Methods

#### 2.1. Test samples

We used SWCNTs (AIST/TASC Super-growth SWCNTs) with a tube diameter of approximately 3 nm and a carbon purity of 99.9% [2], and MWCNTs (Nanocyl NC7000) with a tube diameter of approximately 10 nm and a carbon purity of 90% [3]. As one of the most widely used plastics, PS was used as a composite matrix. Although PS has poor heating resistance and impact durability, it is expected that the addition of CNTs can modify its properties.

Five types of test plate samples were prepared: PS (CNT-free), 5wt% well- and poorly dispersed SWCNT-PS composites, and 7.5wt% well- and poorly dispersed MWCNT-PS composites. The well-

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dispersed CNT–PS composites were prepared as follows: (1) CNTs were added into an aqueous solution with 1wt% sodium dodecyl sulfate (SDS) and dispersed using a jet mill (JN20, Jokoh, Tokyo, Japan); (2) PS dissolved in toluene was mixed with 1wt% SDS aqueous solution using a mixer (Filmix Model 56-50, Primix, Osaka, Japan) to obtain a latex solution with 4wt% toluene; (3) the CNT and latex solutions were mixed using a magnetic stirrer, and the mixture was dripped into 2-propanol; (4) precipitating composites composed of PS, and well-dispersed CNTs were obtained by filtration and dried under vacuum to remove 2-propanol and water; and (5) the composite material was formed into a plate by pressing. On the other hand, the poorly dispersed CNT–PS composites were prepared as follows: (1) as-produced CNTs were directly mixed with PS using a compounding machine (DSM Xplore, Geleen, The Netherlands); (2) the compounded composites were pelletized; and (3) the pellets were pressed into a plate.

Figure 1 shows photographs of the test plate samples. For the poorly dispersed SWCNT-PS composite, agglomerated CNTs were visually observed, whereas the other samples were entirely black.



Figure 1. Photographs of test plate samples.

#### 2.2. Experimental setup

The composites were ground by hand using a micro-grinder in a conductive-antistatic plastic box that had an air supply opening with a high efficiency particulate air (HEPA) filter (see figure 2). The rotating speed was set at 20,000 rpm; grinding time was 30 s; and the air change rate was approximately 1 min<sup>-1</sup>.

Prior to the test, purified air was passed through the box to eliminate background particles, and the aerosol particles released into the box by the grinding process were then measured using real-time aerosol measuring instruments: a scanning mobility particle sizer (SMPS, model 3936L75, TSI Inc., USA), an optical particle sizer (OPS, model 3330, TSI Inc., USA), and a condensation particle counter (CPC, model 3776, TSI Inc., USA). Each sample was tested at least three times.

In addition, the released aerosol particles were collected on Nuclepore membrane filters (25-mm diameter, 0.080-µm pore size, Nomura Micro Science Co., Ltd., Japan) and holey carbon film-coated transmission electron microscope (TEM) grids (Quantifoil R1.2/1.3 on 200 mesh Cu, Agar Scientific, Stansted, UK) with a TEM grid holder (Mini Particle Sampler, MPS, Ecomesure, Janvry, France) [4, 5] for analysis using a scanning electron microscope (SEM; Quanta 250 FEG, FEI Company, USA) and a TEM (JEM-1010, JEOL, Tokyo, Japan), respectively.



Figure 2. Schematic of experimental setup.

## 3. Results and discussion

### 3.1. Real-time aerosol measurements

Figure 3 shows the particle size distribution of the released aerosol particles measured using real-time aerosol measurements. The results for idle running of the micro-grinder (without grinding) indicate that particles were generated from the micro-grinder itself. Increases in the concentration of nanometer- and micrometer-sized aerosol particles were recorded during the grinding of the samples, regardless of the sample type used, and similar increases were observed even when the CNT-free PS was ground. It is likely that the nanometer-sized particles were volatile particles released by frictional heat produced during the grinding of the composites [1].



**Figure 3.** Number-based particle size distributions of released particles measured using SMPS and OPS.

### 3.2. Electron microscopic observation

Figures 4–8 show SEM and TEM images of particles released into the air during the grinding of the test samples. Although particles with protruding CNTs were observed for the well-dispersed SWCNT– PS and MWCNT–PS composites (see figures 5 and 7), free-standing CNTs were not found. However, for the aerosol particles released from the poorly dispersed SWCNT–PS and MWCNT–PS composites, particles like agglomerated CNTs were found (see figures 6 and 8).



Figure 4. SEM and TEM images of particles released into the air during the grinding of CNT-free PS.



**Figure 5.** SEM and TEM images of particles released into the air during the grinding of welldispersed SWCNT–PS composite.



**Figure 6.** SEM and TEM images of particles released into the air during the grinding of poorly dispersed SWCNT–PS composite.



**Figure 7.** SEM and TEM images of particles released into the air during the grinding of welldispersed MWCNT–PS composite.



**Figure 8.** SEM and TEM images of particles released into the air during the grinding of poorly dispersed MWCNT–PS composite.

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