Composites Part B 141 (2018) 92–99

Contents lists available at ScienceDirect

Composites Part B

journal homepage: www.elsevier.com/locate/compositesb

Effect of filler particle shape on plastic-elastic mechanical behavior of high density poly(ethylene)/mica and poly(ethylene)/wollastonite composites

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ARTICLE INFO

Keywords:
HDPE
Mica
Wollastonite
Impact testing
Tensile testing
Vibration damping
SEM
Thermal analysis

ABSTRACT

It was found in this study that both fillers (mica and wollastonite) trigger an increase in Young's modulus of elasticity with increasing filler concentration in a HDPE composites matrix. In the case of HDPE/mica the same improvement was also found for the upper yield point vs. filler concentration dependencies indicating higher stiffness. However, for the HDPE/wollastonite composites the opposite trend was observed, i.e. a decrease of the upper yield point and strain at break. These findings were also confirmed by mechanical vibration damping testing where there was found a more intense shift of the first resonance frequency peak position to higher frequencies with increasing filler concentrations for HDPE/mica in comparison to HDPE/wollastonite composites. Both composites exhibited decreasing strain at break with increasing filler concentration indicating a more brittle mechanical behavior in comparison to the virgin HDPE polymer matrix. However, for HDPE/wollastonite composites at 5 w. % filler concentration a 15% increase in the magnitude of the strain at break was found indicating an increase in ductility at 50 mm/min deformation rate. Fracture toughness measurements show, that both studied fillers function as the stress concentrators in the HDPE polymer matrix, which was reflected in the exponentially decreasing dependencies of the fracture toughness vs. filler concentrations. SEM analysis of the fracture surfaces show typical elongation bands of high plasticity deformation regions characteristic of typical shearing bands, interpenetrated with cavities created around filler particles. Thermal analysis data showed for HDPE/mica a strong increase of the crystallinity with increasing filler concentration, however in the case of HDPE/wollastonite the opposite effect of a higher amorphous polymer phase content was found.

1. Introduction

At the present time, the engineering and materials science interests in automotive and aerospace industries are focused on the development and application of composite structures in construction of complex products exhibiting specific physico-chemical and material properties [1]. One of the aims is to obtain the elasto-mechanical behavior of complex structures to be capable to withstand the applied external mechanical loads without damage of the individual structural components. Traditional reinforcing fillers such as glass, carbon [2–4], boron fibers, calcium carbonate [5], carbon black, titanium dioxide, kaolin [6], silicon dioxide, wollastonite [7] and mica particles were added to polyolefin matrices to improve their rigidity, high temperature resistance, toughness etc. [8–12]. Furthermore, there were numerous applications of novel fillers in the production of polyethylene based composites, such as carbon nanotubes [13], cellulose fibers of different nature [14–16], metal powders [17,18], peat ash [19] etc. Thermoplastics, such as poly (ethylene) (PE) can offer useful mechanical, chemical, electrical properties, with low density, high formability and the ability to be recycled. Due to its low price per unit volume and its unique physico-chemical properties it is therefore, the world's number one per volume most used thermoplastic [6]. This semi-crystalline

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[1] Deceased 16.9.2017.

https://doi.org/10.1016/j.compositesb.2017.12.035
Received 31 July 2017; Received in revised form 30 November 2017; Accepted 18 December 2017
Available online 24 December 2017
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polymer can be classified according its density and divided into four groups: high density polyethylene (HDPE), low density polyethylene (LDPE), linear low density polyethylene (LLDPE), and very low density polyethylene (VLDPE). In general, semi-crystalline polymers such as HDPE are regarded as a three-phase continuum composed of an amorphous phase, a crystalline phase, and an inter-phase. The crystalline skeleton is formed by mutually connected spherulites, each of them consist of a number of crystalline lamellae with an amorphous phase located in between the crystallites and lamellae [5,20–22]. Different crystalline morphologies, such as spherulites, cylindrites, shish-kebab and fibrous crystal can be obtained with variety processing conditions [23,24]. It was found, that the type and size of the filler particles has a strong effect on the HDPE crystallization kinetics and melting behavior [25,26] as well. However, no changes in the thermal oxidation mechanism of HDPE based composites filled with different inorganic fillers (e.g. mica, wollastonite, kaolin, talc or diatomite) were found [27]. In general, stress transfer in composite matrices is affected by structural, morphological and surface properties of the filler/matrix interface [11]. It is well known, that the polymer/filler interface quality performance is essential for excellent overall composite system material/mechanical properties. The exact adjustment of the polymer matrix modulus and adhesive bond strength is vital for the final synergistic increase in mechanical strength of the resulting composite system [28]. In most cases, silane coupling agents were used to create covalent bonds between filler particles and the polymer matrix [6,28–30]. In a paper [31] a prediction of the complex modulus of elasticity was studied at various strain rates by means of dynamic mechanical analysis. There was found a linear viscoelastic response to a given strain history. The paper of Xiang et al. [12] was studying DMA (Dynamic Mechanical Analysis) of HDPE/mica composites in a single cantilever mode at a frequency of 1 Hz. Tested composites were prepared by a dynamic packing injection molding (DPIM) technique allowing proper control of the central and skin layers of the prepared testing articles. They proposed the effect of the additional mica delamination induced by the injection flow and its orientation within the flow direction, allowing thus HDPE macromolecules melted matrix its intercalation in between galleries of mica layers under applied shear conditions. As a result, an increased storage modulus was found reflecting higher stiffness of the DPIM prepared HDPE/mica composite materials.

In this study, the mechanical properties of composites prepared from commercially available filler materials of mica (muscovite type) and wollastonite in HDPE matrix were investigated. Mica belongs to a group of silicate minerals, with the most common being muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$). Micas are used as a filler in insulators, condensers, plastics, cosmetics and paints. Micas are sheet silicate minerals whose TOT-type (tetrahedral-octahedral-tetrahedral) sheets are made of two tetrahedral layers sandwiching an octahedral layer. The tetrahedral layers consist of a hexagonal pavement of tetrahedra $(SiO_4)^{4-}$ in which each tetrahedron shares three apexes with the neighboring tetrahedra: the chemical composition of such layers is $(Si_4O_{10})^{4-}$. In each sheet, the tetrahedra of the upper tetrahedral layer point downwards, and the ones of the lower tetrahedral sheet point upwards [32]. The most prominent characteristic of mica is nearly a perfect basal cleavage. Wollastonite is a calcium silicate ($CaSiO_3$) industrial mineral, which is commonly used as a filler in paints and plastics [33,34]. It is also used in the construction industry as a substitute for asbestos; ceramic applications including ceramic glazes and bodies; in metallurgical applications wollastonite is commonly added to formulated powders for steel casting and welding. It is the only naturally occurring needle-shaped crystal, and the shape is an important economic aspect of wollastonite, with highly acicular samples being most expensive. There are three polymorphs: triclinic pseudowollastonite of very high temperature (above 1120 °C), monoclinic wollastonite-2M and triclinic wollastonite-Tc at lower temperatures. The usual form of wollastonite is the triclinic form.

As mentioned above, the reinforcing platelet shaped silicates in polymeric materials have been widely used due to their high aspect ratio, the effect of two-dimensional reinforcing as well as the overall materials cost reducing effect. However, the majority of the studies were performed in the static mechanical tensile testing configurations. Hence the better knowledge of the dynamic-mechanical performance at a wide frequency range is missing. This paper aims to study the effect of planar shape mica and prism shape wollastonite filler particles in HDPE polymer composites, specially investigating the mechanical properties (both static and dynamic in a frequency range of 2–3200 Hz), and the thermal and fracture mechanical behaviors. This will be combined with SEM analysis.

2. Materials and processing

High density poly (ethylene) (HDPE) type 25055E (The Dow Chemical Company, USA) was purchased in the form of white pellets (lot. No. 1I19091333). As filler particles the inorganic minerals muscovite mica (Imerys, Kings Mountains, USA) (specific surface area of 9.7 m^2/g, d_{50} of 17 μm, aspect ratio of 1.7) and wollastonite type VANSIL W-10 (Vanderbilt Minerals, Norwalk, USA) (specific surface area of 0.5 m^2/g, d_{50} of 49 μm, aspect ratio of 13.5) were used. There were prepared 250 composites samples of each filler type (dog bone shape for tensile testing, Charpy's pendulum and vibrator testing) of virgin HDPE and 5, 10 and 15 w.% of inorganic filler concentrations of HDPE/mica and HDPE/wollastonite composites.

Composite samples were made using the injection molding technique on the injection molding machine Arburg Allrounder 420C (Germany). Parameters for the injection molding machine: 1 × 40 mm diameter rotating screw, length 800 mm (L/D = 20). The processing temperature ranging from 190 to 220 °C, the mold temperature was kept at 30 °C, the injection pressure was 60 MPa, and the injection rate was 20 mm/s, injection cycle time was 45 s, holding pressure time was 15 s. Studied melted matrices were filled at the central part of the mold during injection molding process, thus enhancing flux of the material in the direction of the longer side of the dog bone testing articles. There were found parallel orientations of the filler particles in the final composite testing articles by SEM analysis. (For the visualization of the flux of the polymer matrix/filler melt in the mold see Appendix A. Supplementary data). As a feed material for injection molding granules prepared by melt blending of the polymer resin and the mineral filler were used. The latter granules were prepared by means of extrusion technique on extrusion machine LABTECH engineering model LTE20-40 Scientific (Thailand). Parameters of the extrusion machine were: 2 × 20 mm diameter co-rotating screws, length 800 mm (L/D = 40), extrusion rate 200 rpm, feeding rate 30 rpm. For the virgin HDPE processing temperature profile ranged from 136 to 172 °C. The mica and wollastonite filled HDPE samples temperature profiles ranged from 140 to 174 °C.

Supplementary video related to this article can be found at http://dx.doi.org/10.1016/j.compositesb.2017.12.035.

3. Methods

3.1. Scanning electron microscopy

Scanning electron microscopy (SEM) was used to determine the shape and size of the studied mineral composite filler particles. SEM images were captured using a Scanning Electron Microscope Hitachi SU 6600 (Japan). The source of the electrons is Schottky cathode. This microscope has the resolution in secondary electron mode (SE) 1.3 nm and in back scattered electrons (BSE) 3 nm. For these images, the secondary electron mode (SE) and an accelerating voltage of 5 kV (Fig. 1) or 1 kV (Fig. 2) were used. The distance between sample and detector was 6 mm. Studied materials were placed on double sided carbon tape on aluminum holder. All samples were metallized by gold with the thickness of 15 nm on sputter coater Quantum Q150T, LOT-Quantum

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