



Plasma modified Polytetrafluoroethylene (PTFE) lubrication of α -olefin-copolymer impact-modified Polyamide 66

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ARTICLE INFO

Article history:

Received 10 February 2015

Received in revised form

31 May 2015

Accepted 1 June 2015

Available online 11 June 2015

Keywords:

Polyamide 66

Impact modification

Polytetrafluoroethylene (PTFE)

Plasma treatment

Solid lubricants

Wear

ABSTRACT

Tribological and mechanical properties of α -olefin-copolymer, impact-modified Polyamide 66, containing pristine and plasma treated Polytetrafluoroethylene (PTFE) micro-powders as solid-lubricants have been investigated. The PTFE powders were subjected to low pressure 2.45 GHz microwave plasma treatment with H_2 and NH_3 as the process gases to aid their dispersion in PA66. Formation of polar surface groups in conjunction with significant defluorination was observed for both H_2 (F/C atomic ratio 1.30) and NH_3 (F/C atomic ratio 1.13) plasma treated powders. The H_2 PTFE-impact modified PA66 composites exhibited a 25% increase in their impact energy absorption capabilities (25.0 J) than their pristine counterparts (20.0 J), along with a significant reduction of the specific wear rate at higher p_v -values. At the p_v -value of 6 MPa m/s, the specific wear rates for composites containing hydrogen plasma treated PTFE and nitrogen plasma treated PTFE were reduced by 33% ($0.7 \times 10^{-6} \text{ mm}^3/\text{N m}$) and 50% ($0.6 \times 10^{-6} \text{ mm}^3/\text{N m}$), respectively, as compared to pristine PTFE- α -olefin PA66 composites ($1.1 \times 10^{-6} \text{ mm}^3/\text{N m}$); while showing similar coefficient of friction values. The use of plasma functionalized PTFE powders thus provides a facile route for the production of impact modified PA66 compounds with significantly lower coefficient of friction and higher wear resistance for applications like bearings and sliding elements where impact strength as well as good tribological properties are required.

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1. Introduction

Polyamide 66 (PA66) possesses high stiffness, wear resistance and coupled with ease of processing makes it a widely used polymer in tribological engineering applications such as gears and bearings [1,2]. These excellent physical properties result from the presence of hydrogen bonds in molecular chains of the polyamide [3]. However, PA66 shows low notched impact strength and to improve its properties against stress concentration, impact modification of PA66 with polyolefin rubbers/elastomers is well known [4]. Recently, polyolefin elastomers have been introduced as a soft material that can be used as an efficient impact modifier [5–7]. The α -olefin-copolymer is a copolymer of ethylene and another α -olefin such as butane or octane and is produced using metallocene catalysts with narrow molecular weight distribution. As α -olefin-copolymer is a non-polar polymer and has poor miscibility with polar polymers such as

polyamides, it is usually functionalised with low molecular weight polar groups on the polymer backbone. During the melt compounding of Polyamide 66 and maleic anhydride grafted α -olefin-copolymer, the amine groups of polyamide react with maleic anhydride to form a graft copolymer which increases the interfacial adhesion between the two phases. This leads to a chemical coupling across the interface during blending with the amine end groups of the polyamides, and to the formation of small particles in the polyamide matrix [7,8]. In fact, the Izod impact value of these modified compounds is more than one order of magnitude higher than for pristine PA66 [6]. Yu et al. have reported that the impact modification with elastomer/rubber is also beneficial for the friction and wear behaviour of these compounds [5], as the modification works as a surface lubricant. However, Chen et al. reported that polyolefin modification in Polyamide 66 compounds are only able to form a discontinuous patchy transfer film on the steel counter surface, which causes inconsistent friction and wear rates [9]. Therefore, it is desirable to develop α -olefin-copolymer impact modified Polyamide 66 compounds in conjunction with solid lubricants like Polytetrafluoroethylene (PTFE), which are able to form continuous

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friction films and thereby promoting a consistent tribological behaviour.

In the literature, the friction and wear behaviour of pristine nylons have been enhanced by using fillers and modifications [10–13] or by blending it with other polymers [14,15]. One of the predominant solid lubricants used with Polyamide 66 is Polytetrafluoroethylene (PTFE), which – due to its low coefficient of friction – is a well-known anti-adhesive material [16]. Significant amount of literature has been published on the influence of PTFE modification on polyamide and its compounds [17–20]. It was shown that PTFE decreases the coefficient of friction (CoF) and potentially also the wear rate, however, the mechanical properties of the compounds are reduced significantly. This reduction in the mechanical properties is largely due to the poor wettability and distribution of PTFE in the polyamide matrix. Polyamide–PTFE compounds are often produced via the melt mixing process, wherein the different surface energies of polar polyamide and non-polar Polytetrafluoroethylene results in its poor distribution in the polyamide matrix [21]. As a consequence, the friction and wear properties can vary in a wide range and are often poorly reproducible, even between samples of the same batch. The reported CoF and wear rates, especially when established as single point data, often do not reflect this inconsistent friction and wear behaviour. Furthermore, the presence of PTFE as a second phase weakens the mechanical properties of the compound. Therefore, a number of efforts have been made to alter the chemical structure of PTFE to incorporate functional groups via chemical etching, such as reduction with e.g. sodium naphthalene [21,22] or electron beam irradiation of PTFE [23–25]. The incorporation of functional groups changes the polarity and therefore the surface energy of PTFE, which enables the formation of inter-molecular forces to the polyamide matrix [25]. PTFE can also be functionalized by the use of low pressure microwave plasma and the method offers a versatile route to alter the surface of PTFE without affecting the bulk properties of the material [26]. It is easy to control [26], environmentally friendly, and relatively low in cost [27]. The functionalization of PTFE sheets, films or sintered components is already well known and the authors have recently reported on the functionalization of PTFE powders using low-pressure microwave plasma and their possible use as a tribological filler in high temperature Polyethersulfone composites [28,29]. The available literature reveals that H_2 and NH_3 plasma treatment of PTFE leads to defluorination accompanied by the production of hydrocarbons, cross-linking, chain scission and, depending on the feed gas used, incorporation of oxygen- and nitrogen-containing groups [30–32].

In this work, PTFE micropowders functionalised via low pressure NH_3 , H_2 microwave plasma treatment are used as a solid lubricant for enhancing the tribological properties of α -olefin-copolymer impact modified Polyamide 66. The compounds are further analysed using a host of characterisation techniques such as mechanical testing, dynamic mechanical thermal analysis, X-ray photoelectron measurements and tribological measurements.

2. Experimental

2.1. Low pressure microwave plasma treatment of PTFE powders

Commercially available PTFE micro-powder TF9201Z (Dyneon™, Burgkirchen), with a specific surface area of $10\text{ m}^2/\text{g}$ (ASTM 4567) and an average primary particle size of $6\text{ }\mu\text{m}$ (ASTM 4464), was plasma treated in a “Nano” microwave plasma device (Diener electronic GmbH+Co. KG, Ebhausen, Germany). The plasma device is equipped with a rotary glass drum and is suitable for the treatment of polymer powders of up to 3 kg quantity at a time. The plasma treatment conditions used were similar to our previous works and

are briefly described below [28,29]. A base pressure between 0.2 and 0.8 mbar was created using a rotary pump after loading the PTFE powder in the plasma chamber. Once the desired base pressure was achieved, the process gas (H_2 or NH_3) was fed into the chamber and 2.45 GHz microwave plasma (270 W) excited using high voltage at the preferred working pressures. The gas flow and rotation rate of the glass drum were held constant to ensure uniform treatment of powders as they passed the excited plasma between the electrodes. The modified PTFE samples were then removed from the chamber and sealed in a plastic bag, at ambient laboratory conditions, for subsequent analysis and further use with α -olefin-copolymer impact modified Polyamide 66.

2.2. Preparation of α -olefin-copolymer impact modified PA66–PTFE compounds

The PA66–PTFE compounds were produced using commercially available α -olefin-copolymer impact modified PA66 and pristine, plasma modified PTFE powders. The impact modification of PA66 is typically achieved via the incorporation of 20% grafted α -olefin copolymer [33,34]. The α -olefin copolymer itself is composed of ethylene-1-butene copolymer grafted with maleic anhydride (trade name TAFMER MA7010) consisting of 67.9 wt% ethylene, 31.6 wt% butene and grafted with 0.5 wt% maleic anhydride. In fact, the α -olefin-copolymer impact modified PA66 is considered as a two phase system, consisting of a hard phase (80% PA66) and a soft phase (20% α -olefin copolymer) and shows a density of 1.07 g/cm^3 with a melt volume-flow rate (MVR) of $3.84\text{ cm}^3/10\text{ min}$ ($275\text{ }^\circ\text{C}/5\text{ kg}$).

The PA66–PTFE composites were melt compounded on a co-rotating twin screw extruder, Coperion ZSK26 MCC with subsequent water bath cooling and pelletizing facilities. The temperature settings on the six zones of the barrel were chosen in the range of $260\text{--}285\text{ }^\circ\text{C}$ with the die plate temperature of $280\text{ }^\circ\text{C}$. For comparative purposes, and the impact modified PA66 is compounded with both pristine and plasma treated PTFE powders at 10 wt% loading. The formulation of the compounds and the corresponding sample names are shown in Table 1. Further preparation of samples for mechanical and tribological testing is described in the characterisation and testing section.

2.3. Characterisation and mechanical, tribological testing

X-ray photoelectron spectroscopy (XPS) data were obtained on an AXIS Nova Spectrometer (Kratos Analytical Ltd., UK) having a monochromated Al $K\alpha$ X-ray source (excitation energy of 1486.6 eV) by pressing the PTFE powders onto a high-vacuum carbon tape using clean glass slides to provide a smooth surface for analysis. For binding energy calibration, the signal was calibrated to the F-C-F peak in the high-resolution C1s spectra at 292.8 eV or the F1s signal at 689 eV . Dynamic mechanical thermal analysis (DMTA) were carried out in tensile mode with a TA Instruments DMA 2980 at a frequency of 1 Hz, a heating rate of $10\text{ }^\circ\text{C min}^{-1}$ and a temperature range of $30\text{--}200\text{ }^\circ\text{C}$.

Standard tensile test bars, according to ISO 527 and dart drop test plates with dimensions of $60\text{ mm} \times 60\text{ mm}$, with a thickness of 2 mm were moulded on Krauss Maffei KM125-390 injection moulding machine. The moulding process temperatures were chosen in the region of $255\text{--}280\text{ }^\circ\text{C}$ and the PA66–PTFE compounds were dried in a dessicant drier prior to the moulding process. Values from the tensile test according to DIN EN 527-2 were determined on a Zwick universal tensile tester Z020 (Zwick GmbH & Co. KG, Ulm). The melt volume-flow rates (MVR), according to DIN ISO 1133, were generated on a Zwick Aflow plastometer by applying a temperature of $275\text{ }^\circ\text{C}$ and a load of 5 kg. In order to investigate multiaxial impact behaviour of the compounds, an instrumented dart drop test was conducted according

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