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Properties and microstructure of expandable graphite particles pulverized with an ultra-high-speed mixer

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Abstract

The expandable graphite (EG) particles were pulverized to achieve different and smaller sizes using an ultra-high-speed mixer. The microstructure of particles was observed by a scanning electron microscope (SEM). The as-received EG particles showed an irregular flake shape. With the increase of the mixing time, the EG particles tended to be circular and the collapses and cracks in the EG surfaces appeared, and their average diameter and average area rapidly reduced. At the same time, their expansion volume after thermal treatment greatly decreased, resulting from the reduction of particle sizes and the direct release of the oxidant inside the EG particles instead of exfoliating the particles. The expanded EG particles revealed the typical wormlike structure.

Keywords: Expandable graphite; Expanded graphite; Fine particle; Microstructure; Particle size

1. Introduction

Expandable graphite (EG) is a graphite intercalation compound in which some oxidants, such as sulfuric acid, potassium permanganate, etc., are inserted between the carbon layers of the graphite [1–4]. When experienced a heat source, EG, occupies hundred times its initial volume and generates a voluminous structure, thus providing fire-retardant performance for the polymeric matrix [4,5]. Some studies implied that EG can produce good fire-retardant properties for some polymers, such as polyolefins [6,7], polyurethane foam [5,8,9], coating [10], etc. In addition, EG after expansion can be used as biomedical materials due to its pore structure and absorptive capacity [3]. And for its high electrical conductivity and a unique layered nano-structure, it was compounded with some polymers, such as polymethyl methacrylate (PMMA) [11], poly(styrene-co-acrylonitrile) [1], etc., to fabricate the so-called nano-composites with excellent electrical conductivity. In our previous study [5], EG can efficiently improve the fire-retardant properties of high-density rigid polyurethane foam (RPUF) as a halogen-free fire-retardant additive. However, in the open literature, little attention has been paid to the EG pulverization, and the microstructures of finer EG particles before and after expansion, and their influences on the flame behaviors of polymers, etc. Usually a material as a filler for polymers should be fine and uniformly dispersive, that is, the finer and the more uniformly dispersive and distributive filler particles bring out better properties of composites [12–15]. As sheet fire-retardant additives, the fine and uniform dispersion of EG is believed to give a considerable effect on the fire-retardant properties of RPUF. In this series studies, accordingly, the EG particles were crushed into fine ones, and subsequently added to RPUF for further improvement of fire behaviors. In the present work, an ultra-high-speed mixer, which was specially designed for dispersion of nano fillers in polymers depending on its high shear and intense mixing [15], was used for pulverization of the EG particles. After pulverization, EG particles with various sizes were obtained. The structure before and after expansion, size distribution and expansion volume of these particles have been investigated.

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2. Experimental

2.1. Materials

Expandable graphite (Model KP9932300) was purchased from Haida Graphite Co. (QingDao, China). The manufacturer specifies the following properties: ash, 1.0%; moisture, 1.0%; weight loss after expansion, 15%; pH value, 3.0; expansion rate, 300 ml/g.

2.2. Pulverization of expandable graphite particles

EG particles were broken up into smaller ones using an ultra-high-speed mixer whose configuration is shown in Fig. 1. This mixer can provide high speed (as high as 6000 rpm) and high shear force, with a special rotor designed to cut glomerate particles. It has been demonstrated that this mixer can effectively reach dispersion of nano-$CaCO_3$ in polypropylene, and lead to a considerable increase in mechanical properties of nano-$CaCO_3$/polypropylene composite [15]. The shear mixing time is 4 and 13 min, and the EG particles thus obtained are signed as EG4 and EG13 for simplicity, respectively. Correspondingly, the initial EG particles (as received) are referred to as EG0.

2.3. Characterization

The morphology of EG particles before and after expansion was observed by a JSM-9600 (JEOL, Japan) scanning electron microscope (SEM). The EG particles were suspended in water by stirring, and then some suspension mixture was dripped on the sample stage. After the water volatilizes completely, the surface of the sample was coated with a conductive material and then used for observation. The accelerating voltage was 20 kV. Sizes of EG particles were estimated using SEM micrographs and an image analysis system (some quite small scraps in the EG4 and EG13 were neglected).

The expansion multiple of different EG particles was measured after they were expanded at 950 ± 10 °C for 15 s according to GB 10698-89 (China standard).

The apparent density of various EG particles was tested according to ASTM D2854.

Thermogravimetric analysis (TGA) was carried out in air at a heating rate of 10 °C/min using a SEIKO EXSTAR6000 instrument thermogravimetric analyzer. In each case, a 4.0 mg sample was examined at an air flow rate of 100 L/min at temperatures ranging from room temperature (25 °C) to 700 °C.

3. Results and discussion

3.1. Morphology of EG particles

Fig. 2 shows the SEM micrographs of EG particles. All the EG particles assume irregular flake shape. And the size of processed EG particles (Fig. 2b and c) is much smaller than that of the unprocessed one (Fig. 2a). With the increase of the mixing time, the EG particles become circular, and their size smaller (see Fig. 2c). Fig. 3 shows the surface microstructure of

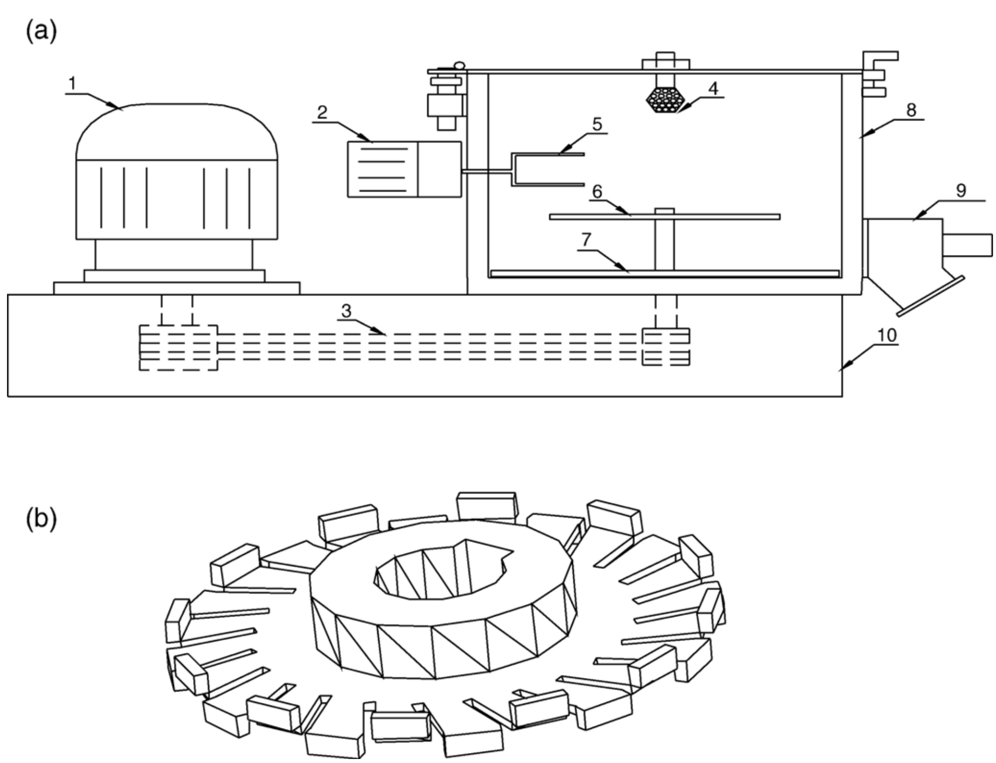

Fig. 1. Structural illustration of ultra-high-speed mixer: (a) overall schematic, (b) shape of upper gear type blade wheel. (1) Main engine, (2) Engine for cutting tool, (3) Driving belt, (4) Atomization nozzle, (5) Cutting tool, (6) Upper blade wheel, (7) Lower blade wheel, (8) Mixing kettle, (9) Discharge, (10) Base.