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Nanomechanical and surface frictional characteristics of a copolymer based on benzoyl-1,4-phenylene and 1,3-phenylene

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Abstract

Surface mechanical and tribological properties of a copolymer based on benzoyl-1,4-phenylene and 1,3-phenylene were evaluated using nanoprobe investigation techniques and compared to the properties obtained at the macroscale. These copolymers are commonly referred to as self-reinforced polymers (SRPs) because of their intrinsic high strength and modulus without addition of a reinforcing agent. Specimens were prepared by spin casting, solvent casting, and compression molding. Surface mechanical properties and film thickness were measured by nanoindentation and scratching techniques. Friction properties were found using lateral force microscopy (LFM), and surface topography was imaged by tapping mode atomic force microscopy (AFM). Macroscale friction testing revealed a kinetic coefficient of friction of 0.08 for SRP, approaching that of Teflon. Similarly low relative friction coefficients were obtained in nanoprobe measurements. Nanoindentation of SRP, polycarbonate (PC), and polyetherimide (PEI) demonstrated superior surface hardness and modulus of SRP copolymer thin films.
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1. Introduction

Poly(paraphenylene) is a rigid rod polymer that in theory should possess ultra high strength and stiffness due to the rigid nature of its backbone, consisting exclusively of phenylene linkages. However, during the polymerization reaction only 6–10 repeat units are incorporated into the polymer chain before it precipitates from solution [1]. Incorporation of a comonomer with benzoyl substituents renders the copolymer soluble and a copolymer with a high molecular weight and intrinsic viscosity can be produced (Fig. 1). The strength and stiffness of the material is retained by the rigid phenylene linkages throughout the backbone, while the side group attachment gives the polymer its solubility [2,3]. Linear copolymers of 1,4-phenylene with benzoyl-1,4-phenylene, however, remain difficult to process via traditional melt processing techniques such as injection molding. Incorporation of 1,3-phenylene comonomer yields a copolymer with improved melt processability, but somewhat reduced modulus. These copolymers are commonly referred to as self-reinforced polymers (SRPs) because of their intrinsic high strength and modulus without addition of a reinforcing agent. Compared to most linear polymers, which possess a more coil-like structure, SRPs have reduced conformational and rotational motion. This inhibits their ability to flex and produces a much stiffer material. The strength of SRPs is also directly related to the aspect ratio of the rod-like segments of the polymer molecule, with a higher aspect ratio yielding a stronger material [2]. The solubility, stiffness, and melt processability of the copolymers can be specifically tailored by adjusting the copolymer composition. The SRP used in this study is a commercially available copolymer with approximately 15 mol% 1,3-phenylene and 85 mol% benzoyl-1,4-phenylene [4].

SRPs are amorphous polymers that can be processed by both solution and melt techniques into transparent, amorphous films and plaques [5,6]. SRPs exhibit dramatically increased strength, modulus and hardness properties in comparison to traditional engineering thermoplastics, as exhibited in reported properties for commercial SRP, polyetherimide (PEI) and polycarbonate (PC) polymers of similar molecular weights (Table 1) [7–9]. Additionally, SRPs have been demonstrated to form miscible blends with polycarbonate with intermediate modulus levels, providing potential opportunity for more easily processable high strength transparent materials [4]. Their ultra high strength, hardness and strength to weight ratio make SRPs of interest for applications ranging from light weight structural

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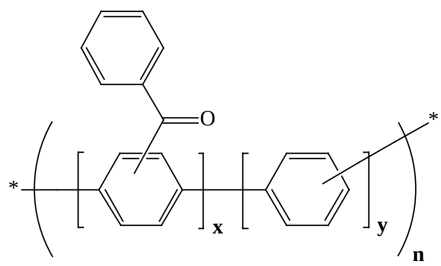

Fig. 1. Generalized chemical structure of a copolymer of benzoyl-1, 4-phenylene and 1,3-phenylene.

components to protective films and coatings. In particular, their demonstrated dielectric capabilities [6,10] combined with their high strength and hardness, indicate their potential for thin film and micro/nanoelectronic applications. For these types of applications, however, it is critical to understand not only the bulk mechanical properties, but also surface and nanomechanical performance of the materials, and property correlation from nano to macroscale. Specifically, the friction properties of SRP from nano to macroscale have been evaluated in comparison to polystyrene, which was chosen as a reference amorphous material that has been extensively characterized and whose nanotribological behavior has been previously reported [11]. Additionally, nanomechanical properties of SRP were evaluated in comparison to PEI and PC polymers, and their surface properties compared to reported bulk mechanical properties. The widely used engineering thermoplastics PEI and PC were chosen as reference comparative materials for evaluating SRP performance, and to provide materials with varying rigidity (SRP stiffness > PEI > PC). SRP, PEI and PC grades of similar molecular weights were chosen to minimize performance differences based on molecular weight.

Atomic force microscopy (AFM) and nanoindentation techniques are emerging as effective methods for measurement and prediction of thin film properties, including friction, wear, surface roughness, adhesion, lubrication, hardness, and modulus [11–14]. In this paper properties measured at both the nanoscale and macroscale are compared to assess the translation of properties from the molecular to macroscopic level, of particular interest for assessing performance for advanced thin film applications.

Amonton's law describes friction at the macroscopic level, where the frictional coefficient (μ) is the ratio of the frictional force (F_f) to the total normal force (F_n) [15,16].

$$\mu = \frac{F_f}{F_n} \tag{1}$$

However, polymeric materials deviate from this law due to effects from adhesion and surface tension [15,17]. AFM is used to evaluate relative friction measurements taking into account the adhesive forces experienced at the surface of materials under ambient conditions. The capillary forces between the tip and the liquid layer on the sample surface produce an adhesive force (F_a), which is added to the applied load (F_l) to give the total normal force applied to the sample.

Table 1
Bulk mechanical properties of SRP vs. traditional engineering thermoplastics

	SRP[a]	PEI[b]	PC[c]
Flexural modulus (MPa) ASTM D790	8300	3510	2340
Tensile stress at yield (MPa) ASTM D638	207	110	62
Rockwell hardness ASTM D785	80B (B scale)	109M (M scale)	70M (M scale)

[a] PARMAX® 1200 SRP Technical Data Sheet, http://www.mptpolymers.com.
[b] ULTEM® 1000 Technical Data Sheet, http://www.ge.com/en/.
[c] LEXAN® 144R Technical Data Sheet, http://www.ge.com/en/.

$$\mu = \frac{F_f}{(F_l + F_a)} \tag{2}$$

After rearrangement the frictional force can be expressed as:

$$F_f = \mu F_l + F_a \tag{3}$$

Fiction coefficient, μ, is found from the slope of a plot of F_f as a function of F_l [18].

Indentation characterization is a valuable method for evaluating the nanoscale response of materials. These methods are used to determine local hardness and modulus on the surface of a material. Measurements are based on a force curve generated as a stiff probe penetrates the material surface. A force curve plots the applied load to the probe with respect to displacement into the specimen, and information about modulus, hardness, elastic recovery, and plastic deformation is obtained [19].

Property measurements are based on the contact mechanics of an axisymmetric indenter with an elastically isotropic half space, developed by Oliver and Pharr [20]. Hardness values (H) are calculated as:

$$H = \frac{P_{max}}{A} \tag{4}$$

P_{max}, maximum applied load; A, contact area between the probe and the specimen.

Reduced modulus (E_r) values are taken from the slope (dh/dP) of the unloading portion of the force curve and are dependant upon the contact area by the relation:

$$E_r^{-1} = \frac{dh}{dP}\frac{2A^{1/2}}{\pi^{1/2}} \tag{5}$$

h, depth of penetration; P, applied load.

2. Experimental

2.1. Materials

Materials were used as received unless noted otherwise. Polystyrene (PS) of weight average molecular weight (M_w) 280,000 g/mol was purchased from Aldrich. SRP resin and compression molded discs were supplied by Mississippi Polymer Technologies, Inc., (MPT) Bay St Louis, MS.

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